Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage:
https://semarakilmu.com.my/journals/index.php/fluid_mechanics_thermal_sciences/index
ISSN: 2289-7879

Boost Pectin Production using Continuous System of Subcritical Water Extraction

Noor Azwani Mohd Rasidek[1,*], Zuhaili Idham[2], Ahmad Hazim Abdul Aziz[3], Norlisa Mili[4], Nicky Rahmana Putra[1], Ahmad Syahmi Zaini[1], Mohd Azizi Che Yunus[1,♣]

[1] Centre of Lipids Engineering & Applied Research (CLEAR), Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, UTM, 81310 Johor Bahru, Johor, Malaysia
[2] Ibnu Sina Institute for Scientific and Industrial Research (ISI-SIR), Universiti Teknologi Malaysia, UTM, 81310 Johor Bahru, Johor, Malaysia
[3] Faculty of Food Science and Nutrition, Universiti Malaysia Sabah, 88400, Kota Kinabalu, Sabah, Malaysia
[4] Department of Chemical Engineering and Energy Sustainability, Faculty of Engineering, UNIMAS, 94300 Kota Samarahan, Sarawak, Malaysia

ARTICLE INFO

Article history:
Received 15 April 2022
Received in revised form 13 August 2022
Accepted 24 August 2022
Available online 19 September 2022

Keywords:
Soluble pectin; fast extraction process; interaction temperature; continuous system SWE; response surface

ABSTRACT

The dumping of waste banana peels from local chip manufacturing has encouraged sustainable food trends to valorize these peels as pectin sources. In this study, soluble pectin from banana peels was extracted using a continuous subcritical water extraction system (SWE). This method was a promising fast extraction process with a water solvent that was environmentally friendly. Temperature (100 to 160 °C) and extraction time (5 to 50 minutes) were manipulated, with a fixed pressure of 15 MPa, a flow rate of 15 mL/min, and a particle size of 0.6 mm. The response surface methodology (RSM) was used to generate approximately 13 experimental trials from the central composite design. Based on linear and square models, the results revealed that the interaction of temperature and time to pectin yield had a significant effect. At 100°C for 5 minutes, an optimal condition for producing pectin yield was achieved, which risen the highest pectin yield by 26%. As a result, the SWE continuous system promoted increased pectin production in the shortest extraction time. In short, banana peel pectin is a powder that serves a primary function in numerous food, pharmaceutical, and cosmeceutical applications.

1. Introduction

The rapid growth of various manufacturing industries such as foods, cosmetics, and medicines has stimulated the need for pectin sources. According to Grand View Research, the pectin market is estimated to be worth USD 1.0 billion in 2019 and is expected to grow at a CAGR of 6.5 percent from 2019 to reach USD 1.5 billion by 2025. The pectin industry is growing due to rising convenience food consumption, increased health consciousness, and the multi functionality of pectin that is applicable for a wide range of applications. The importance of pectin sources in various applications has

---

[*] *Corresponding author.*
E-mail address: noor.azwani@utm.my
[♣] *Corresponding author.*
E-mail address: r-azizi@utm.my

https://doi.org/10.37934/arfmts.99.2.168179

triggered many researchers to explore aggressively by extracting the pectin using a current or advanced method from various local agriculture sources that are readily available. Every year, 998 million tons of agricultural waste are generated globally. For instance, apple pomace and citrus peel are the main primary waste product in juice manufacturing [1,2]. Contributed locally, banana peel is one of the main agricultural wastes in the chips manufacturing sector [3].

Banana (Musa spp.) is Malaysia's second most extensively cultivated fruit and it can be consumed ripe or unripe [4]. The ripe banana is regularly used to make fried bananas, whereas the unripe banana is used to make products such as chips and juices. Banana peels, which account for approximately 40 % of the total weight of fresh bananas, were indeed typically discarded in landfill space, rotten out without meaningful reuse, and triggered environmental issues [5]. Banana peel contains a large number of carbohydrates, a vast quantity of dietary fiber, mainly hemicelluloses and pectin polysaccharides at 10 to 21 % by weight, and others constitute is natural sugar [2]. One way to utilize this banana peel was by using it as a source of pectin.

Pectin is known over the past decade that is a basic element holding the cell wall and middle lamellae of a plant cell. Pectin consists of a group of heteropolysaccharide block copolymers mainly comprised of high proportionate 1,4-α linked galacturonic acid (GA) units attached in a chain-like connection. Pectin has a variety function that widely acted as a gelling, thickening, or stabilizing agent [6,7]. It is used as a fat or sugar replacer in jams, jellies, frozen foods, and more. It is also used in the pharmaceutical industry to lower blood cholesterol levels and treat gastrointestinal disorders. Other than that pectin is used for packaging material or known as edible films, paper substitutes, foams, and plasticizers. Commercial natural pectin is derived from various fruit peel sources mainly contributed from the food manufacturing process [8]. Usually, pectin is produced from the extraction process and normally pectin was extracted from citrus fruits in the form of white or light brown powder [6]. Commercial pectin production involved strong acid or alkaline as a solvent and this results in several drawbacks such as longer process time.

The extraction process has always been a pivotal unit operation in recovering interest compounds such as pectin from plant cell walls. Lately, the advanced extraction technologies such as subcritical water extraction (SWE), supercritical fluid extraction (SFE) and others demonstrated high selectivity in enrolling a naturally clean process [9,10]. Up till now, the extraction method uses chemical substances as the main solvent had promising a high proficiency of production. However, using a large volume of organic solvents are utilized by industry might have unwanted effects on the surrounding and product components and will eventually harmful to human health and cause environmental stress.

To address these drawbacks, the continuous system subcritical water extraction (SWE) method that is efficient, economical, eco-friendly, harmless, and fast is vital. Continuous SWE using water as the solvent is one of the most exciting methods because water is non-flammable, non-toxic, inexpensive, and sustainable. Subcritical water, also called pressurized hot water, compressed hot water, or superheated water; is hot water that needs to maintain its condition in a liquid state under temperatures between 100°C and 374°C and pressure between 1MPa and 22.1 MPa [11]. Thus, continuous system SWE was the best solving extraction process to extract pectin from agricultural waste since SWE gave a higher efficiency, lower extraction time, and is not harmful to the environment. There are very few studies on pectin from banana peels using advanced technology. For that reason, it promoted the idea of using one of the advanced technologies with maximizing output.

The empirical statistical modeling procedure known as response surface methodology (RSM) implements the multiple regression analysis has been used to explain simultaneous multivariate equations [12]. It is a greatly resourceful statistical tool that brings out optimization for manifold processes. The experimental trials desired to evaluate independent factors and their interfaces can be reduced. In this study, RSM was used to boost the continuous system SWE (temperature and time) extraction of pectin from banana peels. The study attentions on optimizing the extraction factors to increase pectin yield using the central composite design (CCD). Hence, the objective of this study is to boost the production of pectin sources from banana peels using the continuous system SWE and the CCD is used to develop the regression model and optimize the effect of temperature and time towards maximized the pectin yield.

2. Methodology

2.1 Sustainable Source of Banana Peels

Routinely abundance of banana peels has been discarded as agricultural wastes from the food industry, Azhar Food Manufacturing Sdn. Bhd is located at Rengit, Pontian Johor. These banana peels have been utilized properly as it is taken more than a year to biodegrade. Thus, the collection of banana peels mainly from of 'Pisang Tanduk' variety collected from that factory.

In this initiative, the banana peels were kept in a zipper bag to minimize an oxidation reaction. Then, the banana peels were cut into 1 cm sizes and soaked in fruit detergent to remove any impurities. Wash thoroughly and oven-dried the banana peels at a temperature of 50°C. The dried banana peels were ground using the commercial blender and sieved with particles size of 0.6 mm. Then, the banana peel powder was kept in a tight container and stored at room temperature for further process.

2.2 Simplified Experimental Design

A Design Expert 13th software was used to simplify the experimental work consisting of temperature and time as parameters. The Central Composite Design (CCD) of 2 independent variables was fitted to the response function. The selection of design is based on a few experimental combinations of the variables required to approximate the complex response function. Thus, the response surface second-order polynomial model is expected to take the following form of Eq. (1) below:

$$Y = B_O + \sum_{i=1}^{k} B_i X_i + \sum_{i=1}^{k} B_{ii} {X_i}^2 + \sum_i \sum_j B_{ij} X_i X_j \tag{1}$$

Where Y is the predicted response; B_O is a constant; B_i, B_{ii}, and B_{ij} represent the interactions of the effect; X_i and X_j are the coded value parameters.

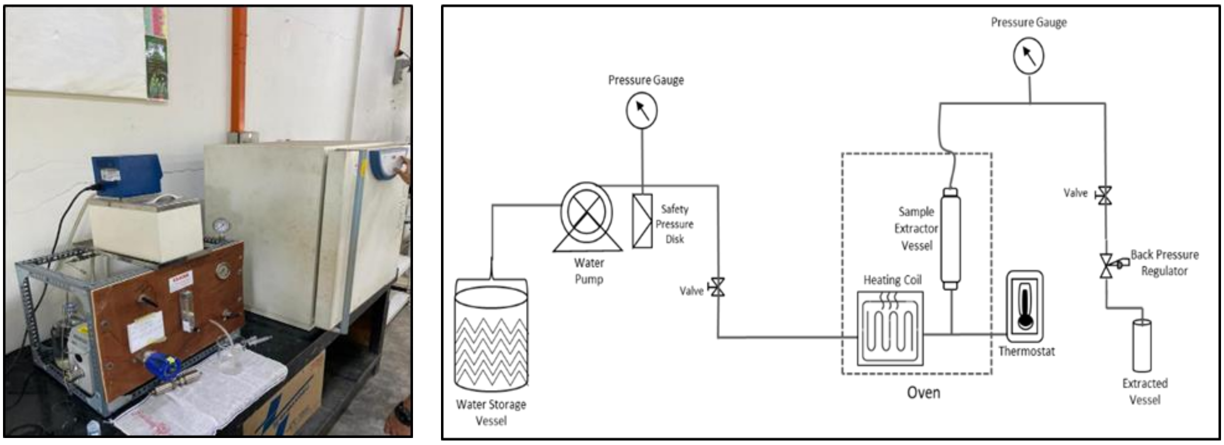
For this extraction process, 13 experimental run order under the Central Composite Design (CCD) with 2 parameters were generated and the level of parameters are shown in Table 1:

Table 1
Level of Extraction Parameters

Parameters	Point Type		
	Minimum level (-1)	Central point (0)	Maximum level (+1)
Temperature (°C)	100	130	160
Time (min)	5	10	15

2.3 Continuous System Subcritical Water Extraction

The production of pectin was established using the continuous system of subcritical water extractor (SWE) at the Centre of Lipid Engineering and Applied Research (CLEAR) laboratory, UTM Skudai Johor as represented in Figure 1.



(a) (b)

Fig. 1. Fabricated Continuous System Subcritical Water Extractor (a) Photograph (b) Schematic Diagram

About 1 g of dried banana peels were loaded into the sample extractor vessel and about 200 mL of distilled water flowed in during the extraction process. The extraction condition consisted of temperature and time ranging from 100°C to 160°C, 5 to 15 min at a fixed pressure of 15 MPa. The continuous system SWE is fabricated with a continuous flow rate at 15mL/min associated with high pressure to maximize the pectin yield from banana peel wastes.

2.4 Wetted to Dried Banana Peel Pectin

After the extraction process, about 100 mL of banana peel extracts were produced and precipitated by adding 95% of ethanol into the extracts in a ratio of 2:1 (v/v). The mixture of ethanol and the extracts was stirred using a magnetic stirrer for about 10 min at room temperature to homogenize the mixture as referred to Canteri-Schemin *et al.,* [7] study. The mixture was left overnight at room temperature to allow the precipitation of pectin polysaccharides. Then, pectin precipitates were collected, which is known as the wetted pectin shown in Figure 2(a).

Initially, the wetted pectin that still contained 95% ethanol was vortexed and centrifuged (Centrifuge 5840, Eppendorf, Germany) at 7000 rpm for 5 min. The supernatant was removed and the residue was washed using 70% ethanol. This procedure was repeated twice. After that, about two drops of acetone were dropped on the residue to remove the unwanted color. Then, banana peel pectins were collected using ashless filter paper (Munktell Filter, Sweden) and dried using an oven (Memmert, UM100, Germany) at 50°C approximately for 3 hours until a constant weight was achieved. The dried banana peel pectin as shown in Figure 2(b) was grounded using a porcelain mortar and kept stored in a glass bottle before further analysis.

(a) (b)

Fig. 2. Banana Peel Pectins (a) Wetted Form (b) Dried Powder Form

Determination of pectin yield percent was calculated as a gram of dried pectin obtained per gram (g) of the dried peel of raw material used as shown in Eq. (2) below:

$$Pectin\ Yield\ (wt\ \%) = \left(\frac{Dried\ pectin\ (g)}{Dried\ peel\ raw\ (g)}\right) x\ 100 \tag{2}$$

2.5 Statistical Analysis

The statistical analysis is generated and analyzed from Design Expert 13th, USA, which comprises of individual desirability (*D*) and evaluates the effect of factors to optimize a single response. As a reference, if the Desirability, *D* value is close to 1, it designates the factors seem to achieve favourable results for all responses as a whole. Also, the *D* indicates that the settings are more effective at maximizing or minimizing responses. Meanwhile, the probability of an upper 95% exact confidence level is set to exclude 0.05 is calculated for each resulting group testing design.

3. Results

3.1 Development Regression Model Equation

From the central composite design (CCD), there are thirteen (13) run orders of the experiments were carried out according to the temperature and extraction time conditions. Table 2 illustrated the overall design of two factors and responses that have been used to obtain the quadratic models.

Table 2
Experimental Runs of Central Composite Design

Run Order	Parameters		Response
	Temp (°C)	Time (min)	Pectin Yield (%)
1	100	5	24.51
2	130	5	13.64
3	160	5	0.38
4	130	10	17.01
5	100	10	26.48
6	160	10	21.09
7	130	10	18.02
8	100	15	15.81
9	130	15	10.62
10	160	15	21.43
11	130	10	18.82
12	130	10	16.91
13	130	10	14.21

Table 2 showed, that at 100°C, the pectin yield achieved was from 15.81 to 26.48 %, while at 130°C the pectin yield was from 10.62 to 18.02 % was obtained, followed by 160°C, which obtained the pectin yield is from 0.38 to 21.43 %. At a high temperature of 160°C with exceeded of extraction time 15 min, the increment of solubility extracted pectin has occurred. It is caused by the extraction condition in continuous system SWE giving a higher rate of extraction. Otherwise, in our previous study using the batch system of SWE was in ranging from 0.72 to 4.88 %. At higher temperatures in the batch system, SWE would activate to break down the pectin molecules as pectin is composed of α-(1–4) linked units of galacturonic acid (GA) or methyl ester ($-CH_3$), that happened at 160°C [13]. Contrarily, Swamy and Muthukumarappan [14] reported a lower banana peel pectin yield that was only achieved at 2.58 %, as they use the intermittent microwave extraction method. Thus, it could be related to the efficiency of the extraction method within the dominant parameters such as temperature, time, and pressure which play an important role in maximizing the output of pectin yields.

Extraction time is also a crucial parameter in the extraction of pectin yield. Most of the findings reported the extraction time is mainly related to the yield extraction. So, extraction yield increase with time increase [15]. Throughout the continuous system, SWE agreed as the pectin yield was significantly increased as time increased to 15 min and it is different by using the batch system SWE. However, both studies produced the highest yield within 5 min and the selection of 5 min is the most efficient and promising more economic.

From the assortments of 13 CCD matrixes, data were used to determine the coefficient model. The estimated coded coefficients were shown in Table 3 along with the coefficient of determination R^2, adjusted R^2, and predicted R^2.

Table 3
Coded Coefficients for Pectin Yield

Source	Coded Coefficients	*p*-value
Regression Model	+166.874	0.0023
Linear		
A	-2.1179	0.0095
B	-0.9350	0.2097
Interaction		
AB	+0.04966	0.0010
Quadratic		
A^2	+0.0057	0.0172
B^2	-0.2600	0.0058
R^2	0.8984	
Adjusted R^2	0.8259	
Predicted R^2	0.2189	

As can be seen from Table 3, the coefficient determination of yield revealed R^2 value of 0.8984. It was indicated that the model could be described at 89.84 % of the data variation and only 10.16 % of the total variations were not described by the model. For a model to be acceptable, the R^2 value should not be less than 0.75 [16]. However, Koocheki *et al.,* [17] proposed that a large value of R^2 did not always denote that the regression model was a good one. Such inference could only be made based on a similarly high value of adjusted R^2 [17]. The adjusted determination coefficient (adj R^2) for yield was comprised of 0.8259. The p-value obtained was 0.0023 which is less than 0.05 indicating model terms are significant and important. The predicted R^2 shown is relatively lower at 0.2189 than the adjusted R^2 as it can guard against models that are too complicated.

In this case, A, AB, A^2, and B^2 are significant model terms. Therefore, it was confirmed that the quadratic model was equitable significant, which specified a respectable agreement between the experimental and predicted values of extraction parameters.

Therefore, obtained models could be applied to determine the relative effects of factors. In achieving the optimum combination of factors for a maximum pectin yield as well as to predict the test results for other conditions. The quadratic models were stated as in Eq. (3)

$$Pectin\,Yield = 166.8742 - 2.1179\mathrm{A} + 0.0497\mathrm{AB} + 0.0057\mathrm{A}^2 - 0.2600\mathrm{B}^2 \quad (3)$$

where A is the temperature coefficient and B is the extraction time coefficient. From the equation model, the positive (+) and negative (-) signs indicate the interaction effects of factors [18]. The positive (+) meant the synergetic effects of a factor, while the negative sign (-) indicated an antagonistic effect [19]. The synergetic effect has been raised between two or more factors that produced an effect greater than the sum of their individual effects. Moreover, the antagonistic effect is wherein two or more factors in combination have an overall effect that was less than the sum of their individual effects. Eq. (3) also reveals that the effect of temperature was the most influential variable during the extraction process. This is due to the high value of coefficient (2.1179) and low $p<0.0095$ and the effect of extraction time was not significant due to the low coefficient value and large $p>0.2097$.

To avoid poor or misleading response surface results, the adequacy and fitness of the model have been verified using regression analysis and analysis of variance (ANOVA) [20]. Thus, the statistical model was checked by *f*-tests and summarized in Table 4 and definite that the equation could efficiently describe the correlation among the factors and responses.

Table 4
ANOVA Table for the Response of Pectin Yield

Source	Sum of Squares	Degrees of Freedoms	Mean Square	*f*-value	*p*-value
Model	470.85	5	94.17	12.38	0.0023
Linear					
A	95.20	1	95.20	12.52	0.0095
B	14.51	1	14.51	1.91	0.2097
Interaction					
AB	221.27	1	221.27	29.10	0.0010
Quadratic					
A^2	73.40	1	73.40	9.65	0.0172
B^2	116.68	1	116.68	15.35	0.0058
Residual Error	53.23	7	7.60		
Lack of Fit	41.08	3	13.69	4.51	0.0898
Pure Error	12.14	4	3.04		
Total	524.07	12			

Table 4 indicated yield response as showed the model *f*-value of 12.38 implies that the model is significant. There is only about a 12 % chance that the model of *f*-value could occur because of noise. For linear terms of B ($p<0.2097$, $f=1.91$) has no significant effect during extraction process. Otherwise, Wang *et al.,* [21] stated that changing the extraction time had highly significant effects on the yield ($p<0.0001$). It was observed that the *f*-values for interaction term gave highest at 29.10. Thus, a large f-value showed that the deviation in the responses could be clarified by the regression equation, indicating that the model term is greatly significant [14]. Another piece of confirmation is the lack of fit *f*-value. The lack of fit f-value for yield gave at 4.51, which implies the lack of fit is significant relative to the pure error. According to Yolmeh and Jafari [22], a significant lack of fit has indicated the unsuitability of the model. However, in this case, all the model coefficients for namely A, AB, A^2, and B^2 are significant.

Furthermore, comparing residual plots were investigated to determine whether the quadratic model meets the assumptions of the analysis. The residual plot was displayed in Figure 3 which included the value of predicted versus actual data. It also illustrated that the model has successfully fitted due to the 0.23% minor difference between the values of predicted and actual.

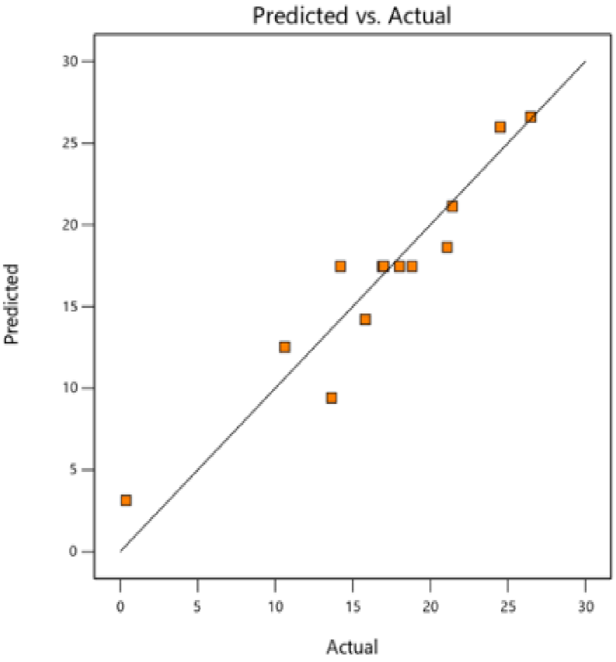


Fig. 3. Residual Plot of Pectin Yield

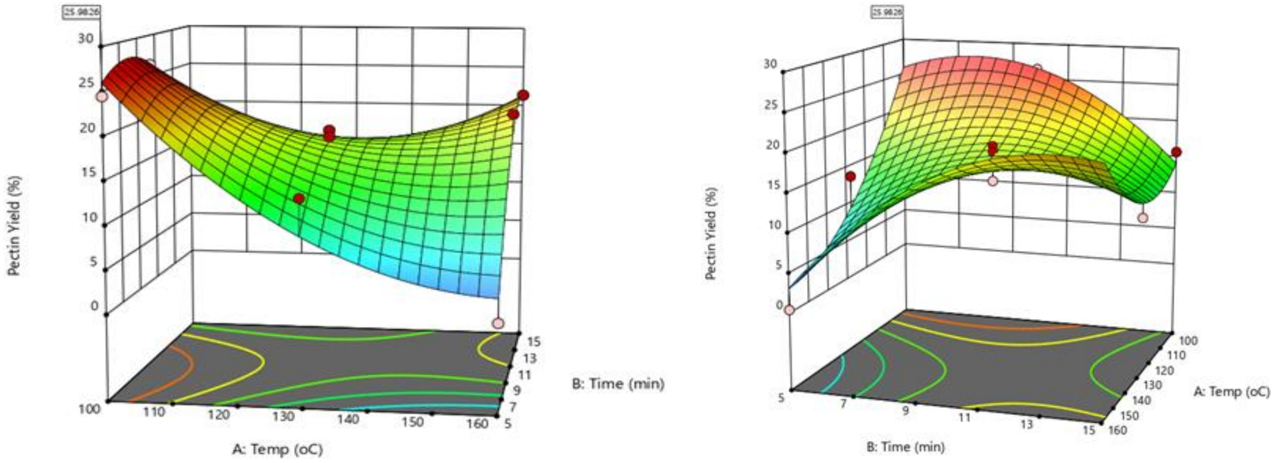
As can be seen in Figure 3, the residual analysis consists of normal probability presented the quadratic model successfully captured the correlation between the process conditions of extraction owing to the predicted values were nearly close to the actual values. Thus, it is clear that the values resulting experimentally match closely with that developed by the model. Related studies have been reported for pectin extraction from durian rinds and pomelo peels [23,24].

3.2 Interaction Effect towards Pectin Yield

To visualize the correlation between the response and process factors, a three-dimensional (3D) response surface was generated from the model equation. The coefficient of the 3D dimension response surface plot showed mutual interactions between the extraction temperature and time. Initially, the 3D response surfaces were plotted to explore the interaction factors at middle holding values at temperature 130°C and time 10 min against the pectin yield. Hence, the interaction effect of temperature*time and time*temperature on pectin yield was illustrated in Figure 4(a) and Figure 4(b).

Figure 4(a) and Figure 4(b) represented the three-dimensional (3D) response surface plot of the interaction effect between temperature and time toward the pectin yield percent. In Figure 4(a), a low point of the temperature of 100°C and time of 5 min contributed the highest percent of pectin yield. Moreover, at the middle point of the temperature of 130°C and time of 10 min, the pectin yield is gradually decreased. At the high point of the temperature of 160°C and time, 15 min has progressively increased the pectin yield. These changes are due to the transition state of pectin that is affected by thermal changes during the subcritical extraction process [3]. In subcritical conditions, the temperature is the most dominant parameter for the flow deformation properties. As temperature rises, the dielectric constant of the solvent decreases, resulting in enhanced pectin solubility in water [25,26].

Meanwhile, in Figure 4(b), the interaction of time and temperature towards pectin yield showed similar trends. This proves that the combination of both interactions on pectin yield is very significant.



(a) (b)

Fig. 4. Interaction Effect of (a) Temperature*Time towards Pectin Yield (b) Time*Temperature towards Pectin Yield

3.3 Pectin Yield Optimization

To achieve the maximum response that jointly satisfied all process conditions, thus the response optimization plot was generated. The response optimization is associated with several constraints as described in Table 5 and followed the optimization solutions with its desirability, *D* as shown in Table 6. The selected extraction condition was based on the highest *D* which was more than 70%.

Table 5
Optimization Constraints

Name	Goal	Lower Limit	Upper Limit	Importance
Temperature (A)	Minimize	100	160	5
Time (B)	Minimize	5	15	5
Pectin Yield	Maximize	0.38	26.48	5

Table 6
Optimization Solutions

Number	Temperature (°C)	Time (min)	Pectin Yield (%)	Desirability, (*D*)
1	100.000	5.000	25.983	0.994
2	100.000	5.125	26.156	0.992
3	100.000	5.315	26.405	0.988
4	100.000	5.361	26.642	0.988
5	100.771	5.000	25.427	0.982

As highlighted in Table 6 is the optimum condition that denoted at a temperature of 100°C and 5 min of extraction time with its highest desirability, *D* of 0.994. Therefore, the point of optimum condition was illustrated in Figure 5.

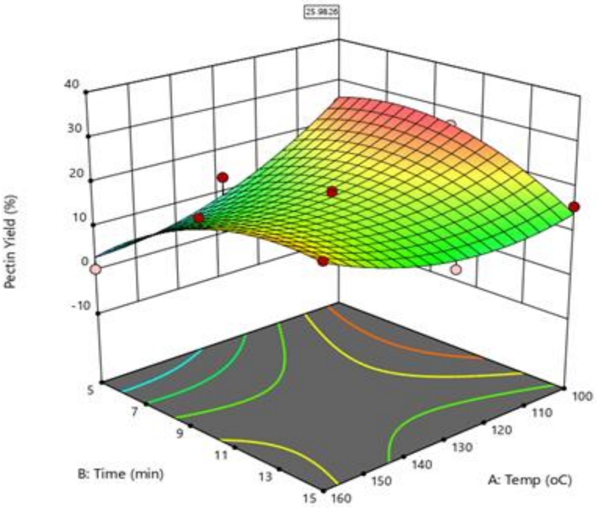


Fig. 5. Optimization Plot for Maximum Pectin Yield

Figure 5 shows the prediction of response was based on yield targeted settings at the maximum set. It also provides an overview of the current setting factors of temperature at 100°C, a time of 5 min was highlighted to fulfil the best extraction condition and be more economical. As supported by Pasandide *et al.,* [15] the temperature range is the most significant parameter because it frequently has a resilient effect on the yield. As the results revealed, the pectin yield was lower at 160°C, 5 min, and the pectin yield became increased as exceeded the extraction time of 15 min. At 160°C, the extraction rate increased giving a significant reaction to banana peels being fully broken causing pectin solubility in the water to increase. Another factor affecting yield was extraction time. Many researchers described that the extraction time is continuously related to the yield extraction so that the extraction yield increase by increasing time [15]. Yet, other features must be considered, such as the use of different extraction methods within a low-temperature range, which could increase the yield percent even in excess time.

4. Conclusions

This study puts forward that the CCD design establishes to be effective and adequate in constricting down the optimal results of the continuous system SWE extraction of pectin from banana peels. The observational data present that the independent factors have a significant, important effect on the extraction process and response surface plots assessed the interaction effect input factors on the reaction. Second-order polynomial models projected the pectin yield. ANOVA results specified a high coefficient of determination values R^2 of 0.8984 for continuous system SWE of pectin yield. Hence, the fit of the regression model with the experimental values was assessed and established to be fit. Thus, the optimum condition with desirability, D is 0.994 using the continuous system SWE at 100°C, 5 min successfully produced the maximum pectin yield until 26 %. The interaction effect of temperature showed the most significant parameter which boosted pectin yield within the shortest extraction time.

Acknowledgement

The authors would like to prompt their thankfulness for UTMIcon 2.1 (Q.J130000.4351.09G56) from Universiti Teknologi Malaysia and UTM Post-Doctoral Fellowship Scheme bestowed to Noor Azwani Mohd Rasidek is gratefully acknowledged.

References

[1] Wang, Xin, Quanru Chen, and Xin Lü. "Pectin extracted from apple pomace and citrus peel by subcritical water." *Food Hydrocolloids* 38 (2014): 129-137. https://doi.org/10.1016/j.foodhyd.2013.12.003

[2] Emaga, Thomas Happi, Rado Herinavalona Andrianaivo, Bernard Wathelet, Jean Tchango Tchango, and Michel Paquot. "Effects of the stage of maturation and varieties on the chemical composition of banana and plantain peels." *Food Chemistry* 103, no. 2 (2007): 590-600. https://doi.org/10.1016/j.foodchem.2006.09.006

[3] Rasidek, Noor Azwani Mohd. "Subcritical Water Extraction of Banana Peel Pectin from Food Industry Waste." *PhD diss., Universiti Teknologi Malaysia*, 2018.

[4] Tock, Jing Yan, Chin Lin Lai, Keat Teong Lee, Kok Tat Tan, and Subhash Bhatia. "Banana biomass as potential renewable energy resource: A Malaysian case study." *Renewable and Sustainable Energy Reviews* 14, no. 2 (2010): 798-805. https://doi.org/10.1016/j.rser.2009.10.010

[5] Zhang, Jixian, Chaoting Wen, Haihui Zhang, Yuqing Duan, and Haile Ma. "Recent advances in the extraction of bioactive compounds with subcritical water: A review." *Trends in Food Science & Technology* 95 (2020): 183-195. https://doi.org/10.1016/j.tifs.2019.11.018

[6] Kanmani, P., E. Dhivya, J. Aravind, and K. Kumaresan. "Extraction and analysis of pectin from citrus peels: augmenting the yield from Citrus limon using statistical experimental design." *Iranica Journal of Energy & Environment* 5, no. 3 (2014): 303-312. https://doi.org/10.5829/idosi.ijee.2014.05.03.10

[7] Canteri-Schemin, Maria Helene, Heloísa Cristina Ramos Fertonani, Nina Waszczynskyj, and Gilvan Wosiacki. "Extraction of pectin from apple pomace." *Brazilian Archives of Biology and Technology* 48 (2005): 259-266. https://doi.org/10.1590/S1516-89132005000200013

[8] Kratchanova, M., E. Pavlova, and I. Panchev. "The effect of microwave heating of fresh orange peels on the fruit tissue and quality of extracted pectin." *Carbohydrate Polymers* 56, no. 2 (2004): 181-185. https://doi.org/10.1016/j.carbpol.2004.01.009

[9] Zainuddin, Nur Ain Mohd, Farahhanis Tuah, and Mohd Azahar Mohd Ariff. "Effect of Pressure on Essential Oil Yield of Chromolaena Odorata Leaves Extract using Supercritical Fluid Carbon Dioxide." *Journal of Advanced Research in Applied Sciences and Engineering Technology* 17, no. 1 (2019): 29-35.

[10] Wahab, Izzati Mohamad Abdul, Mariam Firdhaus Mad Nordin, Nabilah Zaini, Kamyar Shameli, Siti Nur Khairunisa Mohd Amir, Nurul'Uyun Ahmad, and Norrashidah Mokhtar. "A Comparative Study on Zerumbone Concentration, Radical Scavenging Activity and Total Phenolic Content of Zingiber Zerumbet Extracted via Green and Conventional Extraction." *Journal of Advanced Research in Applied Sciences and Engineering Technology* 27, no. 1 (2022): 1-8. https://doi.org/10.37934/araset.27.1.18

[11] Wiboonsirikul, Jintana, and Shuji Adachi. "Extraction of functional substances from agricultural products or by-products by subcritical water treatment." *Food Science and Technology Research* 14, no. 4 (2008): 319. https://doi.org/10.3136/fstr.14.319

[12] Swamy, Gabriela John, A. Sangamithra, and V. Chandrasekar. "Response surface modeling and process optimization of aqueous extraction of natural pigments from Beta vulgaris using Box-Behnken design of experiments." *Dyes and Pigments* 111 (2014): 64-74. https://doi.org/10.1016/j.dyepig.2014.05.028

[13] Rasidek, Noor Azwani Mohd, Mariam Firdhaus Mad Nordin, Hideaki Tokuyama, Yuichiro Nagatsu, Norlisa Mili, Ahmad Syahmi Zaini, Zuhaili Idham, and Mohd Azizi Che Yunus. "Subcritical water-based pectin from banana peels (Musa paradisiaca cv. Tanduk) as a natural gelation agent." *Materials Today: Proceedings* 47 (2021): 1329-1335. https://doi.org/10.1016/j.matpr.2021.02.815

[14] Swamy, Gabriela John, and Kasiviswanathan Muthukumarappan. "Optimization of continuous and intermittent microwave extraction of pectin from banana peels." *Food Chemistry* 220 (2017): 108-114. https://doi.org/10.1016/j.foodchem.2016.09.197

[15] Pasandide, Bahare, Faramarz Khodaiyan, Zeinab E. Mousavi, and Seyed Saeid Hosseini. "Optimization of aqueous pectin extraction from Citrus medica peel." *Carbohydrate Polymers* 178 (2017): 27-33. https://doi.org/10.1016/j.carbpol.2017.08.098

[16] Le Man, H., S. K. Behera, and H. S. Park. "Optimization of operational parameters for ethanol production from Korean food waste leachate." *International Journal of Environmental Science & Technology* 7, no. 1 (2010): 157-164. https://doi.org/10.1007/BF03326127

[17] Koocheki, Arash, Amir Ghandi, Seyed MA Razavi, Seyed Ali Mortazavi, and Todor Vasiljevic. "The rheological properties of ketchup as a function of different hydrocolloids and temperature." *International Journal of Food Science & Technology* 44, no. 3 (2009): 596-602. https://doi.org/10.1111/j.1365-2621.2008.01868.x

[18] Sriamornsak, Pornsak. "Analysis of selected physico-chemical properties of pectin and alginate gels intended for drug delivery." *PhD diss., Charles Sturt University*, 2002.

[19] Owolabi, Rasheed Uthman, Mohammed Awwalu Usman, and Abiola John Kehinde. "Modelling and optimization of process variables for the solution polymerization of styrene using response surface methodology." *Journal of King Saud University-Engineering Sciences* 30, no. 1 (2018): 22-30. https://doi.org/10.1016/j.jksues.2015.12.005

[20] Sangamithra, A., V. Sivakumar, K. Kannan, and Swamy Gabriela John. "Foam-mat drying of muskmelon." *International Journal of Food Engineering* 11, no. 1 (2015): 127-137. https://doi.org/10.1515/ijfe-2014-0139

[21] Wang, Sijin, Fang Chen, Jihong Wu, Zhengfu Wang, Xiaojun Liao, and Xiaosong Hu. "Optimization of pectin extraction assisted by microwave from apple pomace using response surface methodology." *Journal of Food Engineering* 78, no. 2 (2007): 693-700. https://doi.org/10.1016/j.jfoodeng.2005.11.008

[22] Yolmeh, Mahmoud, and Seid Mahdi Jafari. "Applications of response surface methodology in the food industry processes." *Food and Bioprocess Technology* 10, no. 3 (2017): 413-433. https://doi.org/10.1007/s11947-016-1855-2

[23] Maran, J. Prakash. "Statistical optimization of aqueous extraction of pectin from waste durian rinds." *International Journal of Biological Macromolecules* 73 (2015): 92-98. https://doi.org/10.1016/j.ijbiomac.2014.10.050

[24] Le-Pham-Tan, Quoc, Anh Le-Thi-Lan, Tien Mai-Vo-Thi-Kieu, and Trang Le-Thi. "Optimization of the pectin extraction from pomelo peels by oxalic acid and microwave." *Banat's Journal of Biotechnology* 5, no. 9 (2014): 67-73. https://doi.org/10.7904/2068-4738-V(09)-67

[25] Ueno, Hiro, Masahiro Tanaka, Munehiro Hosino, Mitsuru Sasaki, and Motonobu Goto. "Extraction of valuable compounds from the flavedo of Citrus junos using subcritical water." *Separation and Purification Technology* 62, no. 3 (2008): 513-516. https://doi.org/10.1016/j.seppur.2008.03.004

[26] Mao, Yujie, John Robinson, and Eleanor Binner. "Understanding heat and mass transfer processes during microwave-assisted and conventional solvent extraction." *Chemical Engineering Science* 233 (2021): 116418. https://doi.org/10.1016/j.ces.2020.116418