



Extraction of Pectin from Orange Residue: Optimization Studies using Microwave-assisted Method

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Abstract: Pectin, a key hydrocolloid is widely used in various food systems because of its ability to emulsify and thicken. The disposal of fruit waste, like peels and pulp, creates environmental issues, but these materials can also be a source of pectin. Although orange peels has been used for pectin production, however, there is need to improve on the extracting parameters to fully maximize these residues; not just the peel but also the pulp and find the best extraction conditions. This study use microwave-assisted method for extraction while Response Surface Methodology (RSM)- Box-Behnken design software was used for the optimization process. This study examined the impact of three factors: pH (1.0, 2.0, and 3.0), microwave power (300, 450, and 600 W), and irradiation time (3, 5.5, and 8 min) on pectin yield. The pectin yield ranged from 6.12% to 15.37%. The best conditions found were; a pH of 2.79, a microwave power of 389.90 W, and an irradiation time of 5.74 min, which achieves a pectin yield of 14.05% with a desirability value of 1.0. The results showed that all three factors- pH, power, and time-had a significant effect on the yield. Higher microwave power and longer irradiation time increased the yield, while higher pH levels reduced it. However, too long irradiation time actual lowered the yield. This method demonstrates how agricultural waste, such as orange residue (peel and pulp), can be transformed into useful products like pectin.

1. Introduction

Citrus fruits are part of the Rutaceae's family grown globally in a large-scale because of their unique taste, smell, and flavor, as well as their health benefits (Suri *et al.*, 2021). Among all the types of citrus fruits, oranges is one of the most cultivated by farmers. They are often used in making juice, drinks, syrups, and other products. The food industry uses about 40% of citrus fruits for juice processing, generating significant waste including seeds, peels, and pulp (Dubey *et al.*, 2023; Odeh *et al.*, 2014). This waste, however, is actually a valuable resource that can be used to make pectin. Pectin is mainly found in the cell walls of plants, made of α -galacturonic acid units, with some methyl ester groups attached. Because of its many useful properties, pectin plays a major role in food systems, helping make food thicker, giving it a better texture, and excellent mouth feel. It's a fat substitute in foods like spreads, ice cream, and processed meat products (Liu *et al.*, 2006; Saidi *et al.*, 2022). There is an ongoing effort to find more efficient methods for producing pectin from these waste materials in less time and with higher quality (Doe and Smith, 2023).

In recent years, novel technologies—including microwave and ohmic heating, moderate and pulsed electric fields, high-pressure processing, and ultrasound—have been investigated as alternative approaches for pectin extraction (Gavahian *et al.*, 2021). Among the emerging techniques, microwave-assisted extraction (MAE) demonstrates significant advantages over conventional water bath methods. It enables markedly lesser extraction times, reduces solvent consumption, achieves higher yields, and produces superior-quality pectin at lower cost. Whereas traditional approaches often require several hours, sometimes exceeding 60 min. MAE can accomplish the process within minutes or even seconds. The apparatus for MAE is also simpler and cheaper (Hao *et al.*, 2002). Environmentally friendly techniques such as these may enable chemical-free pectin production, enhancing sustainability while producing higher-quality, purer products suitable for diverse industrial applications. (Gavahian *et al.*, 2021).

There are many reports on pectin extraction from orange peels, but the optimal extraction conditions from orange residue (peel and pulp) have not yet been reported. The valorization of orange residue for pectin production is a great opportunity to address environmental pollution and wealth creation. Therefore, this study aimed to extract pectin from orange residue using microwave-assisted methods, to explore how factors such as pH, microwave power, and extraction time affect pectin yield. The goal is to identify the best conditions for maximizing pectin recovery. RSM provides a robust framework for process optimization. This framework enables the simultaneous evaluation of multiple parameters, while also accounting for potential interactions and combined effects that may influence the desired response. (Shivamathi *et al.*, 2022).

2. Methodology

2.1 Sourcing and preparation of orange residue

Fresh orange (*Citrus sinensis*) fruit was bought from Oje fruit market in Ibadan, Nigeria. Clean water was used to wash the fruits, to remove adhering soil and debris, then juiced (Figure 1). The leftover part, which includes the peel and pulp, was cut into small sizes and dried in an oven at 50°C until it reached a constant weight. The residue was washed again to remove any sugars and then blanched for 5 min to stop enzyme activity. After blanching, the residue was treated with absolute ethanol for 25 min to remove oils. The treated residue was then dried in the oven at 50°C until it attained a stable weight, pulverized and kept for further analysis.

2.2 Experiments

2.2.1 Extraction of pectin from pulverized orange residue

Pectin was extracted (Figure 3), based on the method described by Sommano *et al.*, 2018, with a few changes. A microwave oven operating at 2450 MHz with adjustable settings was used. About 10 grams of the pulverized orange residue was placed in a 1-liter Pyrex beaker at ratio of 1:20 (w/v). Distilled water mixed with citric acid was added to make the pH 1.0, 2.0, or 3.0. The beaker was positioned exactly at the center of the microwave to ensure even heating at microwave power levels of 300, 450, or 600 W at different duration: 3, 5.5, or 8 min (Figure 2). Once heated, the mixtures were cooled, filtered, and spun in a centrifuge at 10,000 rpm for 20 min. The liquid on top was separated, mixed with ethanol (two equal parts of 95%) and left over the night at 4°C. The coagulated pectin was carefully poured off, washed several times with ethanol until the solution was clear. The wet pectin was then freeze-dried, ground into a fine powder, and stored in a sealed bag.

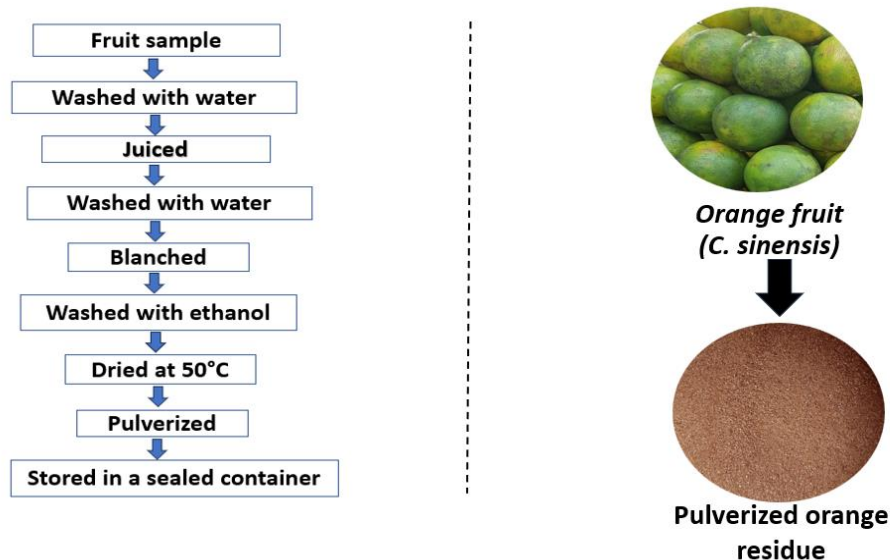


Figure 1. Sample preparation

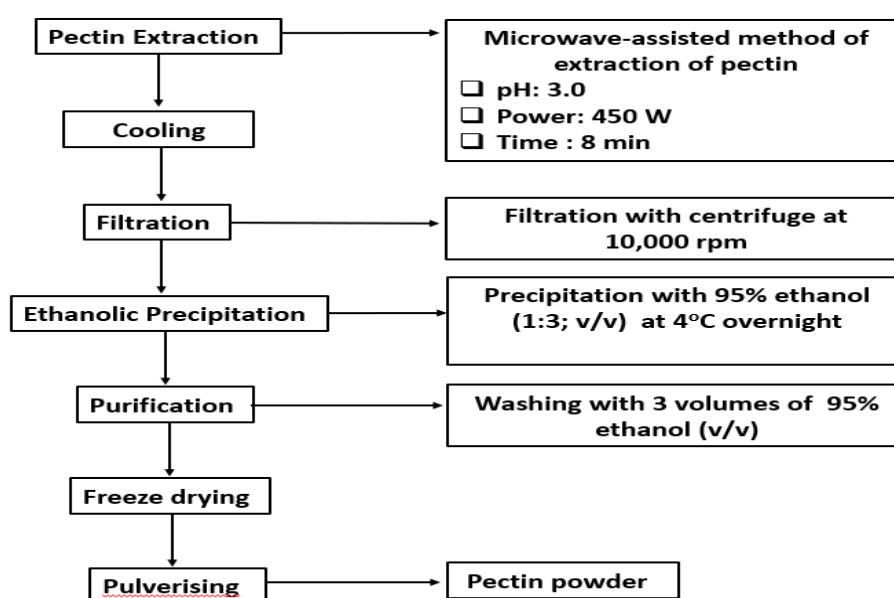


Figure 2. Flow chat of pectin extracted from orange residue

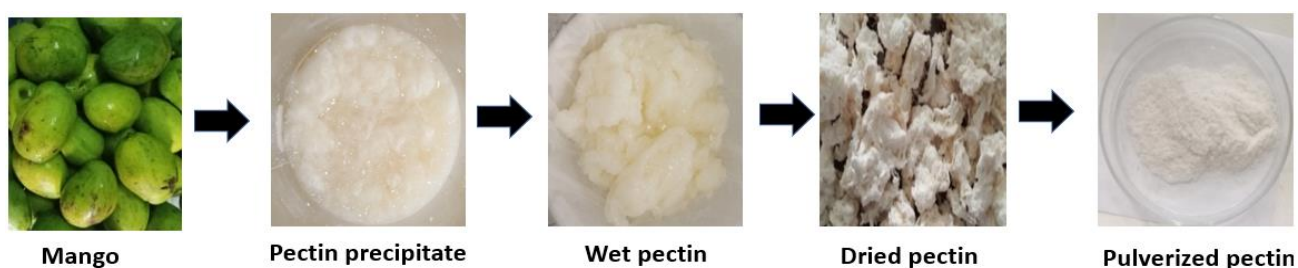


Figure 3. Micrograph of pectin extracted from mango fruit residue

2.2.2 Determination of pectin yield

Wet pectin was dried in an oven at 50°C until it reached a stable weight. The percentage pectin yield (Y%) was calculated using the method described by *Li et al., 2012* and *Casas-Orozco et al., 2015* (Eqn 1):

$$\text{Yield (\%)} = \frac{M_o}{M} \times 100 \quad (\text{Eqn. 1})$$

Where; M_o (g) - weight of pulverised pectin

M (g) - weight of pulverised fruit residue

2.2.3 Experimental design and statistical analysis

The data were analyzed using Design Expert version 13 (State-Ease Inc., Minneapolis, MN, USA) with Analysis of Variance (ANOVA) to assess the effects of the process variables—microwave power, extraction time, and pH—along with their interactions (Maran *et al.*, 2013). This helped in determining how these factors together influence the response (Y), which represents the pectin yield from orange residue. Based on a three-level, three-factor Box-Behnken design (BBD), the extraction yield of pectin will be observed. The yield is strongly influenced by individual process variables which includes; temperature, pH, extraction time, and solvent type, as well as their interactive effects, which often determine the best balance for maximizing recovery without affecting the quality. The ranges of the independent variables and their corresponding levels are summarized in Table 1. For statistical analysis, the variables were coded as -1, 0, and +1. A total of seventeen runs were performed, with six replications at the center points incorporated to assess pure error. The interaction of the response with the three independent variables was described using a quadratic polynomial function as outlined by Prakash *et al.*, 2013.

Table 1. The experimental and coded levels of the three variables employed in the microwave-assisted extraction of pectin from orange residue.

Variables	Factor coding	Unit	Coded levels		
			-1	0	+1
Microwave power	A	W	300	450	600
pH	B	-	1	2	3
Extraction Time	C	min	3	5.5	8

3. Results and Discussion

3.1 Statistical analysis

The results from each experimental run for the extraction of pectin are shown in Table 2. ANOVA identified which model terms had a statistically significant effect, and these important findings are detailed in Table 3. The F-value of 96.57, along with p-values below 0.0001, showed that the model is strong and that the regression equation explains most of the response variation. The lack-of-fit F-value (2.23) indicated that the model can reliably predict the responses. High R^2 (0.9920), adjusted R^2 (0.9817), and predicted R^2 (0.9154) values showed that the model accurately represents the relationship between the response and the independent variables. The low coefficient of variance (2.99) showed that the experiments were very precise and reliable (Prakash *et al.*, 2013).

3.2 Model fitting

A second-order polynomial equation was used to find the best conditions for maximizing pectin extraction yield and to examine how the process variables interact with the response. The second-order models in terms of coded variables are stated below (Eqn. 2).

A quadratic polynomial equation of pectin from orange residue on Yield (Y):

$$Y_{MAE} (\%) = +14.99 - 0.188A + 1.73B + 2.43C + 0.248AB + 0.440AC - 0.055BC + 0.0053A^2 - 1.65 B^2 - 3.09C^2 \quad (\text{Eqn. 2})$$

Table 2. Box-Behnken design (BBD) experimental design matrix with experimental values for the microwave-assisted method of extraction of pectin from orange residue.

Run	Fator 1 A: pH	Factor 2 B: Power (W)	Factor 3 C: Time (min)	Response Yield (%)
1	2	450	5.5	15.23
2	2	300	3	6.12
3	1	450	3	9.72
4	3	600	5.5	14.84
5	1	300	5.5	12.35
6	1	600	5.5	15.18
7	2	450	5.5	14.67
8	2	450	5.5	15.37
9	3	450	3	8.92
10	2	600	8	14.27
11	3	450	8	14.97
12	1	450	8	14.01
13	2	450	5.5	14.71
14	2	300	8	10.79
15	2	600	3	9.82
16	2	450	5.5	14.98
17	3	300	5.5	11.02

Table 3. Analysis of variance for Quadratic model for the microwave-assisted method of extraction (MAE) of pectin from orange residue.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	126.98	9	14.11	96.57	< 0.0001	significant
A-pH	0.2850	1	0.2850	1.95	0.2052	
B-Power	23.91	1	23.91	163.65	< 0.0001	
C-Time	47.34	1	47.34	324.01	< 0.0001	
AB	0.2450	1	0.2450	1.68	0.2364	
AC	0.7744	1	0.7744	5.30	0.0548	
BC	0.0121	1	0.0121	0.0828	0.7818	
A ²	0.0001	1	0.0001	0.0008	0.9783	
B ²	11.46	1	11.46	78.44	< 0.0001	
C ²	40.26	1	40.26	275.58	< 0.0001	
Residual	1.02	7	0.1461			
Lack of Fit	0.6398	3	0.2133	2.23	0.2273	not significant
Pure Error	0.3829	4	0.0957			
Cor Total	128.00	16				

3.3 Diagnostics of model adequacy

The model's accuracy was checked using several diagnostic plots, including predicted versus actual values, normal percentiles plots, and internally studentized residuals, as shown in Figure 4. The predicted values from the model closely matched the experimental data and aligned well with the straight line, showing good agreement with the actual data (Figure 4a). Figure 4b shows the normal probability plot of residuals for a normally distributed response, which lie close to a straight line and show no noticeable deviations, indicating that the response is normally distributed. An internally studentized residuals plot was made to check how well the model fits the data. The results (Figure 4c) show that all the data points are within the expected range.

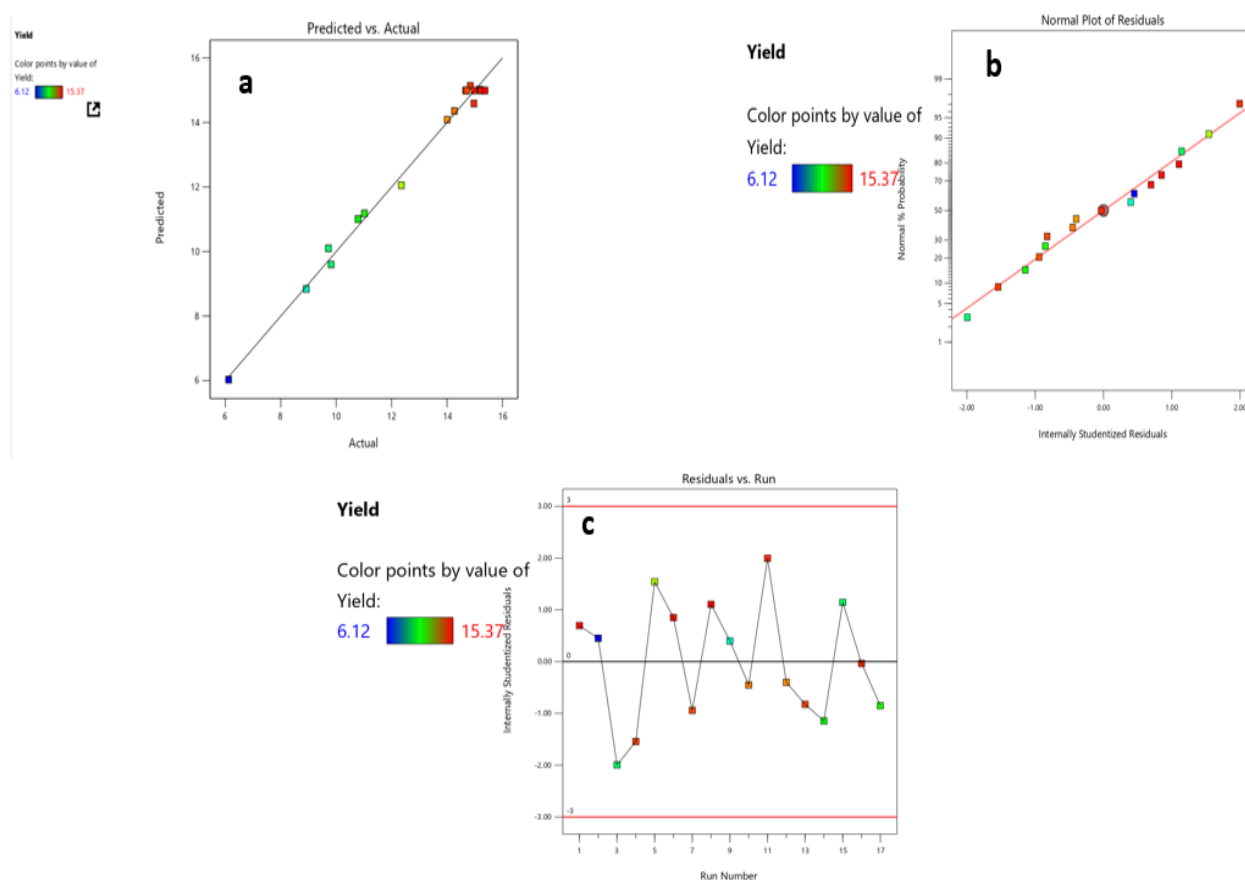


Figure 4. Diagnostic plots for the model adequacy

3.4 Effect on process variables.

3.4.1 Effect of microwave power

The impact of microwave power on extracting pectin from pulverised orange residue was studied by using power levels from 300 to 600W. The increase in pectin extraction with higher microwave power is because the microwave energy directly affects the plant material. Microwave radiation breaks down the cell walls, quickly and widely opening the outer layers (Kratchanova *et al.*, 2004). This makes it easier for the extracting solution to interact with the plant material. Microwave energy improves the transfer of electromagnetic energy to the plant's molecules through ionic interactions, causing the molecules to move and heat up in the extraction process. This speeds up the

extraction process. The amount of pectin obtained from orange residue varied between 6.12% and 15.23% (Table 2). This change depends on the settings used. During the extraction process, there is a sudden rise in temperature, which improves pectin solubility and boosts the extraction rate. The steady increase in yield with rising temperature is because insoluble protopectin changes into soluble pectin. Under the conditions of Run 1 (pH 2, 450W, 5.5 min), the highest pectin yield was recorded at 15.23% (Tumisi and Jeremiah, 2025). Phaiphan *et al.*, 2020, found that longer exposure time and higher microwave power both improve pectin yield. However, when the power increases to 600W, the pectin yield drops (Run 10; pH 2, 600W, 8 min), and gave a yield of 14.27%, because of pectin breakdown (Sarah *et al.*, 2020).

3.4.2 Effect of irradiation time

Irradiation time has a big impact on how much pectin you can extract, and finding the right amount of time is important to get the most pectin from orange residue. Results showed that as the time increased from 3 to 5.5 min, the amount of pectin extracted went up steadily, reaching the highest level at 5.5 min (Figure 5) (Awoyemi 2024). Bagherian *et al.*, 2011, found that longer exposure helps extract more pectin by breaking down plant material, making it easier for the pectin to come out. But if the time is too long, the heat can damage the pectin, lowering its quality and the amount you get. The increase in pectin up to 5.5 min is because the microwave energy is absorbed by the liquid, causing more heat buildup and making the pectin dissolve better. However, if you leave it in the microwave for too long, the pectin can start to break down (Xianzhe *et al.*, 2011). For example, Run 2, which had a pH of 2, 300 W power, and 3 min, gave the least pectin. This happened because lower power and shorter time didn't give enough energy to extract much pectin (Iñiguez-Moreno *et al.*, 2024).

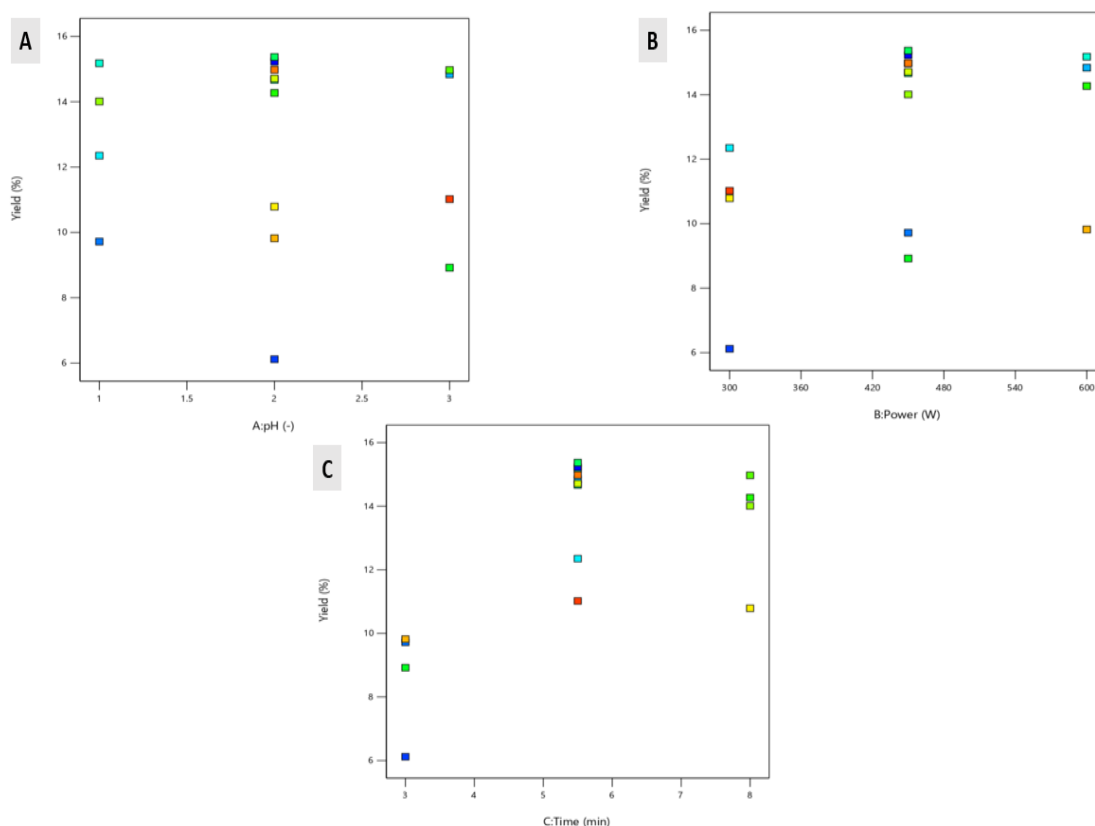


Figure 5. Scatter plots showing the influence of extraction parameters on the Y (A-C) for the microwave-assisted method of extraction of pectin from orange residue

3.4.3 The effect of pH

It was also clear. As the pH got lower, the amount of pectin extracted went up (Figures 5 and Figure 6; Awoyemi 2024). At lower pH levels, the acid in the solution can get into the insoluble pectin more easily, helping to break it down into soluble parts. This makes it easier to extract the pectin, with the best results seen at pH 2 (El-Nawawi and Shehata, 1988). Stronger acids can interact with the pectin more effectively, breaking it down into parts that dissolve better, which helps get more pectin out of plant materials (Ma *et al.*, 2013). A similar study was done by Hosseini *et al.*, 2015. However, when the pH went above 2 (specifically at pH 3), the amount of pectin produced dropped. (Zioga *et al.*, 2022). This might be because pectin started to clump together, which slowed down its release.

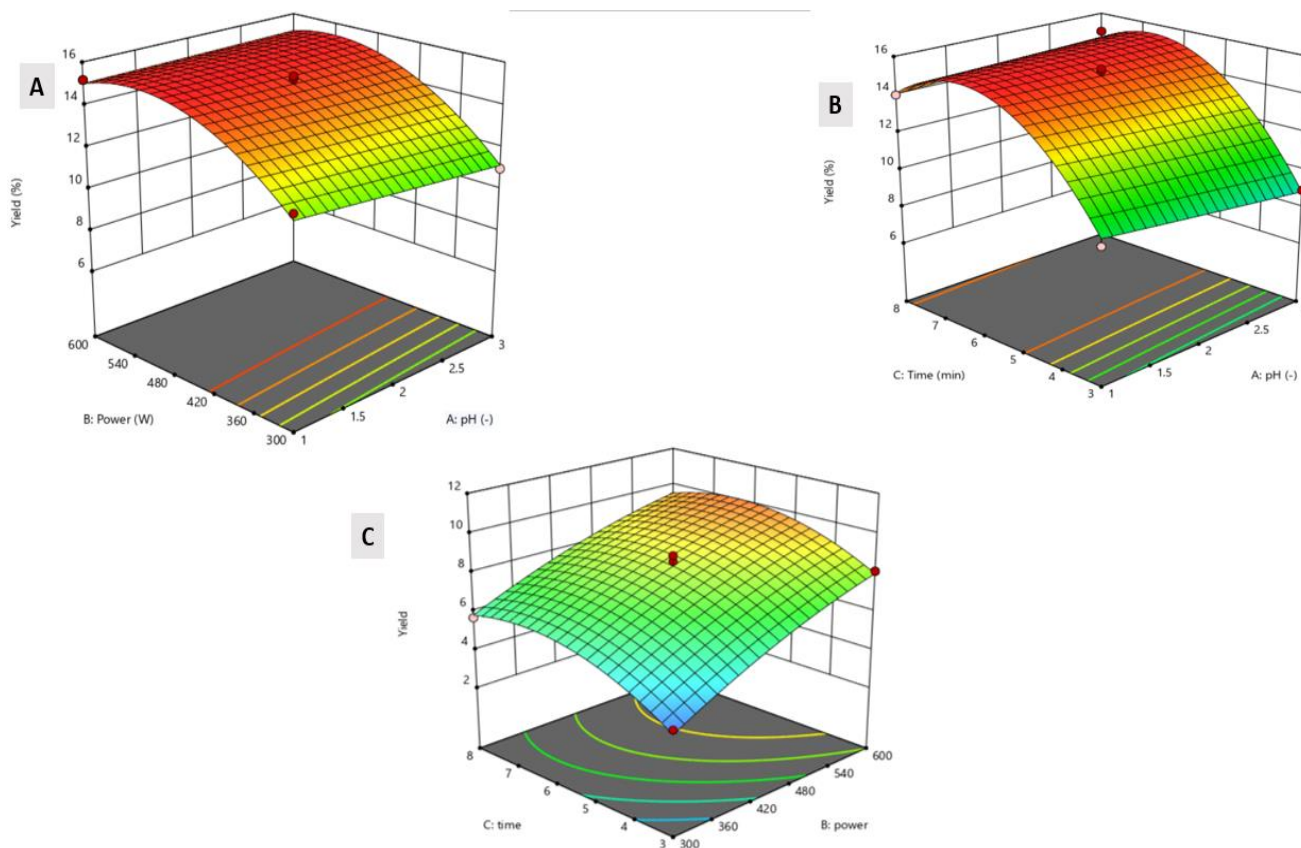


Figure 6. 3D plots showing the influence of extraction parameters on the Y (A-C) for the microwave-assisted method of extraction of pectin from orange residue

3.5 Optimization of extraction parameters and validation of the best conditions for extracting pectin from orange residue

The best conditions found through response surface methodology (RSM) were: a microwave power of 389.904 W, an irradiation time of 5.744 seconds, and a pH of 2.785. These conditions gave a pectin yield of 14.055% and a desirability value of 1.0 shown in Table 4. (Maran *et al.*, 2013). The experimental results supported these optimized conditions, proving that they are reliable.

Table 4. Optimized condition of the RSM experiment of orange residue

pH	Power (W)	Time (min)	Yield	Desirability
2.785	389.904	5.744	14.055	1.0

Conclusion

This study demonstrated that microwave-assisted extraction, optimized using Response Surface Methodology (RSM), is highly effective for maximizing pectin yield from orange residue. The findings revealed that microwave power, irradiation time, and pH significantly influence extraction efficiency, both individually and in combination. Specifically, higher microwave power coupled with longer irradiation at low pH enhanced pectin yield. The process was shown to be highly sensitive to acidity, with lower pH values increasing the yield. Optimal conditions were identified as 389.904 W microwave power, 5.744 seconds irradiation time, and a pH of 2.785, producing a maximum yield of 14.055%. These optimized parameters provide valuable guidance for future research. Moreover, the study confirms that microwave-assisted extraction is a feasible and efficient method for producing pectin, with strong potential for application in the food industry.

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Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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