Optimization of Ultrasound-Assisted Pectin Extraction from Durian Rind

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Durian (*Durio zibethinus*) rind is fruit waste and can be used as a source of pectin. This study aimed to optimize the pectin extraction process using the ultrasound-assisted extraction (UAE) method and evaluate its physicochemical properties. Initially, influencing factors in UAE, such as solvent type (HCl, citric acid, acetic acid, and n-hexane), extraction time (10, 15, 20, 25, and 30 minutes), extraction temperature $(40, 50, 60, 70$, and $80^{\circ}C)$, sample:solvent ratio $(1:14, 1:17, 1:20, 1:23$, and $1:26$ g/mL), duty of cycle $(20, 40, 60, 80$, and $100\%)$, and amplitude (20, 40, 60, 80, and 100%) were evaluated. Using central composite design (CCD), a temperature of $74^{\circ}C$ and sample:solvent ratio of 1 : 20 g/mL were determined as optimum conditions with an estimated pectin yield of 6.07%. This value is in accordance with the experimental result of 6.12%. Extraction with UAE resulted in a higher pectin yield compared to the conventional extraction method using a water bath shaker. The pectin had a degree of esterification (DE) of 44.35%, a moisture content of 4.34%, and an ash content of 1.08%. The FTIR spectra proved the presence of functional groups in pectin, the XRD patterns suggested the structure was more amorphous than crystalline, and SEM showed a smooth pectin surface.

Keywords: Central composite design, degree of esterification, durian rind, pectin extraction, physicochemical properties, ultrasound-assisted extraction

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1. Introduction

Durian (*Durio zibethinus*) is a popular fruit in Southeast Asian countries such as Thailand, Malaysia, Indonesia, and the Philippines, the leading suppliers that account for 95% of world durian exports in 2021 [1]. Only a third of the durian fruit is edible; the rest, such as seeds and rinds, are considered waste. The total weight of durian fruit consists of about $20-35\%$ pulp, $5-15\%$ seed, and the remaining rind of the fruit, which weighs up to $65-70\%$ [2]. With durian production from Indonesia reaching around 1.710 .000 million tons in 2022 [3], the amount of durian rind waste in Indonesia is estimated to be around 1.1 million tons in 2022. The common practice for handling durian rind waste is throwing it into landfills or burning it, which can cause environmental pollution if it is not appropriately managed [4]. Durian rind is rich in pectin as much as 2.1-10.25% [4, 5], so this waste can be utilized as raw material for pectin production and at the same time can reduce waste disposal problems.

Pectin is a common ingredient that is widely used in food industry as a food additive [6, 7], in pharmaceutical industry [8], and for biomedical applications [9]. The properties of pectin, such as nontoxicity, emulsion behavior, diverse chemical composition, biocompatibility, and high stability, make pectin a commonly used biopolymer in food, pharmaceutical, and biomedical industries. One important parameter that determines the quality of pectin is the degree of esterification. Based on the DE, pectin is divided into two types, namely high methoxyl pectin ($DE > 50\%$) and low methoxyl pectin ($DE < 50\%$) [10]. High methoxyl

pectin can form a gel in acidic conditions (pH 2.0 - 3.5) with high sugar content ($>$ 55%), while low methoxyl pectin (DE $<$ 50%) can form a gel in the pH range (pH $2.0 - 6.0$) without the need of added sugar but with the addition of a small amount of calcium ions. Pectin consists of a linear backbone of 1.4-linked-galacturonic acid that is mainly esterified with methyl and has small amounts of rhamnose in the main chain and arabinose, galactose, and xylose in the side chains [11].

Typically, pectin is extracted by thermal heating in an acidic medium (such as sulfuric acid). For example, extracting pectin from durian rind using H_2SO_4 with a water bath shaker takes 4-5 hours and a temperature of $75 - 95^{\circ}$C [2]. However, this method is inefficient due to the long extraction time [12] and the high amount of wastewater generated [13]. Therefore, a more efficient method is required for extracting pectin from durian rind. Ultrasound-assisted extraction (UAE) offers lower temperatures, shorter extraction times, and higher energy efficiency than the shaking water bath method [14]. This method has been used to extract pectin from eggplant peel [6], finger citron pomace [15], and Kinnow (*Citrus reticulata*) peel [16]. Scarce information was found in literature regarding the extraction of pectin from durian rind using UAE. Extraction of pectin from durian rind has been studied where the extraction was carried out either in a water bath shaker [2] or a stirring chamber [17]. With this background, this study aimed to optimize the pectin extraction process from durian rind using the UAE method. Factors that influence the extraction results are solvent type, time, temperature, sample:solvent ratio, cycle duty, and amplitude, and were evaluated to optimize the UAE factors in pectin production from durian rind using central composite design (CCD) and examined its physicochemical properties.

2. Materials and methods

2.1. Materials

Durian rind was obtained from a local durian shop in Yogyakarta City, Yogyakarta Special Province, Indonesia. The parts of durian rind used in this research were the mesocarp and endocarp (Fig. 1). Durian rind was washed and dried at 50°C until the moisture content was $<$ 10%. The sample particle size used was 0.6 mm. Dried durian rind samples were stored in dry conditions before use. Other materials used for the extraction process were hydrochloric acid (Mallinckrodt, USA), citric acid (Merk, Germany), acetic acid (Merk, Germany), n-hexane (Merk, Germany), sodium hydroxide (Merk, Germany), phenolphthalein indicator (Merk, Germany).

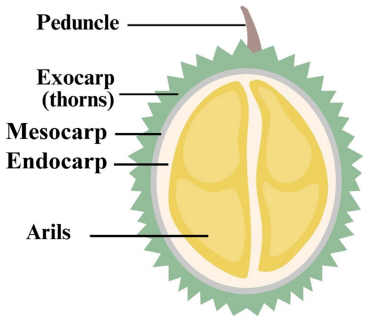

Fig. 1. Cross section of durian rind.

2.2. Extraction of pectin from durian rind

2.2.1. Non-factorial completely randomized design

Initially, the factors that affect the extraction results were selected based on the solvent type, extraction time, extraction temperature, sample:solvent ratio, duty of cycle, and amplitude. The first screening stage was selecting the type of solvent. There were four solvents evaluated, namely HCl, citric acid, acetic acid and n-hexane. After the type of solvent was selected, screening was carried out on to the next factor, namely extraction time. The screening was carried out by determining five extraction time levels $(10, 15, 20, 25,$ and 30 minutes) at a temperature of 70°C, duty of cycle 100%, amplitude 100%, and sample:solvent ratio $1 : 20$ (g/mL). The next screening factor was extraction temperature by determining five levels of temperature conditions $(40, 50, 60, 70, 80^{\circ}C)$. Screening for the next factor was the sample:solvent ratio. The screening was also carried out by determining five levels of sample:solvent ratio, namely $1 : 14, 1 : 17, 1 : 20, 1 : 23,$ and $1 : 26$ (g/mL). After that, duty of cycle was screened by determining five levels, namely $20, 40, 60, 80$ and 100%. The final screening factor was amplitude, which was carried out by determining five levels, namely at 20, 40, 60, 80 and 100%. Optimization experiments with response surface methodology were then determined based on these value ranges.

2.2.2. Experimental design

The factors and levels in this study are shown in Table 1. The most significant independent variable was selected for optimization. Central composite design (CCD) for yield optimization consisted of 13 treatment combinations, with each factor having a lower limit (1), midpoint (0), upper limit $(+1)$, and starting point (α), with the experimental matrix design as shown in Table 2. Repetition of the analysis for each response was carried out three times.

The two factors selected were extraction temperature and sample:solvent ratio. The main effects, interactions, and quadratic terms were then included in the response surface methodology to obtain the following equation.

$$Y = \beta_0 + \beta_i A + \beta_i B + \beta_{ij} AB + \beta_{ii} A^2 + \beta_{ii} B^2 \quad (1)$$

Where Y is the dependent variable, β_0 is a constant, β_i represents the linear regression coefficient, β_{ij} is the interaction coefficient, and β_{ii} represents the quadratic regression coefficient. A and B are independent variables.

2.2.3. Ultrasound-assisted extraction (UAE)

Durian rind was extracted using an ultrasonic system: probe diameter 7 mm with frequency 26 kHz, and power 200 W, UP200St (Hielscher Ultrasonics GmbH, Teltow, Germany). The temperature was controlled using a water bath (J.P. Selecta, Barcelona, Spain). For UAE experiments, 10 g of dried durian rind powder was used for each process. The experiments were carried out according to the procedure described by Yu et al. [15] with modifications. The filter material used was a filter cloth placed in a Buchner funnel. The filtrate was filtered again using a vacuum pump with filter material in the form of filter paper. The pectin was then precipitated using 96% ethanol with a ratio of filtrate and 96% ethanol 1:1 (v/v) for 24 hours. The pectin was washed using 30 mL of 96% ethanol with three repetitions. This study also used a conventional method using a water bath shaker with the best conditions obtained in the UAE. Pectin extracted by a water bath shaker method was used as a comparison in pectin characterization.

2.3. Analysis

2.3.1. Extraction yield of pectin

After the pectin was precipitated and washed, the precipitated pectin was dried in an oven at 50°C until the weight was stable. The pectin yield was calculated using the following equation:

$$\text{Yield } (\%) = \frac{\text{dry pectin yield weight (g)}}{\text{durian rind powder weight (g)}} \times 100\% \quad (2)$$

2.3.2. Degree of esterification (DE)

The DE was determined using a titrimetric method described by Kazemi et al. [6]. Briefly, 0.2 g of dry pectin was mixed with 2 mL of ethanol and 100 mL of distilled water. After that, stirring was carried out to dissolve the pectin using a stirrer at 1000 rpm for 20 minutes. Then, five drops of phenolphthalein indicator were added and titrated with 0.5 M NaOH until a pink color was formed. The titration volume was recorded as V1. Next, 10 mL of 0.5 M NaOH was added to the solution and stirred using a stirrer at a speed of 500 rpm for 1 minute. After that, the solution was left for 15 minutes. After 15 minutes, 10 mL of 0.5 M HCl was added and stirred until the pink color disappeared. Five (5) drops of phenolphthalein indicator were added and titrated using 0.5 M NaOH to form a pink color [18]. The titration volume was then recorded as V2. The DE equation is as follows:

$$\text{DE } (\%) = \frac{V2}{V2 + V1} X100 \quad (3)$$

2.3.3. Moisture, ash, and color analysis

Analysis of moisture and ash content in pectin followed the AOAC 2005 method [19]. Color analysis was carried out using a Chroma meter (KONICA MINOLTA, Japan). The color parameters measured were L^* (lightness), a^* (redness), and b^* (yellowness). The whiteness index (WI) was calculated by Eq. (4) [20]:

$$\text{WI } (\%) = 100 - \sqrt{(100 - L*)^2 + a*^2 + b*^2} \quad (4)$$

2.3.4. Fourier-transformation infrared (FTIR) spectroscopy.

The content of functional groups in the pectin was analyzed using an FTIR spectrometer (Spectrum Two, Perkin Elmer, USA). Analysis was carried out at the integrated laboratory of the Islamic University of Indonesia, Yogyakarta, Indonesia. Analysis and instrument conditions were set at wavenumbers ranging from 450 to 4000 cm^{-1} in attenuated total reflection (ATR) mode with a resolution of 4 cm^{-1} with 64 scans. The peak areas for the esterified carboxyl $(C = O)$ peak between 1730 cm^{-1} and 1720 cm^{-1} and the unesterified carboxyl (COO^-) peak between 1630 cm^{-1} and 1600 cm^{-1} were obtained using instrument software (Spectrum, Version 10.4, UK) [21].

2.3.5. X-ray diffraction

X-ray diffraction patterns of pectin samples were recorded using an X-ray diffractometer (D2 Phaser BRUKER, Germany). The analysis was conducted at the integrated laboratory of the Islamic University of Indonesia, Yogyakarta, Indonesia. The analysis and instrument conditions were set at diffraction angles (2θ) from 10°C to 80°C with a step size and time rate of 0.05° and 4% min, respectively [22]. The total relative crystallinity (RC) of the samples was calculated by Eq. (5) [23]:

$$\text{RC } (\%) = (Ac/At) \times 100. \quad (5)$$

where Ac is the area of the crystalline peak and At is the total area under the curve.

Table 1. Factor and independent variable levels

Factor	$-\alpha$	-1	0	1	$+\alpha$	Unit
Extraction temperature	55.85	60	70	80	84.14	°C
Sample: solvent ratio	5.75	1 : 17	1 : 20	1 : 23	24.24	g/mL

Table 2. Central composite design experiment

Run	Factor		Yield (%)		
	A (extraction temperature)	B (sample: solvent ratio)	Exp.	Pred.	Error
1	-1	-1	3.3	3.55	7.04
2	0	0	5.95	6.07	1.98
3	1	1	6.1	5.45	11.93
4	1	-1	4.33	4.15	4.34
5	$+\alpha$	0	4.56	5.06	9.88
6	0	0	6.3	6.07	3.79
7	-1	1	4	3.78	5.82
8	0	0	5.84	6.07	3.79
9	0	$-\alpha$	3.81	3.67	3.81
10	0	$+\alpha$	4.22	4.75	11.16
11	0	0	6.51	6.07	7.25
12	$-\alpha$	0	3.55	3.45	2.90
13	0	0	5.76	6.07	5.11

2.3.6. *Scanning electron microscope*

The morphology of the pectin samples was observed using a scanning electron microscope system (JSM-6510LA, JEOL, Japan). Analysis was carried out at the integrated research and testing laboratory of Universitas Gadjah Mada, Yogyakarta, Indonesia. The analysis and instrument conditions were set at an accelerating voltage of 15 kV. Under vacuum conditions, samples were sprayed with a thin layer of gold, and images were recorded at 500x magnification [22].

2.3.7. *Statistical analysis*

Data were analyzed using SPSS (version 26.0, IBM, SPSS Inc). Analysis of variance (ANOVA) to determine significant differences ($p < 0.05$). Design-expert 13 software was used for experimental design, data analysis, and modeling using CCD simultaneous with Response Surface Methodology

3. Result and discussion

3.1. Screening of independent variables for pectin extraction

3.1.1. *Evaluation of solvent type on pectin yield*

According to Fig. 2, HCl and citric acid were successfully extracting pectin, whereas acetic acid and n-hexane were not suitable for extracting pectin from durian rind. Acid solvents were preferable to hydrolyze water-insoluble protopectin into water-soluble pectin [24]. The results showed that HCl had a higher extraction capacity than citric acid. The yield of pectin extracted using HCl was significantly higher ($P < 0.05$) than the yield of pectin extracted using citric acid at 6.03% and 4.59%, respectively. According to previous studies, inorganic acid (HCl) was more effective than acetic acid and citric acid for pectin extraction of banana peels [25] and lime peels [26]. The reason for this could be that HCl is able to release H^+ ions quickly, causing protopectin degradation and the release of galacturonan chains [9].

Compared with strong mineral acids (HCl), organic acids such as citric and acetic acid have lower dissociation constants, resulting in lower hydrolysis capacity and lower extraction efficiency [27]. Despite the fact that both citric acid and acetic acid are organic acids, citric acid was more effective than acetic acid in extracting pectin from potato pulp [28]. Even though those two acid solvents were classified as carboxylic acid, only citric acid was able to extract the pectin compound. This might happen because citric acid has three carboxylic acid groups, while acetic acid has one group. The dissociation constant of citric acid was higher than acetic acid.

3.1.2. *Evaluation of extraction time on pectin yield*

Based on Fig. 3, the highest yield was achieved when extraction was conducted in 30 minutes. The longer the extraction time, the pectin yield increased significantly ($P < 0.05$) from 4.08 to 5.90% because solvent diffusion into tissue cells also increased [29]. The longer the extraction, the

longer the contact time of the solvent with pectin, and the longer it takes for the ultrasonic waves to disrupt the cell walls so that more pectin is extracted [30]. The previous report showed that extraction of pectin from durian rind using a maceration method took 5 hours to obtain a pectin yield of only 4.07% [31]. Meanwhile, extraction using the UAE method in this work produced a higher pectin yield (6.02%). The reason may be due to the acoustic cavitation effect, which is able to break the cell wall of the matrix, and the solvent can easily extract the target pectin [22]. Therefore, mass transfer in UAE occurs in a short time [32, 33].

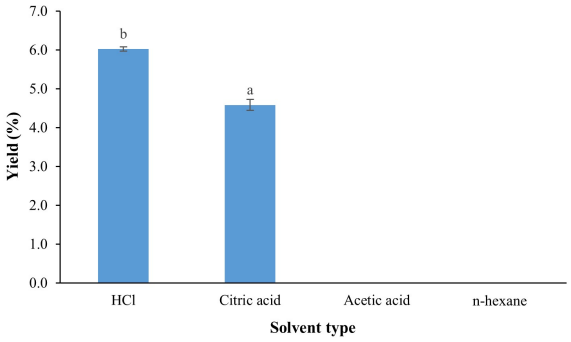

Fig. 2. Effect of different types of solvents on pectin yield. Different letters (a-b) indicate significant differences in each treatment ($p < 0.05$).

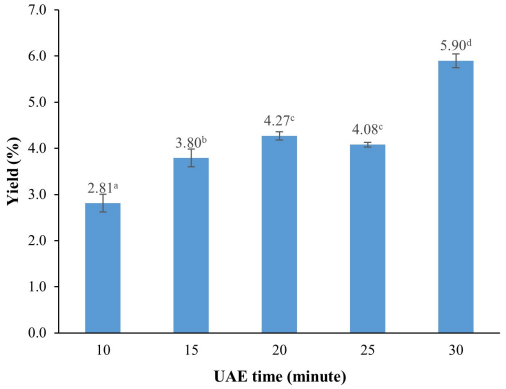

Fig. 3. Effect of different extraction times on pectin. Different letters (a-d) indicate significant differences in each treatment ($p < 0.05$).

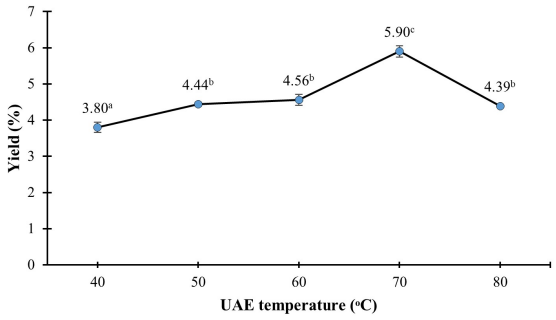

Fig. 4. Effect of temperature differences of $60 - 80°C$ on pectin yield. Mean value $\pm$ SD, n = 3. Compact letter display based on Duncan's test ($p < 0.05$).

3.1.3. *Evaluation of temperature on pectin yield*

Fig. 4 shows that the pectin yield increased with increasing extraction temperature up to 70°C. This is due to the disruption of plant cells at higher temperatures and the rapid separation and breakdown of pectin up to a certain point [34]. More significant decomposition of plant cells occurs at higher temperatures, thereby releasing pectin from plant tissue to produce more pectin [15]. Perina et al. [35] also reported that the solubility and diffusion coefficient will increase with increasing temperature and result in a high extraction rate. However, a further increase in temperature to 80°C showed a significant decrease in pectin yield. This is in accordance with previous findings by Ke et al. [36]; the yield of pectin extracted from chayote (*Sechium edule*) using UAE increased when the temperature increased from 60°C to 70°C and decreased when the temperature exceeded 70°C.

The increase in pectin yield significantly ($P < 0.05$) from 4.56 to 5.90% as temperature increased was caused by an improvement in the swelling of the durian skin cell walls, which results in disruption of the plant cell walls, increasing the rate of solvent penetration and increasing the diffusivity, solubility and release of pectin into in the medium [37]. However, further increasing the temperature can decrease the yield due to degradation of the pectin chains and result in a lack of viscosity and surface tension of the solvent, thereby inhibiting mass transfer [22].

3.1.4. *Evaluation of sample:solvent ratio on pectin yield*

Evaluation of the sample: solvent ratio to pectin yield in Fig. 5 shows that the pectin yield increased as the sample:solvent ratio increased to 1 : 20 g/mL. The significant increase in the yield ($P < 0.05$) from 4.36 to 6.12% was caused by the increasing contact area between the

analyte and the solvent, which makes the polysaccharide completely dissolved and extracted [38]. Kumar et al. [39] reported that the higher the sample:solvent ratio, the higher the fragmentation, erosion, and pore formation, causing an increase in yield. However, higher sample:solvent ratios exceeding 1:20 g/mL led to a decrease in pectin yield. This is caused by the slow rate of molecular diffusion, the high viscosity of the raw material, and the small amount of extraction solution [40]. In addition, a decrease in the ultrasonic energy density distribution in the extraction solution, thereby inhibiting the dissolution of polysaccharides can also occur at high sample:solvent ratios [41]. Therefore, 1 : 20 g/mL was the most preferred for extracting pectin from durian rind in this work.

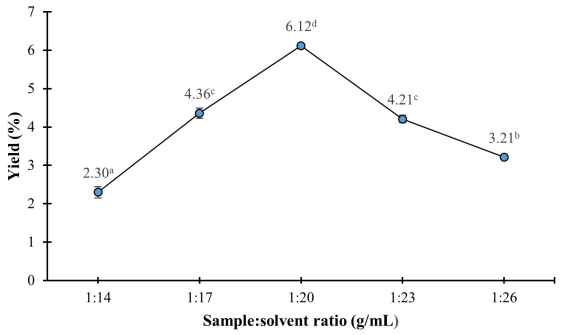

Fig. 5. Effect of sample:solvent ratio on pectin. Compact letter display based on Duncan's test ($p < 0.05$).

3.1.5. *Evaluation of duty of cycle on pectin yield*

Duty of cycle is the release of cavitation bubbles in a unit of time through an elastic medium. As seen in Fig. 6, the higher the duty of cycle applied, the higher the pectin yield obtained. With a duty of cycle ranging from 33% to 50%, the pectin yield from grapefruit peel increased and then the yield decreased in the duty of cycle ranging from 50% to 70% [40]. The increase in yield with the application of a higher duty of cycle was significant ($P < 0.05$), resulting in faster cavitation formation. Cavitation produces a rapidly moving flow through cavities in the surface at the liquid-solid interface. High cavitation produces high-intensity ultrasonic waves that come into contact with the raw material more frequently. Meanwhile, a low duty of cycle does not provide a significant yield because the cavitation effect is not enough to disturb the material and release pectin [40]. The extraction mechanism is based on cavitation bubbles that form in the rarefaction phase and decrease in size in the compression phase [42]. Therefore, a 100% duty of cycle was the most favorable for extracting pectin from durian rind.

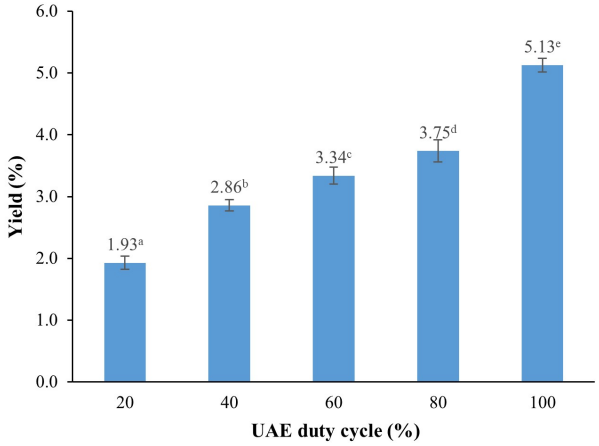

Fig. 6. Effect of duty of cycle on pectin yield. Different letters (a-e) indicate significant differences in each treatment ($p < 0.05$).

3.1.6. *Evaluation of amplitude on pectin yield*

Fig. 7 shows that the higher the amplitude applied to the extraction process, the higher the yield of extracted pectin. The yield value decreased at an amplitude of 40% and then increased slightly at an amplitude of 80%. A further increase to 100% caused a significant increase, namely two times that of the previous treatment. Therefore, 100% amplitude was the most favorable for extracting pectin from durian rind. Amplitude had a favorable linear impact on the pectin yield, according to optimization of the UAE conditions [43]. Pectin from *Malus domestica* 'Fălticeni' apple pomace was extracted using UAE and it yielded 9.183% pectin at 100% amplitude [44]. The results obtained were in accordance with a previous study where there was an increase in the pectin yield value from grapefruit peel from 22.67% to 27.27%, which increased significantly ($P < 0.05$) from an amplitude of 30% to 60%. Since the size of the resonant bubble is related to the amplitude of the ultrasonic pulse, this can be explained by the fact that the collapse of the cavitation bubble becomes more severe with increasing amplitude [45].

Increasing the amplitude will increase the cavitation energy, solvent diffusion into the material, and mass transfer rate. This is caused by the rupture of cavitation bubbles near the cell wall, then a shock wave and liquid jet are

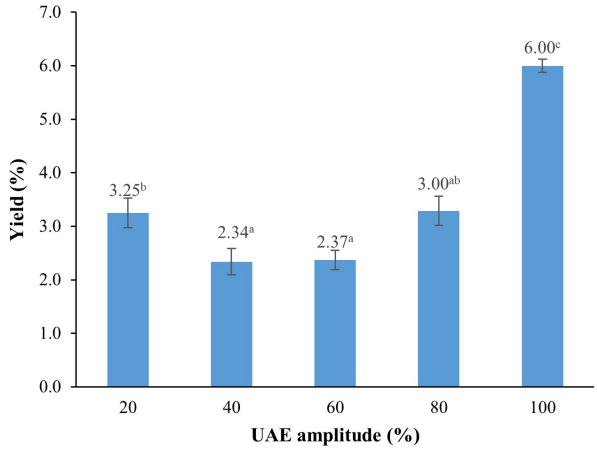

Fig. 7. Effect of amplitude on pectin yield. Different letters (a-c) indicate significant differences in each treatment ($p < 0.05$).

formed, which causes the cell wall to rupture. Therefore, the targeted analytes in cells can be extracted and mixed in solution [46].

3.2. Analysis of response surface optimization

The results of thirteen experiments carried out on two independent variables (temperature and sample:solvent ratio) of pectin extraction and the response to the dependent variable (yield) are presented in Table 2. ANOVA data analysis is linear parameters A (temperature), B (sample:solvent ratio), all quadratic parameters $\left(A^2,\ B^2\right)$, as well as the interaction between extraction temperature and sample:solvent ratio (AB) had a significant influence on the yield (Table 3).

This model also provided the interaction effect of the two variables as well as the quadratic effect of each variable. The higher the temperature and the sample:solvent ratio, the higher the pectin yield. The interaction of extraction temperature with the sample:solvent ratio (AB) had a synergistic effect; this can be seen from the coefficient on the AB interaction that had a positive interaction value with a coefficient of 0.2675 . Significant yield changes are often accompanied by temperature-sample:solvent ratio interactions similar to those reported for jackfruit rags [47] and *Citrus aurantium* [37]. The effect of the interaction of the two variables, namely the independent variable (temperature and sample:solvent ratio) and the dependent variable (yield), can be represented by an equation in the factor code, as shown in Eq. (6).

$$Y = 6.07 + 0.5698\ A + 0.3812\ B + 0.2675AB - 0.9091\ A^2 - 0.9291\ B^2 \quad (6)$$

where:

Y = Yield (%)

A = Temperature (°C)

B = Sample:solvent ratio (g/mL)

A lack of fit test was performed to check whether the selected model adequately describes the observed data or whether a more complex model was needed. Statistical analysis showed that the p-value was less than fit to provide satisfactory results (p-value > 0.05). The lack of insignificant fit value indicates that the selected model is the right model [48]. Moreover, the quadratic model of yield had a coefficient of determination $\left(R^2\right)$ of 0.9040 , which means that the model can be used to predict the response for optimization.

The goal of optimization is to minimize the effort or operations required and maximize the desired results [49]. The optimization process was carried out using the Design Expert-13 application so that the desired response was obtained. Optimization was carried out after obtaining a mathematical response model. The desirability value closest to 1 indicates a good correlation between the optimal optimization process and the desired response variable [48]. Based on the response surface methodology graph (Fig. 8), the optimum UAE conditions to achieve the highest pectin yield (6.07%) from durian rind and the desirability value, extraction was set at a temperature of 74°C and a sample:solvent ratio of 1 : 20 g/mL. The verification response of the pectin yield obtained was in accordance with the predicted value, which was 6.12%.

3.3. Physicochemical properties of pectin from durian rinds

The optimum extraction conditions were used to compare the pectin yield obtained from the UAE method and the conventional method using a water bath shaker. The pectin obtained using UAE was two times higher (6.12%) than using the shaking water bath method (3.66%). Pectin characterization includes DE, water content, ash content, and color (Table 4).

3.3.1. Degree of esterification

The DE is the percentage of acid that reacts with alcohol to form esters. Suwoto et al. [51] reported that the DE determines the properties of pectin, especially solubility and gel formation, as well as the application of pectin. The results showed that there was no significant difference between DE pectin produced by the UAE and the shaking water

Table 3. ANOVA for UAE of pectin from durian rind using RSM

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	14.45	5	2.89	13.18	0.0019	significant
A-temperature	2.60	1	2.60	11.85	0.0108	
B-sample: solvent ratio	1.16	1	1.16	5.30	0.0547	
AB	0.2862	1	0.2862	1.31	0.2907	
A^2	5.75	1	5.75	26.23	0.0014	
B^2	6.01	1	6.01	27.40	0.0012	
Residual	1.53	7	0.2192			
Lack of Fit	1.12	3	0.3749	3.66	0.1212	not significant
Pure Error	0.4099	4	0.1025			
Cor Total	15.98	12				

Table 4. The yield, DE, moisture content, ash content, and color of pectin from the UAE and the shaking water bath method.

Properties	Extraction		Standard (IPPA and FCC) [50]
	UAE method	shaking water bath method	
Pectin yield (%)	6.12 ± 0.09^b	3.66 ± 0.13^a	-
Degree of esterification (%)	44.35 ± 0.08^a	48.56 ± 0.08^a	Max. 50%
Moisture content (%)	4.34 ± 0.22^a	7.39 ± 0.15^b	$< 12\%$
Ash content (%)	1.08 ± 0.05^a	0.94 ± 0.04^b	$< 10\%$
Color			
L*(lightness)	44.38 ± 0.55^a	46.39 ± 0.15^a	-
a* (redness)	7.97 ± 0.35^a	7.37 ± 0.06^a	-
b*(yellowness)	15.36 ± 0.17^a	18.86 ± 0.01^a	-
WI (whiteness index (%))	42.35 ± 0.19^a	42.77 ± 0.07^a	-

Data are expressed as mean value $\pm$ standard deviation $(n = 3)$. Values with different superscripts in the same row are significantly different $(p < 0.05)$. IPPA (International Pectin Producers Association). FCC (Food Chemical Codex).

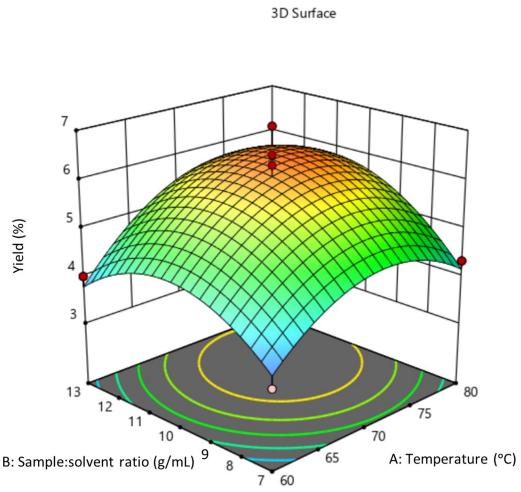

Fig. 8. Response surface plot displaying the effect of UAE factors on pectin yield.

bath method, namely 44.35% and 48.56%. The DE values of both extraction methods are in accordance with the results of Wai et al. [52]. However, Jong et al. [2] reported that the DE value of pectin extracted from durian rind using the shaking water bath method was 18.99%. The difference in results is likely caused by differences in DE measurement methods using the titration method and FTIR method [53]. The DE value obtained in this study was classified as low methoxyl pectin (LMP), meaning that it can form a gel in the pH range $(\text{pH } 2 - 6)$ without requiring additional sugar in the presence of small amounts of calcium [53]. All pectin obtained from both methods was LMP with DE of $< 50\%$. This shows that durian rind pectin can be used as a gelling agent and stabilizer in the production of low-calorie foods and beverages.

3.3.2. Moisture content, ash content, and color analysis results

The moisture content of pectin needs to be known for safe storage because it affects the quality of the pectin [54]. Oloye et al. [55] reported that an increase in moisture content is a sign of spoilage because it stimulates bacterial growth that can reduce the quality of pectin due to the activity of the pectinase enzyme. Based on Table 4, the moisture content obtained from the UAE method and shaking water bath method ranged between 4.34-7.39%. This value meets the International Pectin Producers Association

(IPPA) and Food Chemical Codex (FCC) quality standards (below 12%).

Ash content is an inorganic substance left over from burning organic material. The ash content indicates whether inorganic components remain in the pectin after burning or are still present in it. Furthermore, the ash content affects the level of pectin purity. Aziz et al. [56] reported that the lower the ash content in pectin, the higher the purity level. This study shows that the ash content obtained from the two methods ranged from $0.94 - 1.08\%$, which meets the pectin standards according to IPPA and FCC (below 10%). Based on DE value, moisture and ash content, UAE and shaking water bath pectin meet IPPA and FCC quality standards.

Another essential characteristic of pectin is color because it influences the appearance and consumer acceptability of the gel formed and the final food product containing pectin [22] or other water-soluble pigments trapped in pectin during settling [57]. UAE pectin and pectin obtained from shaking water bath extraction have brightness and whiteness indices that were not significantly different ($P > 0.05$). From these results, it can be concluded that the WI obtained was 42% and had a dark brightness level. In the study of Panwar et al. [22], it was reported that light pectin had a WI of more than 50%.

3.3.3. *Fourier transform infrared spectroscopy (FTIR)*

FTIR analysis was carried out to identify the functional groups and verify the pectin produced. Based on the spectra, several peaks corresponding to certain functional groups were observed at different intensities in both pectin samples. The transmission pattern of UAE pectin was similar to the spectrum of pectin obtained from the shaking water bath method. The wavelengths shown in Fig. 9 and Table 5 follow the standard range of absorption bands in pectin, which confirms that the resulting compound was pectin. Relevant functional groups in pectin, such as hydroxyl ($O - H$), methyl ($-CH3$), carbonyl ($-C = O$), and ether ($R - O - R$), were observed in both pectins produced by UAE and shaking water bath methods.

3.3.4. *X-ray diffraction XRD and Scanning electron microscopy (SEM)*

The X-ray diffraction (XRD) patterns of the two pectin samples (Fig. 10) were analyzed to identify the structural characteristics of pectin (crystalline and amorphous). Crystalline materials have several narrow and sharp peaks in the diffraction peaks [59], while amorphous materials have broad peaks [47].

The XRD pattern obtained from UAE pectin showed that the amorphous part was larger than the crystalline, indicated by the RC value, which was 38.68%. It was shown by the peak areas at 2θ (theta) ($^\circ$), namely $13.5^\circ, 18.1^\circ, 20.9^\circ$ and 30.4°. Meanwhile, the XRD pattern obtained from pectin extracted using the shaking water bath method had a more crystalline structure because it had sharp and strong signals that appeared at $13.6^\circ, 16.7^\circ, 16.9^\circ, 17.2^\circ, 18.1^\circ$, and 20.7°. The RC value of pectin obtained from the shaking water bath method was 58.52%. The XRD patterns of pectin obtained from both methods showed a similarity in the XRD pattern of pectin from walnut waste [60]. The structure of pectin seems to be influenced by the origin of the raw material and the extraction method. For example, pectin extracted from mangosteen peel was amorphous [61], but pectin extracted from *Citrus limetta* peels using the UAE method had a high crystalline portion [22].

Scanning electron microscopy (SEM) was carried out to

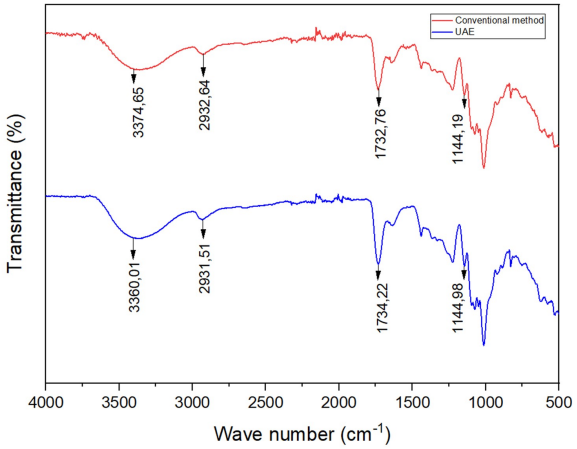

Fig. 9. FTIR spectra of pectin extracted from durian rind by shaking water bath method and UAE.

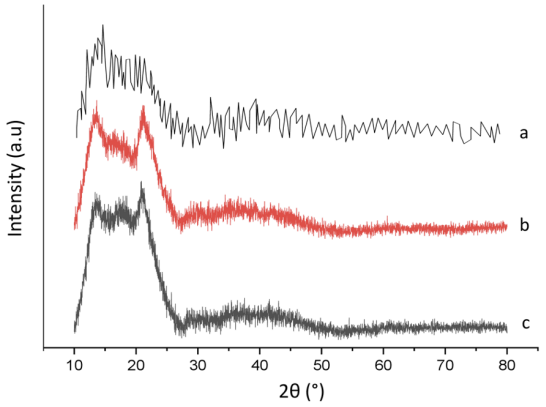

Fig. 10. XRD patterns of pectin extracted from durian rind: (a) literature [60] (b) shaking water bath method (c) and UAE method.

Table 5. FTIR analysis of pectin using UAE and shaking water bath methods.

No	Wavelength number (cm^{-1})		Reference [58] (cm^{-1})	Reference [47] (cm^{-1})	Bond
	Shaking water bath method	UAE method			
1	3374.65	3360.01	3345.6	3457	Hydroxyl $(O-H)$
2	2932.64	2931.51	2931	$2942-2892$	Methyl $(-CH_3)$
3	1732.76	1734.22	1732.19	$1735-1731$	Carbonyl $(-C=O)$
4	1144.19	1144.98	1144.81	-	Ether (R-O-R)

characterize the surface morphology of UAE pectin and pectin obtained from the shaking water bath method by visualizing their structure and morphology. Fig. 11 presents SEM images of the pectin obtained by the UAE method and shaking water bath method, respectively. SEM photos were the results of 100x magnification and 10.000x magnification.

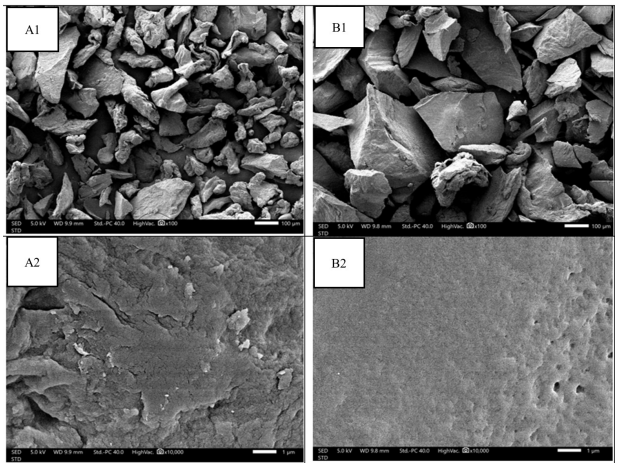

Fig. 11. SEM of pectin extracted from durian rind using shaking water bath method (A) and UAE method (B) at x100 magnification (A1;B1) and x10.000 magnification (A2;B2)

Based on the results in Fig. 11 (A1 and B1), it can be seen that the surface of UAE pectin was more uniform compared to the surface of pectin obtained from the shaking water bath method at a magnification of x100. At a magnification of x10,000, Fig. 11 A2 and B2 show that the surface of UAE pectin was smoother than the surface of pectin extracted by the shaking water bath method. The SEM results in this study show a morphological similarity to pectin from pomelo peels [62].

4. Conclusions

Ultrasound-assisted extraction was successfully used to produce pectin from durian rind using HCl as the solvent. Ultrasound-assisted extraction was proven to be an effective and efficient method for extracting pectin from durian rind. The pectin yield obtained in the extraction process using UAE was higher compared to extraction using the shaking water bath method. The optimal extraction conditions of UAE were at 70°C and 30 minutes. The pectin characteristics in terms of DE, water content, and ash content met the IPPA and FCC standards. From the results of this work, it is interesting to carry out further studies regarding the optimization of UAE to obtain the desired pectin properties.

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