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Application of Response Surface Methodology for Statistical Optimization of the Pectin Recovery from Durian Peel

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Abstract Pectin extraction from durian peel was conducted using response surface methodology (RSM) to determine optimal conditions for maximum yield. The study investigated the independent influence of temperature, time, pH, and solvent/material ratio on extraction yield. The experimental variables were set within specific ranges: temperature ranged from 75 to 85 ℃, extraction time varied from 45 to 75 minutes, pH was maintained at 1, and the solvent/material ratio ranged from 17.5/1 to 22.5/1 mL/g. By employing the Box-Behnken experimental design coupled with a quadratic polynomial regression model, the pectin extraction process was optimized utilizing response surface methodology. The optimized conditions, determined to be a temperature of 80 ℃, an extraction time of 52 minutes, and a solvent/material ratio of 20.0/1 mL/g, yielded a predicted maximum pectin yield of 12.7475% from durian peel. The extracted pectin exhibited characteristics such as a light brown color, 7.16% moisture content, ash content below 3.0%, protein content of 0.43%, methoxyl content of 3.59%, esterification degree of 50.70%, equivalent weight of 617.80 g/mol, and AUA content of 40.25%. The application of response surface methodology in the optimization of pectin from DP is promising for efficient and environmentally friendly waste management.

Keywords Pectin, Durian Peel, Optimization, Response Surface Methodology, Recovery

1. Introduction

Durian (*Druian zibethinus*) is a tropical fruit widely grown in Southeast Asia, specifically Malaysia, Thailand, the Philippines, Indonesia, and Vietnam [1]. This seasonal fruit is famous for its unique taste and aroma and is considered some fruit with high nutritional value that is increasingly gaining popularity globally. Apart from being consumed fresh, durian is also used in the food processing industry to make durian ice cream, candy, jam, and other products [2]. However, the edible part of the fruit only makes up about 15-30% of the whole fruit's weight, and about 70-85% of the remaining part of the durian fruit is considered waste, including the pulp, seeds, and peel.

The widespread consumption of these popular fruit varieties has led to a notable increase in agricultural waste within the environment. Estimates suggest that around 480,000 tons of waste from these fruits are produced annually. The large volume and high density of this waste pose challenges for traditional disposal methods such as landfill and incineration, rendering them inefficient.

Recently, there has been a rising interest and focus on the emerging trend of extracting valuable components from agricultural waste. This approach not only tackles the significant issue of agricultural waste in the environment but also yields economically valuable products [2].

Currently, agricultural by-products such as leaves, fruit peels, fruit pulp, etc. are considered the main source of raw materials for commercial pectin production. Pectin is polysaccharides which consist of several D-galacturonic acid units connected by 1-4 glycosidic bonds, and present in the cell walls and middle lamella of almost higher plants [2-3]. It is considered a common additive in the food industry to thicken, stabilize, emulsify, and gel products without altering their natural taste [2]. It can be applied in fruit jams, fruit juices, desserts, dairy products, jellies, or as a food additive to enhance antioxidant capacity [4]. Besides, pectin also has film-forming properties that make it useful as an edible coating or packaging material for preserving food products. In addition, pectin has many applications in medicine, from drug and gene transport to wound healing and tissue engineering [5]. It has also been shown to have biological activities such as anticancer properties, immune enhancement, and anti-mutagenic ability. Pectin can even aid in weight loss and reduce the risk of cardiovascular disease. Many studies also show that consuming soluble fiber, including pectin, increases bile acid secretion, lowering cholesterol and positively reducing the risk of cardiovascular disease [4]. Pectin is also studied for environmental applications, such as its use as an adsorbent for pollutants in wastewater (methylene blue dye in textile wastewater) [6], fluoride in water [7], and as a corrosion inhibitor for steel and tin [8]. Therefore, pectin finds extensive use across diverse industries. The widespread application of this material has generated a substantial annual demand for large quantities of pectin. Consequently, it is imperative to actively seek out suitable raw materials to meet the requirements of pectin production.

The most used methods for producing pectin are chemical and enzymatic, which involve various physical and chemical processes like hydrolysis, extraction, and dissolution of macromolecules. Among these methods, chemical extraction is preferred due to its high yield, low cost, economy, and short time. Ammonium oxalate, EDTA, sodium hexametaphosphate, or acidic hot water (AHW) are typical solvents used for chemical extraction, with AHW being the most popular, oldest, and most straightforward method that does not require complex equipment [2,9]. Studies have shown that AHW extraction yields 15-25% of pectin, which is higher than other techniques, and the pectin obtained is of higher quality and stability [2]. These advantages have led to the widespread use of the AHW method for extracting pectin from plant materials.

Recently, the response surface methodology (RSM) has been recognized as a valuable tool for optimizing processes where independent variables collectively influence the desired outcome [10]. RSM, a statistical and mathematical algorithm, has proven successful in developing, enhancing, and optimizing processes for extracting compounds [10-12]. Traditional engineering practices for optimizing multivariate systems usually entail altering one factor at a time. However, these conventional techniques involve numerous experiments and often fall short of providing comprehensive effectiveness due to their inability to consider combined effects. Moreover, these methods require a substantial amount of data to determine optimal levels and are characterized by prolonged durations, rendering them unreliable [13]. The primary objective of employing experimental design techniques is to comprehend the interaction among parameters, facilitating the optimization of test parameters and the creation of statistical models [13]. Surprisingly, there has been limited research to date on the statistical optimization of conditions for pectin recovery from Durian peel.

Hence, the current study endeavours to explore statistical optimization to ascertain the optimal conditions for pectin recovery from durian peel, utilizing the acidic hot water extraction method. This article initially delves into the independent effects of individual factors (such as temperature, time, pH, and solvent/material ratio) on pectin recovery efficiency. Subsequently, significant variables are optimized using the Box-Behnken design in conjunction with the Response Surface Methodology (RSM) approach. A model is constructed through experimental design to pinpoint the highest optimal conditions for pectin recovery. Additionally, the article highlights various characteristics of the obtained pectin under optimal conditions, encompassing moisture, consistency, protein content, solubility, DE content, equivalent weight (EW), methoxyl content (MeO), the anhydrobiotic acid content (AUA). The chemical structure of the pectin extracted from durian peel was analyzed using scanning electron microscope (FT-IR) infrared spectroscopy, and the surface morphology was observed through the scanning electron microscope (SEM) analysis.

2. Materials and Methods

2.1. Materials

This research utilized durian peel from the *Durian Zebithinus* Ri6 genus. The Durian peel (DP) was purchased in Hanoi, Vietnam. To prepare DP powder, the DP was first washed with water, dried, and cut into small pieces about 0.5 cm in size. It was done to make the drying and grinding process easier. The raw materials were then dried in a vacuum oven at 80 ℃ until their moisture content was less than 10%. The dried DP was then ground into a powder with a particle size of 250 µm. The resulting powder was stored in sealed bags at room temperature and protected from light. This powder was then used as a raw material for pectin extraction.

2.2. Methods

2.2.1. Pectin Extraction

Extraction of pectin from DP powder was performed by AHW extraction method and then precipitation of pectin with ethanol [3]. Initially, 10g of the DP material was weighed and placed into a conical flask containing water acidified with HCl to achieve the appropriate pH. Pectin extraction was carried out under suitable temperature, time, and S/M ratio conditions. The extracted mixture was then filtered through filter paper, and 95% ethanol was slowly added while stirring for 5 minutes. The mixture was allowed to stand for an hour to collect the precipitate, which was then filtered and washed with ethanol. The obtained precipitate was dried below 55 ℃ until its mass remained constant for 24 hours. The pectin extraction yield (P, %) was calculated based on the percentage of obtained pectin after extraction compared to the initial material (Eq. (1)) [14]:

$$P = \frac{m_1}{m_2}.100 \qquad (1)$$

where P: the pectin yield from DP, %; m_1: the mass of obtained pectin after drying, g; m_2: the mass of initial DP powder, g.

2.2.2. Optimization of Technological Parameters of Pectin Extraction from DP

To improve the pectin yield, optimization of the extraction process was carried out. Three factors were identified as having a significant impact on pectin yield: extraction temperature (X_1), extraction time (X_2), and S/M ratio (X_3).

As shown in Table 1, the Box-Behnken matrix method with three factors and three levels (Table 2) was utilized for optimization [15]. A total of 15 experiments were performed, with four central experiments used for error estimation (refer to Table 1).

To represent the relationship between the independent variables and the response, usually, the quadratic polynomial equation was given in the general form as follows:

$$Y=\beta_0+\sum\nolimits_{i=1}^{k}\beta_i X_i+\sum\nolimits_{i=1}^{k}\beta_{ii} X_i^2+\sum\sum\nolimits_{j<i}^{k}\beta_{ii} X_i X_j \qquad (2)$$

where b_0, b_i, b_{ii}, b_{ij}: the coefficients of the regression equation; X_i, X_j: the experimental factors to be optimized, or are the input variables; i and j range from 1 to k, k: the number of independent parameters, and in this study, k = 4; Y: the response, pectin yield, %.

2.2.3. Determination of Moisture, Ash, Protein Content of Pectin

The moisture content and ash of pectin DP were measured by using method AOAC (AOAC 2015) using a circulating hot air oven (OFA-32-8, Esco, Singapore) and a furnace (LT 5/13, Nabertherm - Germany). The protein content of the obtained pectin samples was measured using the Kjeldahl method [3].

Table 1. Box-Behnken experimental design matrix for pectin extraction from DP with the experimental data and predicted values

Std order	Code variables			Uncode variables			(Pectin yield, %)	
	X_1	X_2	X_4	Temperature, ℃	Time, min	The S/M ratio, mL/g	Experimental results	Predicted results
1	-1	-1	0	75	45	20	10.12	10.3263
2	1	-1	0	85	45	20	10.53	11.2413
3	-1	1	0	75	75	20	12.41	10.3688
4	1	1	0	85	75	20	10.38	8.7638
5	-1	0	-1	75	60	17.5	11.91	10.7463
6	1	0	-1	85	60	17.5	11.19	9.5713
7	-1	0	1	75	60	22.5	12.88	10.0788
8	1	0	1	85	60	22.5	12.34	10.5638
9	0	-1	-1	80	45	17.5	12.53	12.3275
10	0	1	-1	80	75	17.5	10.12	10.4550
11	0	-1	1	80	45	22.5	10.53	11.8350
12	0	1	1	80	75	22.5	12.41	11.2725
13	0	0	0	80	60	20	10.38	12.5833
14	0	0	0	80	60	20	11.91	12.5833
15	0	0	0	80	60	20	11.19	12.5833

2.2.4. Determination of Pectin Solubility

50 mg of DP pectin was dissolved in 40 mL of deionized water at room temperature with a 250 rpm stirrer for 15 minutes. The mixture was then centrifuged at 2500 rpm for 15 minutes, and the insoluble part was collected. The insoluble part was dried to a constant mass at 50 ℃ using a circulating hot air dryer (OFA-32-8, Esco, Singapore) [16]. The solubility was calculated based on the amount of pectin dissolved compared to the total experimental pectin according to Eq. (3):

$$S= \frac{m_0 - m_1}{m_0}.100 \tag{3}$$

where S: solubility of the DP pectin, %; m_0: the mass of the initial pectin, g; m_1: the mass of the insoluble pectin, g.

2.2.5. Determination of the Equivalent Weight (EW), Methoxyl Content (MeO), the Anhydrouronic Acid Content (AUA) and Degree of Esterification (DE) of DP Pectin

The EW and MeO of pectin were measured according to the method described by the authors [17-18]. To determine the EW of the sample, follow these steps: first, 1g of NaCl was dissolved in 100 ml of distilled water. Then, 2 ml of 96% ethanol and six drops of phenolphthalein were added to the mixture. After that, 0.5g of DP pectin powder was dissolved in this mixture and titrate the resulting solution with 0.1N NaOH until the colour changes to pink and persists for at least 30 s [19]. The volume of NaOH in the first titration (V_1) was recorded. Next, 25 ml of 0.25 N NaOH was added to this neutral solution and was stirred continuously at 20 ℃ for 30 minutes. Then, 25 mL of 0.25 N HCl solution and five drops of phenolphthalein were added and titrated with 0.1 N NaOH solution until pink and persisted for at least 30 s (V_2). The EW was calculated using formula (4), the MeO content was calculated using formula (5), the AUA content was calculated using formula (6), and the DE was calculated by the eq. (7):

$$EW = \frac{m}{V_1.N}, \text{ g/mol} \tag{4}$$

$$Meo = \frac{V_2.N.31.100}{m}, \% \tag{5}$$

$$AUA = \frac{176.\ 0.1.\ (V_1 + V_2).100}{m.1000}, \% \tag{6}$$

$$DE = \frac{V_2}{V_1+V_2}, \% \tag{7}$$

where V_1: the volume of 0.1N NaOH solution in the first titration, ml; N: the normality of NaOH, N; V_2: the volume of 0.25N NaOH solution in the second titration, ml; 31: the molecular weight of the methoxyl group.

2.2.6. FT-IR

The FTIR spectra of the studied pectin samples were determined using a FTIR infrared spectrometer (FTIR-6600, Jasco, Japan) with a wavelength range of 4000 to 400 cm^{-1} at a resolution of 4 cm^{-1} at 23 ℃.

2.2.7. Morphological Analysis

The samples were examined for surface morphology by scanning electron microscope SEM (JSM-6500, JEOL, USA) with an acceleration voltage of 5 kV and a pore size of 30 μm.

2.2.8. Data Analysis

The experimental data in this study were calculated using Minitab software Version ® 16.2.4. The DP pectin extraction was optimized by the response surface method with the support of the software Minitab (Verson ® 16.2.4). The significance of each regression coefficient has been assessed using P-values in ANOVA analysis. The differences observed are statistically significant, with a 95% confidence level ($p < 0.05$).

3. Results and Discussion

3.1. Effects of Independent Factors on Pectin Yield

For the pectin extraction from the peels of various plants, factors such as acid type, solvent pH, extraction temperature, extraction time and solvent: material ratio are important factors that significantly affect the pectin yield [20]. The choice of extraction solvent depends on the plant's cell envelope structure. In the case of durian peel, which has a relatively thick and rough structure compared to citrus, watermelon, dragon fruit, etc., a strong acid is necessary to break down the plant cell coat and quickly dissolve pectin into the solvent. It has shown that pectin yield from HCl acid is higher than citric acid extraction [1]. Therefore, following previously published results, this study used solvent HCL as an acidifying agent during the pectin extraction process.

3.1.1. Effects of Extraction Temperature on Pectin Yield

Temperature is considered one of the factors that significantly affect the extraction yield. Typically, a higher temperature leads to a more effective extraction. However, extracting at too high a temperature will result in unwanted components (not the extracted constituents), which will also be extracted into the solvent [9]. At the same time, high temperatures can also damage the structure and properties of the extracted components. Therefore, it is essential to analyze the impact of temperature and select the best temperature for pectin extraction that ensures maximum efficiency. In Fig.1a, pectin yield increases as the extraction temperature rises from 60 ℃ to 80 ℃ but decreases again when the temperature is raised to 90 ℃. The highest pectin yield, 8.08%, was obtained at an extraction temperature of 80 ℃. Similar results have been found during the pectin extraction process from pineapple waste [21], mango peels [22], and sweet tomato peels [23], as observed in this study. However, the extraction process of pectin from sweet tomato peels requires a higher temperature (90 ℃) [23]. This temperature difference can be attributed to the type of acid used and the pH level of the solvent.

When temperature increases, the hydrogen and ester bonds are affected, making it easier to break down the plant cell structure, thus facilitating the dissolution of pectin molecules into a better solvent. At the same time, increasing the temperature lowers the mixture's viscosity, which increases solubility and the ability to diffuse solutes into the solvent, resulting in improved pectin extraction efficiency [9,14,24]. However, further temperature increases can break down the glycosidic bonds in the pectin molecule, leading to its degradation. Moreover, high temperatures promote polygalacturonic chain de-esterification, which can affect the purity of the pectin [25]. Also, pectin extracted at high temperatures tends to be dark brown, significantly affecting the product's sensory appearance. Therefore, high temperature during pectin extraction is undesirable.

3.1.2. Effects of Extraction Time on Pectin Yield

During the extraction process, several processes occur, including diffusion and osmosis. It is essential to ensure that the extraction time is appropriate for these processes to occur fully and extract all the components from the raw materials [26]. If the extraction time is too short, the components may not dissolve entirely in the solvent. On the other hand, prolonging the extraction time may increase the solubility of unwanted impurities [27]. The process was tested under the same extraction conditions at different times to determine the appropriate extraction time for pectin. The results of Fig.1b found that increasing the extraction time from 30 to 60 minutes resulted in a higher pectin yield. However, after this point, the yield decreased with further increases in extraction time. The most efficient extraction time for pectin from DP was within 60 minutes, yielding 8.48%. When extraction with a short amount of time, only some pectin molecules dissolve into the solvent while the rest remain attached to the cell wall, resulting in low extraction yield [27]. However, with AHW extraction, pectin and other molecules like starch, resin, colloids, etc., also can dissolve into the solvent. At high temperatures, these impurities increase the viscosity of the extraction mixture, hindering the pectin molecules from dissolving into the solvent. Besides, high temperature and long reaction time can break some of the less stable bonds of pectin, leading to partial decomposition and decreased pectin yield as the extraction time prolongs [28].

Studies on extracting pectin from passion fruit peel [26,29], orange peel [28,30], and dragon fruit peel [31] have revealed that the extraction process takes longer when compared to durian peels. This delay is due to the cellular composition of the raw peels. The stronger the bonding between pectin molecules and the plant cell walls, the longer the extraction process typically takes.

3.1.3. Effect of pH Value of Extract Yield

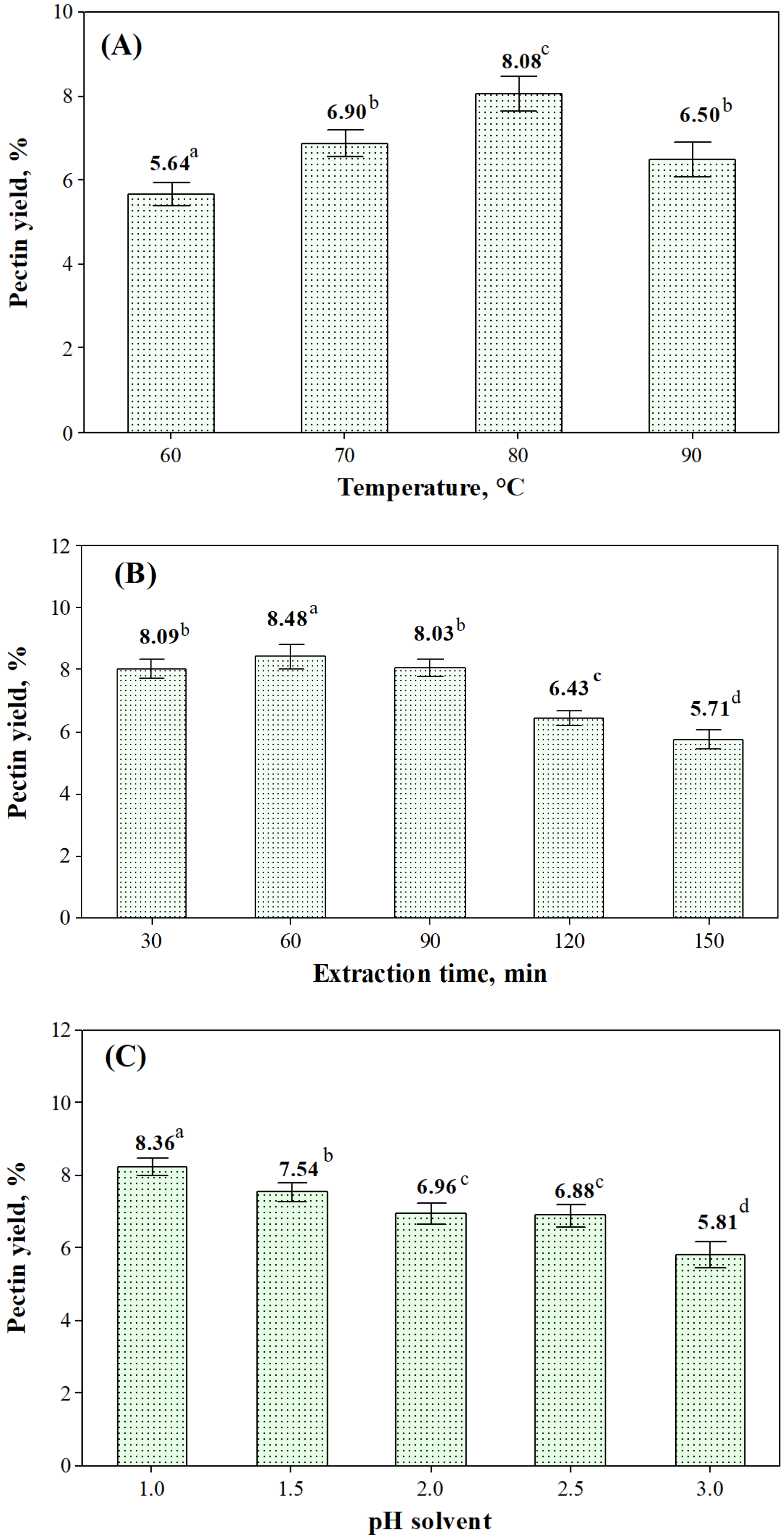
The effectiveness of pectin extraction is significantly influenced by the pH of the solvent [32]. Figure 1c demonstrates the effect of pH on pectin yield from DP, with temperature, time, and solvent ratio held constant. The data reveals that as the pH of the solvent changes from 1 to 3, the pectin efficiency fluctuates between 5.81% and 8.36%. The highest yield of 8.36% was achieved at pH 1. Similar results were observed in the extraction of pectin from citrus sinensis peel [32], banana peel [33], orange peel [28,30] and citrus wastes [34].

The more acidic the solvent, the higher the pectin extraction efficiency. This is because acidic environments neutralize more carboxylic groups, causing pectin to dissolve in more significant amounts of solvent [14,32]. Conversely, pectin is found in the middle lamina and primary cell wall of plant cells through intermolecular polysaccharide bonds. An acidic medium more efficiently breaks down the hydrogen and ester bonds between the pectin and the cell wall, releasing and diffusing pectin into the solvent and producing a high pectin yield under acidic conditions [3,28].

3.1.4. Effects of S/M Ratio on Pectin Yield

For the extraction process, the S/M ratio is also one of the essential factors affecting the yield of the constituents to be extracted. The desired compounds won't be extracted entirely from the raw materials if the ratio is too small. Conversely, if the ratio is too large, it will lead to solvent wastage and be uneconomical. Investigating and choosing the appropriate S/M ratio for the research is essential. The data in Fig.1d indicates that the pectin yield from DP powder is affected by the S/M ratio. As the ratio increases, the extraction yield increases; until it reaches a ratio of 20/1 to 25/1 mL/g. At this point, the pectin yields an insignificant decrease. Among the various S/M ratios investigated, the highest pectin yield was 12.52% at a ratio of 20/1 mL/g. This result can be explained by the fact that a higher S/M ratio causes a difference in concentration between the plant cells on the inside and outside. As a result, the more solvent is diffused into the plant cell, the more favorable the dissolution of pectin into the solvent. Therefore, the extraction efficiency increases. If you increase the S/M ratio beyond 20/1 mL/g, the solution becomes too full of molecules. It causes problems with how quickly the pectin dissolves into the solvent from the raw materials. The mass transfer rate is negatively impacted. As a result, the yield of pectin will decrease.

Based on the data, it has been determined that the best results for pectin extraction are achieved under these specific conditions: extraction temperature: 80 ℃, extraction time: 60 minutes, pH = 1, S/M ratio: 20/1 mL/g.


(A)
Pectin yield, %
Temperature, °C
60
70
80
90
5.64a
6.90b
8.08c
6.50b
(B)
Pectin yield, %
Extraction time, min
30
60
90
120
150
8.09b
8.48a
8.03b
6.43c
5.71d
(C)
Pectin yield, %
pH solvent
1.0
1.5
2.0
2.5
3.0
8.36a
7.54b
6.96c
6.88c
5.81d

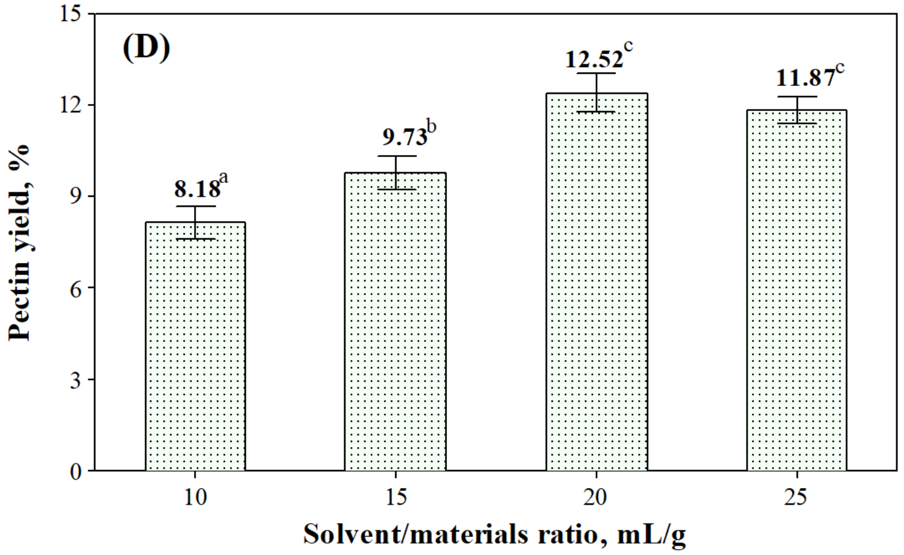


Figure 1. Effect of extraction temperature (A), extraction time (B), solvent pH (C), S/M ratio (D) on pectin yield from DP

In the initial stages of the foundational investigation, it was noted that the pH value significantly influences pectin efficiency; however, lowering it too much could disrupt the structure of the obtained pectin, resulting in changes to its properties. Therefore, three key factors chosen for optimization include extraction temperature, extraction time, and the solvent-to-material ratio. To improve precision in the optimization process, narrower ranges of variation for these factors were selected, as indicated in Table 2. Opting for these narrower ranges allows for a more precise optimization. Table 2 details the baseline levels and variability of each factor, determined through an independent examination of the proposed factors.

Table 2. The baseline levels and variability of factors in pectin extraction from DP

Parameters	Levels			Variation range
	Min (-1)	Mean (0)	Max (+1)	
Extraction temperature, ℃	75	80	85	5
Extraction time, min	45	60	75	15
The S/M ratio, mL/g	17.5/1	20/1	22.5/1	2.5/1

3.2. Optimization of Factors in Pectin Extraction from DP

To improve the process of extracting pectin, the variable ranges of factors listed in Table 2 were utilized based on the survey data regarding the independent influences on pectin yield. Conducting an experiment according to the Box-Behnken matrix and reviewing the results outlined in Table 2 can optimize the factors involved in the pectin extraction process to achieve the highest possible yield. The experimental results in Table 1 show that the pectin yield from durian peel ranges from 9.53% to 12.61%. The results of Table 1 also show that different extraction conditions lead to different pectin yields. The pectin yield obtained from DP is lower than the pectin yields from the lime peel (12.93-29.05%) [28,32], orange peel (27.5%) [27], pineapple waste (13.78%) [21], and mango peel (18.159%) [21]. However, it is higher than the pectin yields from grapefruit peel (7.95-10.84%) [20], apple (5.15%) [20], and sweet tomato peel (2.59%) [23]. The differences in extraction efficiency between this study and previous reports are mainly due to the composition of plant cell materials in the raw materials and different extraction methods [28,35]. Specifically, other raw materials contain varying levels of pectin. Additionally, this study employs the hot acid extraction method without supplementing pretreatment steps such as ultrasound or microwaves. Therefore, it leads to lower pectin yields compared to previous reports.

To build a model describing the simultaneous influence of the factors: extraction temperature (X_1), extraction time (X_2), and S/M ratio (X_3) on the pectin yield obtained from DP, the response surface method is used. The quadratic models related to pectin yield according to the coded variables (factors) are given in Eq. (8):

$$Y=12.583-0.609X_2-1.82X_1^2-0.588X_2^2-0.523X_3^2-0.482X_4^2-0.63X_1X_2+0.415X_1X_3+0.328X_2X_3 \tag{8}$$

Table 3. The results of analysis of variance (ANOVA) for yield pectin from DP

Term	Coefficients	Degrees of freedom (DF)	Sum of squares (SS)	Mean square (MS)	F value	P value
Constant	12.583	9	19.4018	2.1558	51.07	0.000
X_1	-0.173	1	0.2381	0.2381	5.64	0.064*
X_2	-0.609	1	2.9646	2.9646	70.24	0.000
X_3	0.081	1	0.0528	0.0528	1.25	0.314*
X_1^2	-1.820	1	11.3170	12.2360	289.90	0.000
X_2^2	-0.588	1	1.1142	1.2762	30.24	0.003
X_3^2	-0.523	1	1.0096	1.0096	23.92	0.005
X_1X_2	-0.630	1	1.5876	1.5876	37.61	0.002
X_1X_3	0.415	1	0.6889	0.6889	16.32	0.010
X_2X_3	0.328	1	0.4290	0.4290	10.16	0.024
Residual error		5	0.2110	0.0422		
Lack-of-Fit		3	0.0610	0.0203	0.27	0.845
Pure error		2	0.1501	0.0750		
Total		14	19.6128			
R^2	0.9892					
R^2(adj)	0.9699					
R^2(pred)	0.9330					
S	0.2055					
PRESS	1.3133					

* Not signifcant parameter (p> 0.05)

The determination coefficient R^2 is a characteristic value for the goodness model fit with the experimental research results. Table 3 shows that the value R^2 is 0.9892, close to 1 and indicates that up to 98.92% of the variables are explained by the quadratic model given (Eq.8). The predicted and adjusted values R^2 reach 0.9330 and = 0.9699, respectively, which are both relatively high and in agreement with the value of R^2. It clearly shows a correlation between the experimental results and the value predicted by the given model [17,32]. A lower coefficient of variance (S) indicates higher accuracy and reliability of experiments, shown in this study by S reaching 0.2055. The PRESS value expresses the predictive power of the quadratic model, with lower values indicating better predictive power. Table 3 shows that PRESS is 1.3133, indicating the model's appropriate and reasonable predictive ability. The non-significance of the lack of fit F and P values (0.27 and 0.845, respectively) suggests that model (8) can be used to predict pectin efficiency [32].

To easily observe the independent influence and interaction between survey factors on the objective function of pectin extraction efficiency, the coefficients in the regression equation are shown in the chart of Fig.2. In which, linear variables X_1, X_2, X_3 are represented by 1, 2, 3; square variables X_1^2, X_2^2, X_3^2 are represented by 4, 5, 6; and the integrative variables X_1X_2, X_1X_3, X_2X_3 are represented by 7, 8, 9. Table 3 and Fig. 2 also show that the variables X_1, and X_3 (temperature, and ratio S/M) have no significant effect on pectin extraction yield from DP, with a P value greater than 0.05. Therefore, the equation (8) does not include X_1 and X_3 coefficients. The coefficients obtained from the regression equation show that the positive coefficients include X_3, X_1X_3 and X_2X_3, indicating that the linear effect of S/M ratio, and the interactive effect of temperature-S/M ratio, time-S/M ratio promote pectin yield. Meanwhile, the negative coefficients from the regression equation include X_2, X_1^2, X_2^2, X_3^2, X_1X_2, indicating that the linear effect of time, the quadratic of temperature, time, S/M ratio, and the interaction of temperature-time detrimental influence the pectin extraction yield.

Additionally, Fig.3 displays the percentage contribution of the linear, quadratic, and interactive coefficients, demonstrating that the quadratic coefficients have the highest percentage contribution and significantly influence pectin yield. Meanwhile, the linear effect of the independent variable on pectin extraction efficiency was observed to be the lowest. Based on the estimated model, it was found that the square influence of extraction temperature on pectin yield was more significant than the impact of the other three factors.

Besides, the plot of the residuals versus experimental runs was used to assess the fit of the given model to the experimental data. Fig.4 can conclude that all experimental data fall within ± 0.3. It indicates significant agreement between the given model and the experimental data.

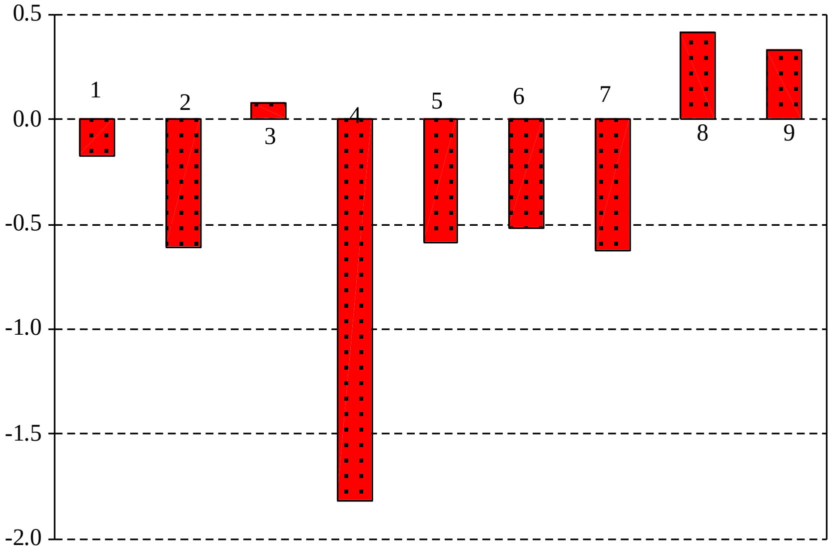


Figure 2. The coefficient of each factor or factor combination for the pectin yield response

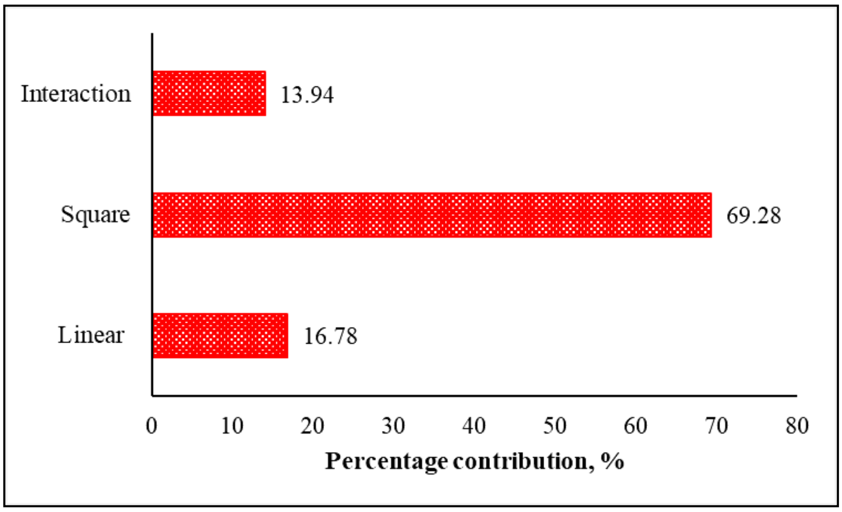


Figure 3. Total percentage contribution of process variables

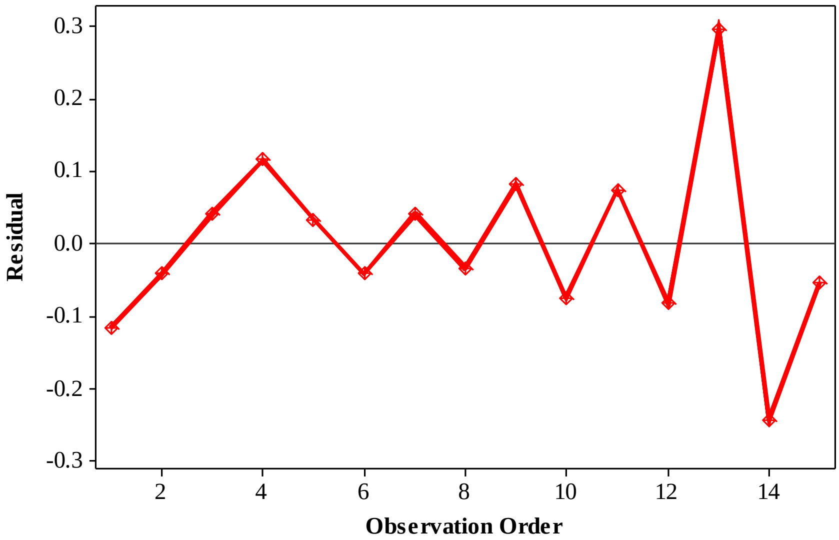


Figure 4. Residuals vs order for pectin yield

Response surface and contour plots (Fig.5) were generated to describe the relationship between factors and pectin yield. One variable is kept constant, and any two variables are changed to obtain the plots. Through plots, it can be advantageous to directly evaluate the independent and interactive correlations of variables concerning the response (pectin yield). The shape of the surface response plot can help understand the interaction between extraction factors and pectin collection efficiency. Figures 5A and 5B illustrate the relationship between temperature and extraction time. They indicate that the pectin yield increases as the extraction temperature rises up to 77.5 ℃. Continuing to increase the extraction temperature beyond 82.5 ℃ shows minimal improvement in extraction efficiency and surpassing 82.5 ℃ leads to a reduction in pectin yield. It has been observed that higher temperatures can help solvent penetration into plant cells, leading to higher efficiency in extracting pectin [38]. However, an increase in extraction time can lead to decreased efficiency. This is because high temperatures and acidic environments (pH=1) can cause the breakdown of pectin structure over time, thus reducing efficiency extraction. This trend differs from the findings in lime peel [32] and *Dillenia indica* fruit [36] pectin extraction. This result is related to the pH of the solvent and the acid used. The observations in Fig. 5A and 5B show that increasing extraction time and temperature can enhance pectin efficiency, reaching its maximum at 82.5 ℃ and 65 minutes, leading to decreased efficiency extraction. This aligns with the same pattern observed in cocoa pod husk [37] and orange peel [28] pectin extraction. Pectin extraction efficiency also gradually decreases when increasing the extraction temperature and the solvent-to-material (S/M) ratio. However, if the extraction temperature exceeds 82.5 ℃ and the S/M ratio goes beyond 22/1 mL/g, there is a noticeable trend of decreasing extraction efficiency (Figures 5C and 5D). A similar pattern is observed in the relationship between extraction time and the S/M ratio. Specifically, as both the extraction time and the S/M ratio increase, the extraction efficiency tends to rise. However, when the extraction time exceeds 65 minutes and the SM ratio surpasses 21.5 mL/g, there is a gradual decline in extraction efficiency. Therefore, from the response surface and contour plots, it can be observed that the pink-colored region represents the area with the highest pectin extraction efficiency. This corresponds to an extraction temperature range of 77.5-82.5 ℃, an extraction time between 45 - 65 minutes, and an SM ratio ranging from 18.5 to 22.0 mL/g.

This study used the regression model (8) to find the optimal extraction conditions to extract the maximum amount of pectin from DP. The optimal extraction conditions were obtained for four factors, at a temperature of 80.1515 ℃, time of 51.6667 min, and S/M ratio of 19.7727 mL/g with the predicted optimal yield of 12.7475% (Fig.6). However, considering the practical operability, the optimal conditions can be adjusted as follows: temperature of 80 ℃, the extract time of 52 min, S/M ratio of 20/1 mL/g. Performing the extraction process at the given optimum conditions, the extraction efficiency of pectin obtained from DP reached 12.813%, equivalent to the predicted yield of 12.7475%.

To verify the stability and accuracy of the experiments, three more randomized experiments were performed with the corresponding conditions, as indicated in Table 4. The results in Table 4 demonstrate very little difference between the experimental and predicted yields, confirming that the proposed model can accurately predict the maximum pectin yield from DP by AHW extraction.

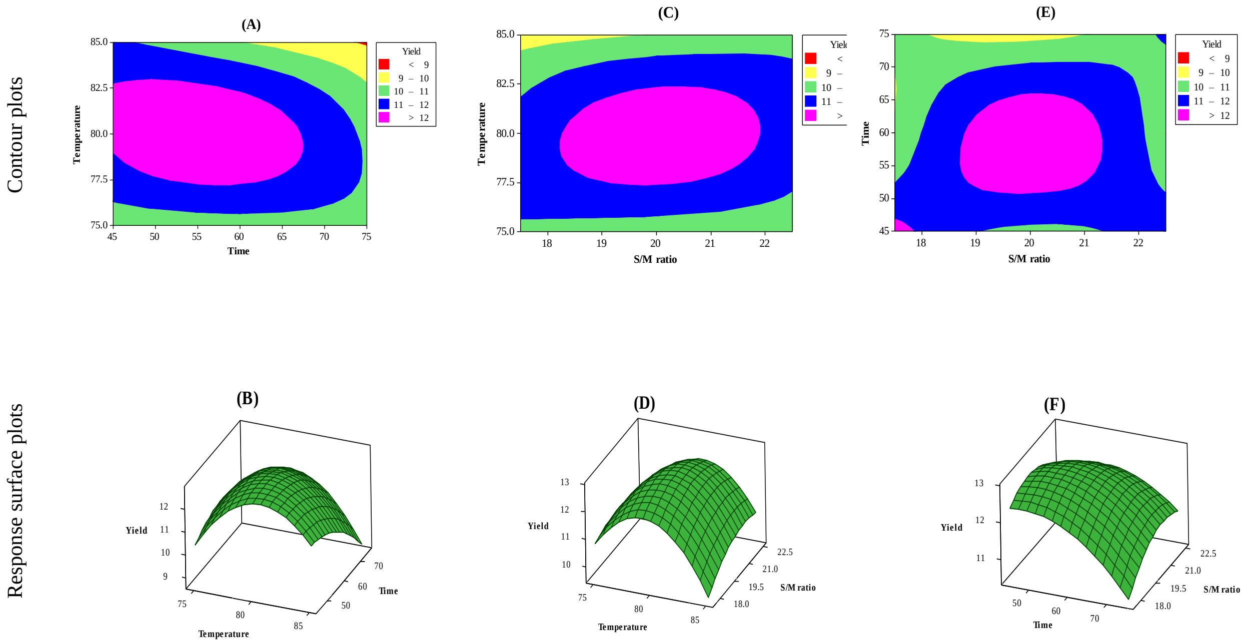


Figure 5. Response surface plots and contour plots for the combined effect

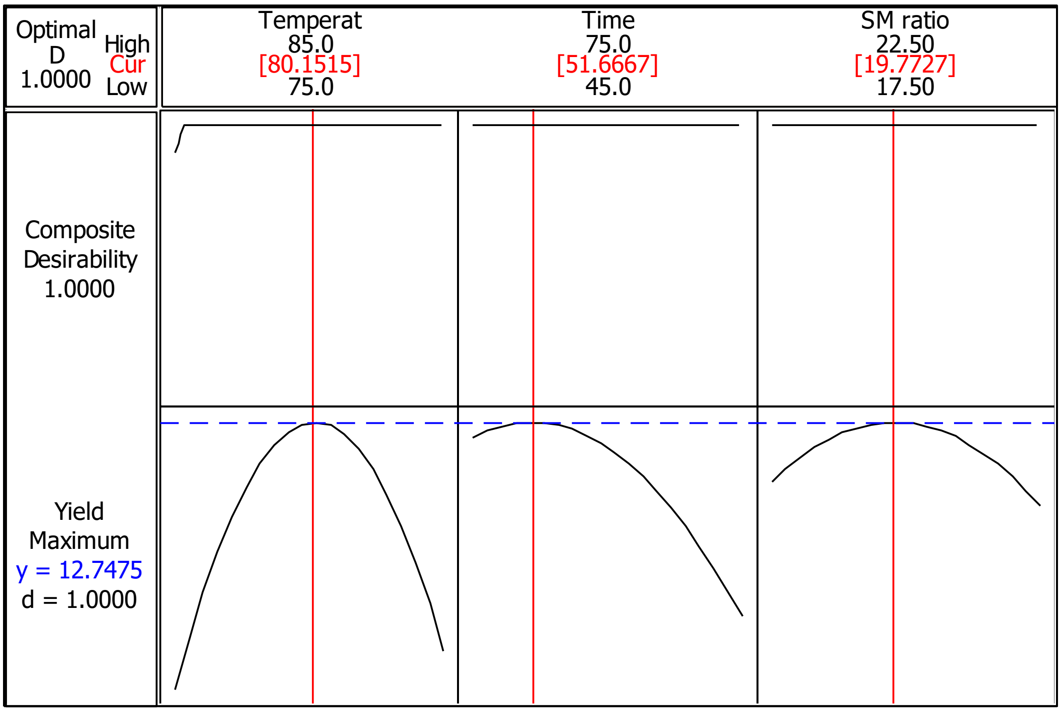


Figure 6. Optimization Plot

Table 4. Selected optimum conditions with predicted and experimental data

Std order	Temperature (℃)	Time (min)	S/M ratio	Predicted yield, %	Actual yield, %	Error with predicted value (%)
The optimal experiments						
1	80	52	20/1	12.7475	12.96	0.2105
2					12.85	0.1025
3					12.63	0.1175
Average yield ± standard deviation					12.813 ±0.168	
The randomized experiments						
1	84	75	22/1	9.9064	10.06	0.1536
2	76	45	21.5/1	10.7512	10.68	-0.0712
3	85	45	22.5/1	10.9982	11.14	0.1418

Table 5. Physicochemical characteristics of pectin extracted from DP

Characteristics	Contents in this study	Standard of pectin according to FAO [40]	Pectin according to IPPA [41]
Moisture (%)	7.16	Not more than 12%	Not more than 12%
Ash (%)	< 3.0	Not more than 1%	Not more than 10%
Protein (%)	0.43	Not more than 2.5%	-
Solubility (%)	90.09	-	-
The EW, g/mol	617.80	-	Not more than 800
The MeO, %	3.59	-	Not more than 7.12
The DE values, %	50.70	-	-
The AUA content, %	40.25	Not less than 65%	Not less than 35%

3.3. Physicochemical Properties of DP Pectin

Typically, the characteristics and properties of pectin will depend on the source material and extraction conditions [3]. The characteristics and properties of the pectin obtained can provide information for the best application [38]. Therefore, it is necessary to consider the properties of pectin extracted from DP. The physicochemical characteristics and some basic properties of DP pectin are presented in Table 5.

The moisture content of the DP pectin sample was 7.16%, which is lower than what has been previously reported for pectin derived from Gac pulp [3], orange peel, papaya peel and watermelon rind [39] and commercial pectin [40-41]. However, it is similar to the moisture content of pectin from citrus fruit peel [39] and pomelo peel [20], but it is higher than the moisture content of pectin from mango peel (6.766%) [22]. Maintaining moisture levels below 10% helps preserve the product by preventing the growth of microorganisms and the action of enzymes [20].

The ash content of DP pectin reached 2.78% (dry matter content), which was lower than previously studied: commercial pectin [40], pectin from orange peel (3.29%), papaya peel (12.06%) [39], mango peel (2.63-3.28%) [22,39] and watermelon rind (2.98%) [39], but higher than the pectin obtained from chayote peel (2.44%) [39]. The variation in ash content can be attributed to the fruit's topography, cultivation, variety, and farming methods in different regions [20]. The differences between studies were wide-ranging, related to the extraction technique and the nature and composition of the ingredients. Pectin with lower ash content is considered higher quality because it contains fewer impurities [20,42].

The amount of protein in pectin dramatically affects its ability to emulsify. In Table 5, it is shown that the pectin from DP had a protein content of 0.43%, which is higher than the pectin from Gac pulp [3] and grapefruit peel [43] but lower than the pectin from sour orange peel [44]. It is shown that the variance in protein content is due to the type of acid used for acidification as well as the low pH of the extracting solvent. In acidic environments, most proteins become denatured. When the pH level drops to 1, protein molecules degrade, producing a relatively low protein content in the obtained pectin [23].

The solubility of pectin is also an important property when investigating the properties of pectin. The solubility of pectin will affect the application of pectin in food. Table 5 reveals that pectin extracted from DP has a high solubility of 90.09%. It has been found that the solubility of pectin from DP is lower in comparison to pectin obtained from pomelo and apple peels [20,39]. This difference in solubility is said to be dependent on the ripeness of the fruit

[45]. As the fruit ripens, the amount of pectinase enzyme in the fruit increases, which leads to the increased solubility of pectin due to the activity of this enzyme [20,46]. With the high solubility value of DP pectin suggests that pectin from DP may be a favorable option for application in food such as acting as a gelling agent, stabilizing agent in fruit preserves, providing stability in fruit juice products, and serving as a thickening agent in pastry fillings. Additionally, due to the high solubility of pectin DP, it also suggests convenient applications for pharmaceutical encapsulation or as a coating material for medications.

The EW value of pectin is related to its gelling ability, thickening properties and stability when applied in food technology [17]. The EW is the total amount of free (non-esterified) galacturonic acid in pectin, which is affected by factors such as pH, extraction solvent, and free acid content [14]. A lower EW value indicates a lower gelling capacity [14,47]. In this study, the EW value of pectin extracted from DP was 617.80 g/mol, which was lower than other sources such as lime peel (1744.66-1829.80 g/mol) [32], orange peel (1371 g/mol) [48], banana-papaya peel mixture (783.69 g/mol) [14], and higher than the EW pectin from the grape pomace (531.04 g/mol) [48], orange peel (599.75 g/mol) [28]. This result could be due to the polymerization of pectin at lower pH, the extraction method, the nature of the plant cells, and the quality of the raw materials used for the extraction [14,32,47].

The MeO content is an essential indicator of pectin quality, indicating the number of free-esterified carboxyl groups in pectin. It dramatically affects gel strength and setting time [14,32]. Additionally, the MeO value indicates the ability of pectin to distribute in water. The higher the gelling capacity of MeO, the stronger the cohesive and adhesive forces and the higher the hardness of food products [49]. In this study, pectin extracted from DP had a MeO value of 3.59%. This result is lower than the MeO content of pectin from grape pomace (4.56 - 5.31%) [14], banana and papaya mixed peels (8.37%), standard pectin according to the International Pectin Producers Association (2.5 -7.8%) [40], lime peel (5.06-5.48%) [32], mango peel (7.33%) [50], banana peel (7.03%) [50]. However, the MeO content found in this study indicates higher levels than that in dragon fruit peel (2.98%) [51]. The MeO value of pectin varies 0.20-12% depending on the source and quality of the raw material, extraction method, extraction conditions and titration procedure [18,32,52]. It has also been reported that the methoxyl content depends on the ripeness of the fruit. As the fruit ripens, the sugar content increases while the methoxyl content decreases [32,53].

The ability of pectin to form a gel is greatly influenced by its degree of esterification (DE), which directly impacts its physicochemical characteristics [54]. Table 5 shows that this study found that the DE value of pectin from DP reached 50.70 %. The DE of pectin in this study is higher than that of pectin from banana peel (47.58%) [54], sweet lemon peel (35.1%) [55]. However, it is lower than pectin from banana and papaya mixed peels (67.91%) [14], grapefruit peels (61.19 - 70.79%), unripe banana peel (75.03%) [56], watermelon rind (57.3%) [24]. The DE value obtained indicates that DP pectin in this study can be classified as high methoxyl pectin with DE >50% [54]. This DE value meets the requirement of commercial food-grade high methoxyl pectin. The DE value can be influenced by various factors such as species, tissue structure and fruit maturity stage, material source and extraction method.

The pectin classification will provide information on gelling potential. For HMP-type pectins, gels can form with sugars and acids. Besides, the setting time of HMP will happen faster, about 20-70 seconds [14]. Thus, the physicochemical properties of DP pectin in this study can be considered high methoxyl pectin, which can be applied to canned products. At the same time, the studied pectin is also proposed for application in the food industry as a gelling agent, thickener and stabilizer.

The AUA content in pectin will evaluate the structure and texture of pectin. Pectin with a higher AUA content results in tighter bonds that are formed during gelation and are indicative of the purity of the pectin [57]. In this study, the AUA content in pectin was 40.25%. This result is lower than pectin from banana peel (69.67% AUA) [32], cocoa pod husk (20-45.37% AUA) [57], jackfruit skin (43.52% AUA) [59], mango peel (53.21%) [50], guava peel (53.92%). The AUA content may depend on the raw material, solvent, and extraction method [14]. The AUA value of commercial pectin, according to the IPPA standard, requires at least 35% [40]; according to FAO, it is at least 65% AUA [41]. This indicates that the pectin in this study is up to the standard of commercial pectin according to the IPPA standard.

3.4. FTIR of Pectin

Fourier transform infrared spectroscopy FTIR was utilized to characterize the bonds in pectin from DP. The resulting FTIR spectra in Fig.7 showcased several peaks indicative of specific vibrations within the pectin molecule. The peak observed at 3417 cm^{-1} characterizes the stretching vibration of O–H in the pectin molecule [18,54]. Meanwhile, the smaller peak observed at approximately 2800-3000 cm^{-1} indicated C–H vibrations, including CH, CH_2, and CH_3 [54,57]. The peaks around 1730 cm^{-1} and 1634 cm^{-1} are related to the CO of the carboxylic acid methyl ester and ionized carboxyl (–COO–) [3,54]. The stretching vibrations at roughly 1400 cm^{-1} corresponded to the CO of the free carboxyl groups [57]. Furthermore, the bands between 1000-1200 cm^{-1} indicated a typical C–O stretch, such as a glycosidic linkage. These findings confirmed the presence of pectin from DP and were consistent with previous FTIR spectra analyses of pectin from Gac pulp [3], apple and grape pomaces [17], and passion fruit peel [54], cocoa pod husk [57].

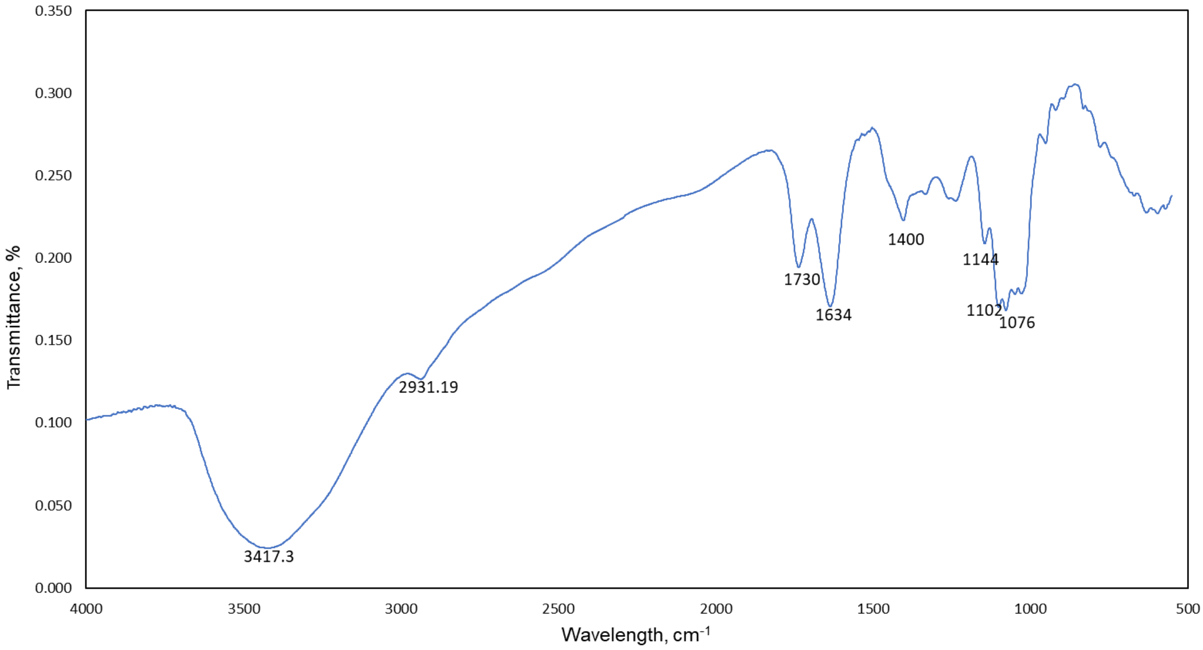


Figure 7. FTIR of DP pectin

3.5. Morphological Analysis of DP Pectin

Fig.8 displays the SEM results for both dried DP powder and pectin powder extracted from DP. These results visually highlight the differing surface morphological characteristics between the two powder samples. As depicted in Fig.8a, the raw material powder exhibits a porous and ridged texture. This texture can be attributed to the cellulose and fibre content in DP [3,19]. The morphology of the pectin reveals that the examined sample is irregular in size and shape, featuring a rough surface with numerous wrinkles and a slightly ruptured appearance (Fig.8b). Fig. 8b depicts the DP pectin structure with elongated, sharp particles. The coarse surface texture of the pectin in this study might be due to the impact of high temperatures during extraction and the extraction technique employing strong acidic water [3]. The irregular particles and rough texture of DP pectin correlate with its high content of insoluble fibers such as cellulose, hemicellulose, and lignin [17]. Similar surface morphologies and structures were also observed in pectin sourced from cocoa fruit pulp [58] and Gac pulp [3], grape pulp [17], lime peel [38], and jackfruit waste [59].

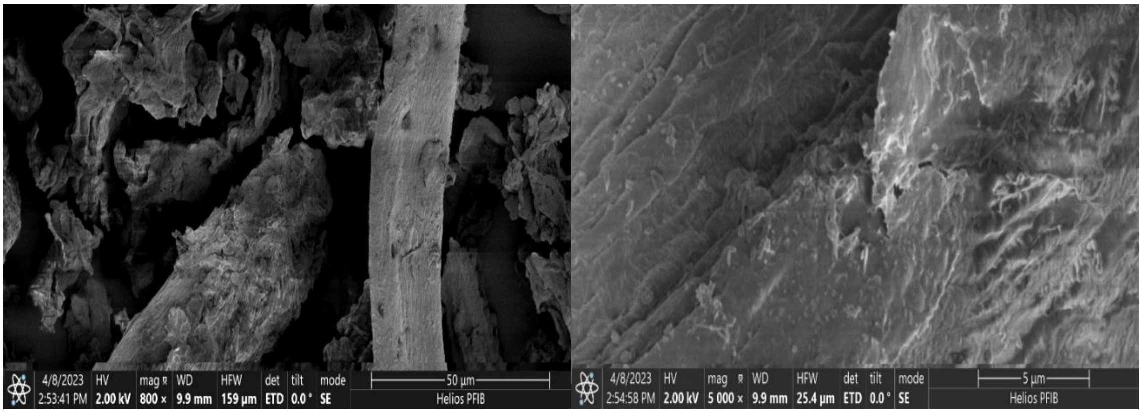
3.6. Discussion about Economic Benefits and Environmental Impact of the Study

In the current landscape, agricultural waste is increasing at an alarming rate. Given the urgent need for environmental sustainability, there's an escalating imperative to find economical and efficient methods to manage this waste. A promising solution is the recovery of valuable components from this waste [60]. A prime example is extracting pectin from durian peels. This offers an effective way to address the significant volume of agricultural solid waste, sidestepping the drawbacks of land-intensive landfill disposal and the environmentally harmful practice of incineration, which releases pollutants into the air.

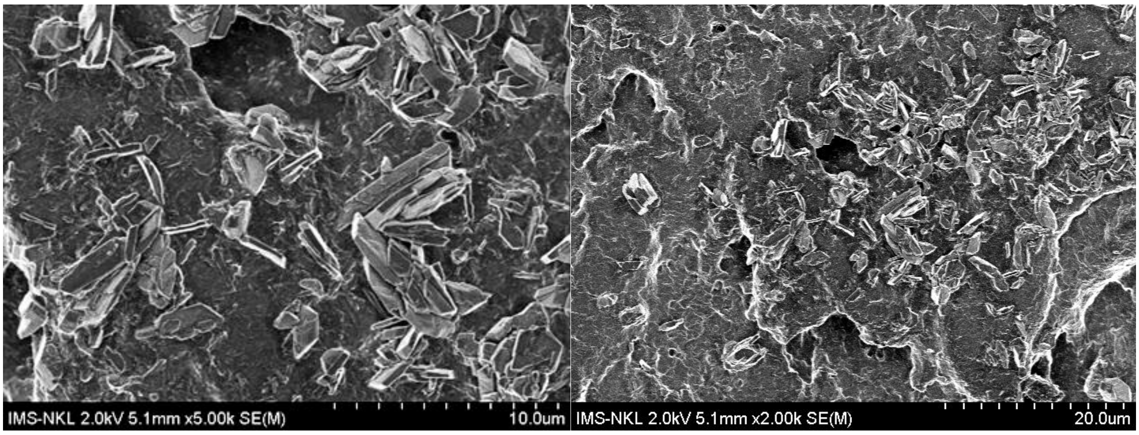
Furthermore, the pectin extracted from durian peels via the simple acidified hot water (AHW) method displays properties comparable to commercial pectin. This broadens the raw material sources for pectin production, reducing reliance on traditional sources like apple pulp and passion fruit peels. As showcased in this study, the pectin from durian peels meets the rigorous standards set for commercial pectin. It can be seamlessly integrated into numerous facets of food technology, such as jam production, stabilizing fruit juices, jelly manufacturing, and preserving canned fruits, to name a few.

This research underscores that durian peel (DP) holds significant potential as a source of pectin for various industry applications. By utilizing raw materials from by-products and waste, we can address the challenge of agricultural solid waste while enhancing the economic value of durian shells [61]. The AHW method, employed to extract pectin from DP, is considered one of the simplest and most cost-effective techniques, producing yields comparable to other methods. In conclusion, the study advocates that pectin production using the hot water acidification technique is a viable, economical, and effective alternative suited for industrial scaling.

In addition, with a simple extraction method, along with research data, a proposal for the pectin production system from pineapple peel specifically and agricultural waste, in particular, is presented, as shown in Fig.9. The production process consists of two main stages: raw material pre-treatment, extraction, and post-extraction. The simulation system in Fig.9 is intended for constructing and designing an industrial-scale production system. A simple, compact system with a small footprint is very feasible for application in industry. Furthermore, with a process design system like Fig.9, it can be observed that the initial investment costs are relatively high. This leads to the application of both small and medium-scale production.



(a) DP powder samples



(b) Pectin samples

Figure 8. Morphology by SEM. (a) SEM result of the raw material powder; (b) SEM result of the pectin extracted from durian peel powder

Figure 9. Simulation of pectin production system from durian peel

4. Conclusions

In this study, Durian peel was utilized to obtain pectin using the AHW method with four independent factors: temperature, time, pH, and SM ratio. The survey data on the factors influencing pectin yield revealed that three levels for each factor were chosen: 75-85 ℃ for extraction temperature, 45-75 minutes for extraction time, pH =1, and 17.5/1-22.5/1 mL/g for the S/M ratio. The response surface optimization method was employed to fine-tune these factors. Results indicated that the maximum pectin yield of 12.7475% was achieved at an extraction temperature of 80 ℃, an extraction time of 52 minutes, and an S/M ratio of 20.0/1 mL/g. The experimental findings correlated well with the predicted model. The pectin extracted under these optimal conditions had a moisture content of 7.16%, ash content below 3.0%, protein content of 0.43%, methoxyl content of 3.59%, esterification degree of 50.70%, equivalent weight of 617.80 g/mol, and AUA content of 40.25%. The preliminary results from this research suggest that durian peels, along with other agricultural wastes like fruit pulps, could serve as potential raw material sources for pectin extraction. However, it is necessary to evaluate and consider more comprehensively and in-depth applying pectin DP to food, pharmaceutical, and medical products in further works.

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Author Contributions

Conceptualization: Tran Y Doan Trang and Nguyen Quang Tung; Data curation: Tran Y Doan Trang, Pham Huong Quynh; Formal analysis: Tran Y Doan Trang and Ha Thi Dzung; Methodology: Tran Y Doan Trang and Do Thi Hanh; Software: Tran Y Doan Trang and Ta Thi Huong; Validation: Tran Y Doan Trang; Investigation: Tran Y Doan Trang and Ha Thi Dzung; Writing - original draft: Tran Y Doan Trang, Vu Phuong Lan and Ta Thi Huong; Writing - review & editing: Tran Y Doan Trang.

Data Availability Statement

Data will be provided in the article.

Ethical Approval

All authors declare that this study does not involve animal and human subjects.

Conflicts of Interest

The authors declare no conflict of interest.

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