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Microwave-assisted extraction of pectin from queen pineapple (*Ananas comosus L.*) peel

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ABSTRACT

This study attempted to optimize pectin extraction from queen pineapple (*Qp*) peels using microwave-assisted method. A three-factor factorial experimental design was employed to test the interaction of moisture content of pineapple peel, pH of the citric acid solution and extraction time. The three-way interaction among the factors has a significant effect on pectin recovery. Two-way interaction between moisture content and pH level, as well as pH level and extraction time caused significant difference in the pectin recovery. Pectin recovery increases with lower moisture content while longer extraction time does not significantly increase pectin recovery. The optimum combination of the parameters is 12% moisture content, pH level 2 of the citric acid solution, and extraction duration of 2 min, which yielded $1.01 \pm 0.01\%$ pectin recovery. This is lower than the results from previous studies on pineapple with 3.88–13.06% pectin recovery using acid extraction method. Hence further optimization is recommended.

KEYWORDS

queen pineapple, pineapple, pectin, microwave-assisted extraction, moisture content, pH level, extraction time

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INTRODUCTION

In 2021, Philippines ranked 3rd among the top pineapple producing countries in the world, closely following Costa Rica and Indonesia at 1st and 2nd, respectively. Its total production for that year was approximately 2.8 million metric tons (Shabandeh, 2023). Majority (57.6%) of the production is from Northern Mindanao, followed by SOCCSKSARGEN and Bicol Region with 33.3% and 5.5% shares, respectively (Philippine Statistics Authority, 2023). The two varieties of pineapple grown in the country are the Sweet Cayenne and the Formosa, the former being processed by multinational companies such as DOLE and Del Monte while the latter is more commonly known as queen pineapple (*Qp*) (Mabeza and Pili, 2005).

Among these major contributors to pineapple production, only the province of Camarines Norte in the Bicol Region traditionally grows the queen pineapple variety. Based on actual data from Labo Progressive Multi-Purpose Cooperative (LPMPC) in Camarines Norte, which processes the queen pineapple into bottled fruit juice, about 40% of the fruit, specifically its pulp, peel, and eye trimmings end up as waste during processing.

Apart from solving the issue of waste management in pineapple processing plants, being able to produce useful products from these waste materials and generate additional income is highly desired. Among the useful products that can be extracted from pineapple peel is pectin. Arollado et al. (2018) reported that Philippines imports 100% of its pectin requirement from other countries, making the product expensive for industries that are utilizing it. The government imported 94,848.93 kg of pectin from various sources in 2011, totaling US$ 52,383,487 at an average landed cost of US$ 490 per kilogram (Caliguiran, 2014). Moreover, it is also noteworthy that the wide gap between production and utilization of pectin is generally noted on a global scale (Venkatanagaraju et al., 2019). Hence, this is a good reason to invest in research on pectin extraction from locally available sources in the Philippines.

Srivastava and Malviya (2011) and (Meena et al., 2022) describe pectin as a structural heteropolysaccharide found in the main cell walls of terrestrial plants, specifically fruits and vegetables. It is a white to light brown powder that is professionally made from citrus fruits like sweet lemon peel (Devi et al., 2014), kiwi peel (Karbuz and Tugrul, 2021), mandarin (Karbuz and Tugrul, 2021), grape pomace (Spinei and Oroian, 2022) and pomelo (Mahmud et al., 2021), and is used in cuisine as a gelling agent, particularly in jams and jellies. It is also utilized in fillings, and confectionery, as a fruit juice and milk drink stabilizer, and as a dietary fiber source. It is also used in pharmaceutical industries as bioactive component and in biomedical application such as drug delivery, tissue engineering and wound healing (Robledo and Vázquez, 2019).

In the industry, pectin is generally extracted using strong acid solutions under the heating environment with hydrochloric acid, sulfuric acid, nitric acid or phosphoric acid, which can be harmful to the environment when improperly disposed (Alekseev et al., 2020). However, due to the environmental risks and the process cost involved, novel innovative extraction technologies have been recently developed (Robledo and Vázquez, 2019). One of which is the microwave-assisted extraction (MAE) method (Alekseev et al., 2020) where microwave energy is absorbed by the extraction system, which results in thermal accumulation that causes dissolution of pectin into the extraction solution (Karbuz and Tugrul, 2021). In MAE, extraction is accomplished at higher temperature but in a shorter time, thereby resulting in higher extraction rates, less solvent consumption making it more efficient and less costly (Sharma et al., 2020). This method of pectin extraction has been employed for different fruit peels like that of mango

(Wongkaew et al., 2021), dragon fruit (Tongkham et al., 2017), jackfruit rinds (Koh et al., 2014), orange (Kachare et al., 2023; Wijana et al., 2023), grapefruit (Kumar et al., 2020) and banana and papaya (Mada et al., 2022), to name a few.

Considering the relevance of queen pineapple as a priority commodity in the province of Camarines Norte and the wastes generated from discarded peel, determining the amount of useful product that can be recovered from it is worthy of investigation. The general objective of this study was to optimize pectin extraction from queen pineapple (*Qp*) peels using the microwave-assisted method of extraction. In accomplishing that, this study also determined the effect of factors such as: (1) moisture content of dried *Qp* peel; (2) pH level of the citric acid solution; and (3) extraction time on the percentage of pectin extracted. As a result, the best combination of these parameters for pectin extraction was obtained.

MATERIALS AND METHODS

Research design

A three-factor factorial experiment was employed where the three factors had three levels each, as follows: Factor A – moisture content of dried *Qp* peel (20, 13.5, and 12% wet basis); Factor B – pH level of the citric acid solution (1, 2 and 3); and Factor C – extraction time (2, 3 and 4 min). The MC levels were selected in order to compare dry (12 and 13.5%) and relatively wet (20%) pineapple peel. The close interval between 12 and 13.5% was chosen in order to test if the 13.5% MC level would yield comparable results with 12% MC. The pH levels and extraction time levels used in this study were based on previous works on microwave-assisted extraction of Karbuz and Tugrul (2021) and Kachare et al. (2023), respectively. These resulted in 27 treatment combinations and two replicates of each were done, giving a total of 54 experimental units.

Experimental procedure

The *Qp* peels were collected from the waste product of Labo Progressive Multi-Purpose Cooperative (LPMPC) in Brgy. Malasugui, Labo Camarines Norte, Philippines. The peels were cut to 1.0 cm thickness and 6.25 cm^2 in cross-sectional area and were washed with clean potable water to remove the remaining pulp deposits. Queen pineapple peel was dried inside a cabinet-type electrical dryer set at a drying temperature of 60 °C to obtain final moisture content of 20, 13.5, and 12%, wet basis. The dried *Qp* peel was ground using a grinding machine that employs a 1.5 hp electric motor and sieved through a mesh-30 screen (hole diameter = 0.60 mm).

Microwave-assisted extraction (MAE) was performed using a 700-W microwave oven. Prior to MAE, 400 mL of distilled water was poured into a 500-ml beaker and pH was adjusted to pH 1, 2, and 3 by adding citric acid powder. Once the desired pH was obtained, 40.0 ± 0.05 g powdered *Qp* peel was added to the citric acid solution and then stirred. Subsequently, the solution was placed on the turntable of the microwave oven set at the maximum setting. The solution remained in the oven for 2, 3, and 4 min following the treatment combinations.

After microwave-assisted extraction, the solution was allowed to cool down at room temperature of 26 ± 2 °C before filtering using two layers of clean cheesecloth. The filtrate was rinsed with an equivalent volume of 95% pure ethanol and allowed to sit for one hour.

The sample was then filtered using Grade 1 qualitative filter paper. The filtrate was rinsed twice with 95% (v/v) ethanol before the coagulated pectin mass was attained. The coagulated pectin was oven-dried at 65 °C for 8 h and weighted after cooling. The dried sample was ground into fine powder, weighed and stored in a zip lock plastic bag.

The pectin recovery (%) from *Qp* peel was calculated following the equation (Rivadeneira et al., 2020)

$$Pectin\ Yield\ (\%) = \left(\frac{m_{cp}}{m_{dp}}\right) x\ 100\%$$

where:

m_{cp} = mass of extracted pectin, g
m_{dp} = mass of dried *Qp* peel, g

The interaction effect and main effect of the three factors were analyzed through analysis of variance (ANOVA) using Statistical Tool for Agricultural Research (STAR) software (International Rice Research Institute, 2013). Response surface model (RSM) was employed to determine the optimum combination of the factors that influence pectin extraction. RSM was done through Design Expert Statistical Software (Stat Ease, Inc.)

RESULTS AND DISCUSSION

Table 1 shows the average pectin recovery (%) for each treatment combination tested in the three-factor factorial experiment. Interaction and main effects were analyzed using ANOVA and the results are discussed in the succeeding sections.

Interaction and main effects

ANOVA (α = 0.05) (Table 2) revealed that the three-way interaction ($p = 0.04$) between moisture content, pH level, and extraction time significantly affected pectin recovery. This means that there is a two-way interaction that varies across levels of a third variable. For example, across different levels of extraction time, the two-way interaction of moisture content and pH level significantly affects pectin recovery ($p = 0.04$). Similarly, across different levels of moisture content, the interaction effect of pH level and extraction time is significant ($p = 0.04$). However, the interaction of moisture content and extraction time ($p = 0.06$) did not have significant effect on the pectin recovery. ANOVA further revealed that the main effect or individual effect of moisture content of the *Qp* peel ($p = 0.00$) and the pH level of the citric acid solution ($p = 0.04$) are both significant, while extraction time ($p = 0.18$) has no significant effect on pectin recovery. These effects are better illustrated in Fig. 1 and are discussed in the succeeding sections.

Figure 1 shows the interaction effect of moisture content and pH and their corresponding main effect on pectin recovery. Specifically, Fig. 1 illustrates that higher pectin recovery is obtained when the pH of the citric acid solution is at 2 and when the pineapple peel was dried to 12% prior to extraction process. For instance, Fig. 1a demonstrates the main effect of moisture content. In most of the experiments at any extraction time and any pH level, the amount of

Table 1. Average pectin recovery at varying treatment combinations

Moisture content (%)	Solvent pH	Extraction time (mins)	Average pectin recovery (%)
20	1	2	0.71 ± 0.06
20	2	2	0.43 ± 0.01
20	3	2	0.24 ± 0.00
20	1	3	0.11 ± 0.04
20	2	3	0.32 ± 0.01
20	3	3	0.27 ± 0.02
20	1	4	0.11 ± 0.04
20	2	4	0.42 ± 0.02
20	3	4	0.35 ± 0.04
13.5	1	2	0.19 ± 0.02
13.5	2	2	0.73 ± 0.01
13.5	3	2	0.07 ± 0.02
13.5	1	3	0.32 ± 0.00
13.5	2	3	0.36 ± 0.02
13.5	3	3	0.10 ± 0.02
13.5	1	4	0.34 ± 0.01
13.5	2	4	0.47 ± 0.03
13.5	3	4	0.08 ± 0.00
12	1	2	0.48 ± 0.04
12	2	2	1.01 ± 0.07
12	3	2	0.38 ± 0.06
12	1	3	0.90 ± 0.03
12	2	3	0.69 ± 0.05
12	3	3	0.60 ± 0.07
12	1	4	0.26 ± 0.04
12	2	4	0.70 ± 0.07
12	3	4	0.48 ± 0.01

Table 2. Significance of interaction and main effects on the pectin recovery as per ANOVA ($\alpha = 0.05$)

Source	DF	Sum of square	Mean square	F value	*P*-value
MC (Moisture content)	2	0.89	0.45	8.03	0.00*
pH level	2	0.42	0.21	3.80	0.04*
Extraction time	2	0.20	0.10	1.80	0.18
MC : pH level	4	0.64	0.16	2.88	0.04*
MC : Extraction time	4	0.15	0.04	0.67	0.61
pH Level : Extraction time	4	0.63	0.16	2.81	0.04*
MC : pH Level : Extraction time	8	1.10	0.14	2.47	0.04*
Error	27	1.51	0.06		
Total	53	5.55			

*There is significant difference at $\alpha = 0.05$.

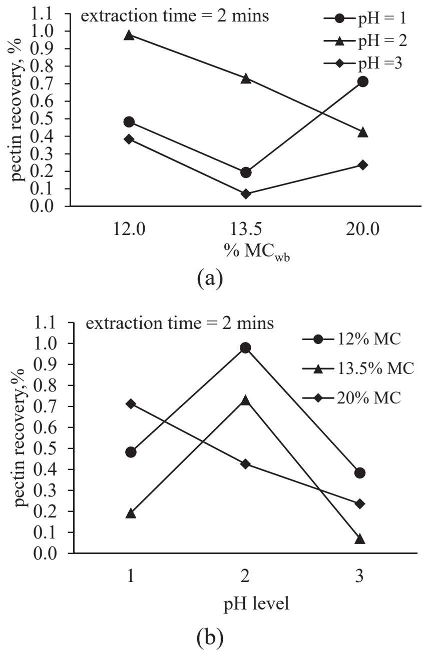

Fig. 1. Pectin recovery (%) at varying combination of moisture content of *Qp* peel and pH of the citric acid solution

pectin extracted from the *Qp* peels is higher at low moisture content of 12%. This is potentially due to the weakening of the structural strength of the extractable material when agricultural product is dried at lower moisture level (Johnson, 2008). This makes drying an important unit operation that aids in the extraction of certain chemical components from a material (Badmus et al., 2019; Markhali, 2021). Saifullah et al. (2019) even elaborated that drying method and conditions may also affect the characteristics and composition of extracted components. However, investigating the composition of the extracted pectin is beyond the scope of this work.

Meanwhile, Fig. 1b illustrates the main effect of pH level. In most of the experiments at any extraction time and moisture content (with Fig. 1b as example), the pectin recovery from the *Qp* peels is higher when the pH of the citric acid solution is maintained at 2. This is further supported by the graph shown in Fig. 2b showing that when fruit peels were dried to 13.5–12% MC, pH level of 2 yielded the highest percentage of extracted pectin. This observation on the effect of pH agrees with that of Zakaria et al. (2021) where pH 2.0 resulted in higher pectin yield through microwave-assisted extraction method while higher pH caused reduction in the amount of pectin extracted. It was explained that this level of pH is sufficient to provide high concentration of hydrogen ions that stimulate the solubilization of insoluble pectin into the solvent (in this case, the citric acid solution). Even in microwave-assisted extraction of pectin from mixed banana and papaya mixed peels, it was also shown that pectin yield is higher at pH 2.0 (Mada et al., 2022). Similarly, pH 2 in the work of Karbuz and Tugrul (2021) with various fruit peels, also yielded the best pectin recovery as compared with pH 1 and 3. It was also observed

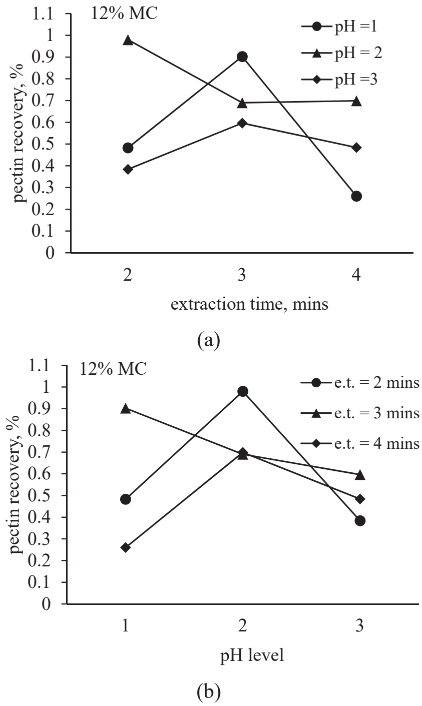

Fig. 2. Pectin recovery (%) at varying combination of extraction time inside the microwave oven and pH of the citric acid solution

that precipitation of pectin occurred at pH higher than 2. In contrast (Hamidon and Zaidel, 2017), observed that pH lower than 2 yielded higher pectin recovery from sweet potato but generally observed that higher pH reduces pectin yield.

The main effects of each of the factors considered are illustrated in Fig. 3. ANOVA (Table 2) revealed that moisture content has significant effect on the pectin recovery and Fig. 3 shows that higher pectin recovery is achieved at lower moisture content of 12%, as compared to 13.5% and 20%. Meanwhile, higher pectin recovery was obtained at samples with pH level of 2 than pH levels 1 and 3. Finally, for extraction time, ANOVA revealed that it did not have any significant effect on pectin recovery. The insignificant effect of extraction time agrees with Zakaria et al. (2021) who observed that increasing the irradiation time (i.e. extraction time inside the oven) from 2.5 to 20 min did not have significant effect on the pectin yield, concluding that longer extraction time is not necessary in microwave-assisted extraction of pectin. Consequently, this is favorable as less time of extraction means less energy consumption of the whole process. Moreover, it was explained that unnecessary extension of extraction time may cause simultaneous extraction of compounds other than pectin or may potentially destroy the pectin molecules chain (Mandal et al., 2007). It is also worth noting that even in the acid extraction method employed by Hamidon and Zaidel (2017) for sweet potato, prolonging extraction time beyond 60 min is not recommended as it might induce the digestion of pectin making it difficult to be precipitated by pectin. Hence,

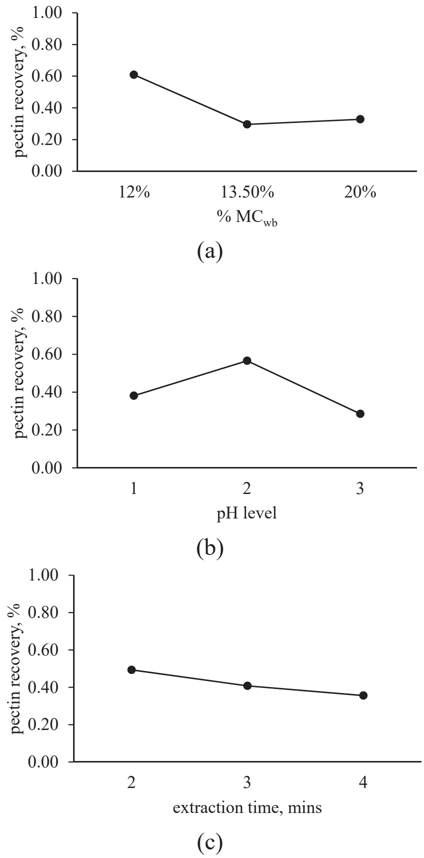

Fig. 3. Main effect of moisture content of *Qp* peel, pH level of citric acid solution and extraction time inside the microwave oven

extraction time even in microwave-assisted method of extraction should be optimized to avoid unnecessary energy spending. Considering that the above results suggest that the effect of microwave is evident even in the shortest time, its threshold value should be determined. Therefore, extraction time shorter than 2 min can be explored in future studies.

The above discussion is further supported by the contour plots generated through Response Surface Methodology shown in Fig. 4. The yellow-green portion of the contour plots is the region where higher pectin yield is observed, which are primarily the region corresponding to pH of 2 and moisture content of 12%. The highest pectin yield was measured to be approximately 1.0%, which is still lower than 2.27–2.79% obtained by Zakaria et al. (2021) who employed MAE on pineapple peel. However, the variety of pineapple used by Zakaria et al. (2021) was not specified, hence the limited comparison. Meanwhile, the 1.0% pectin yield obtained from the present work involving queen pineapple peel is also way below the 3.88–13.06% pectin recovery measured by Karim et al. (2014) and 11.24% by Sarangi et al. (2020) who both employed the conventional acid extraction method followed by ethanol precipitation.

Response surface plot analysis

Figure 4 demonstrates the relationship between moisture content, pH level, and pectin yield at different extraction time. At any extraction time, the pectin yield approaches a higher value (yellow-green portion of the contour plot) at a treatment with 12% MC and pH level 2, in accordance with the preceding discussion. Moreover, it is apparent that lower pectin recovery is achieved at higher MC and at pH levels 1 and 3, as shown by the blue portion of the contour plots.

Figure 5 demonstrates that the pectin yield is not affected by the interaction of moisture content and extraction time. At any pH level, any combination of moisture content and extraction time yields a similar amount of pectin. It is again evident in Fig. 5 that at pH level 2, the pectin recovery values were at a medium-high level (yellow-green) as compared to pH levels 1

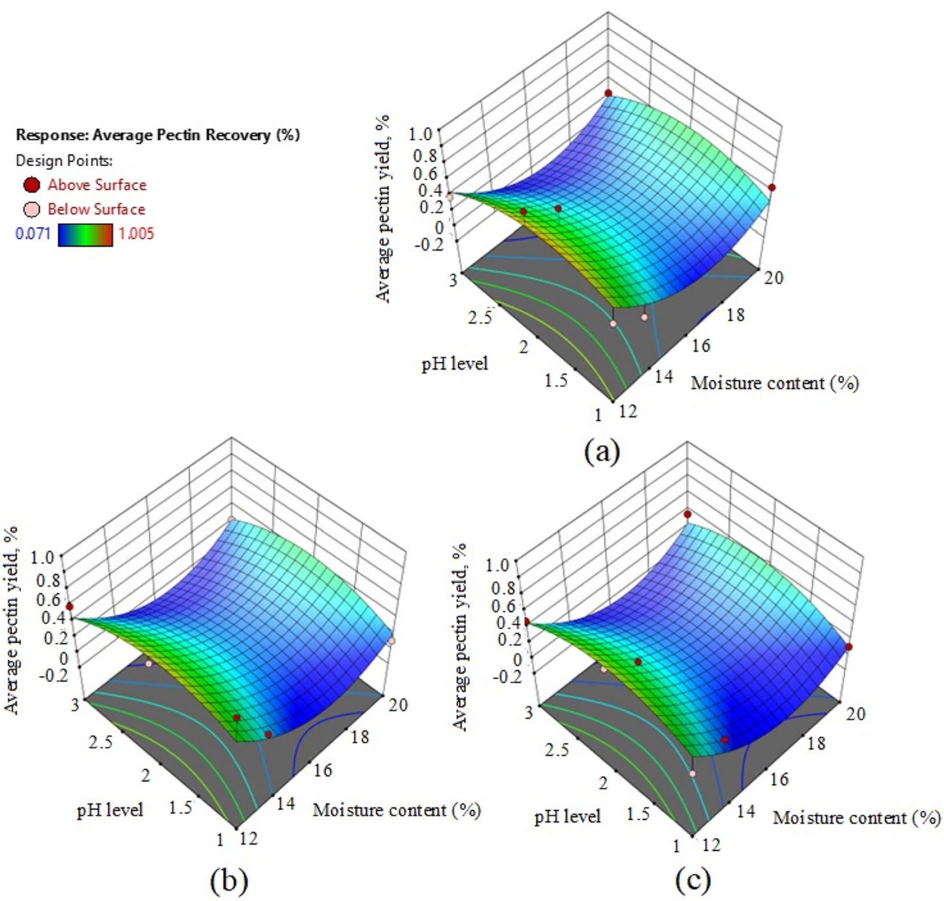

Fig. 4. Contour plots showing average pectin yield as affected by the interaction of moisture content and pH level at extraction time of (a) 2 min (b) 3 min and (c) 4 min

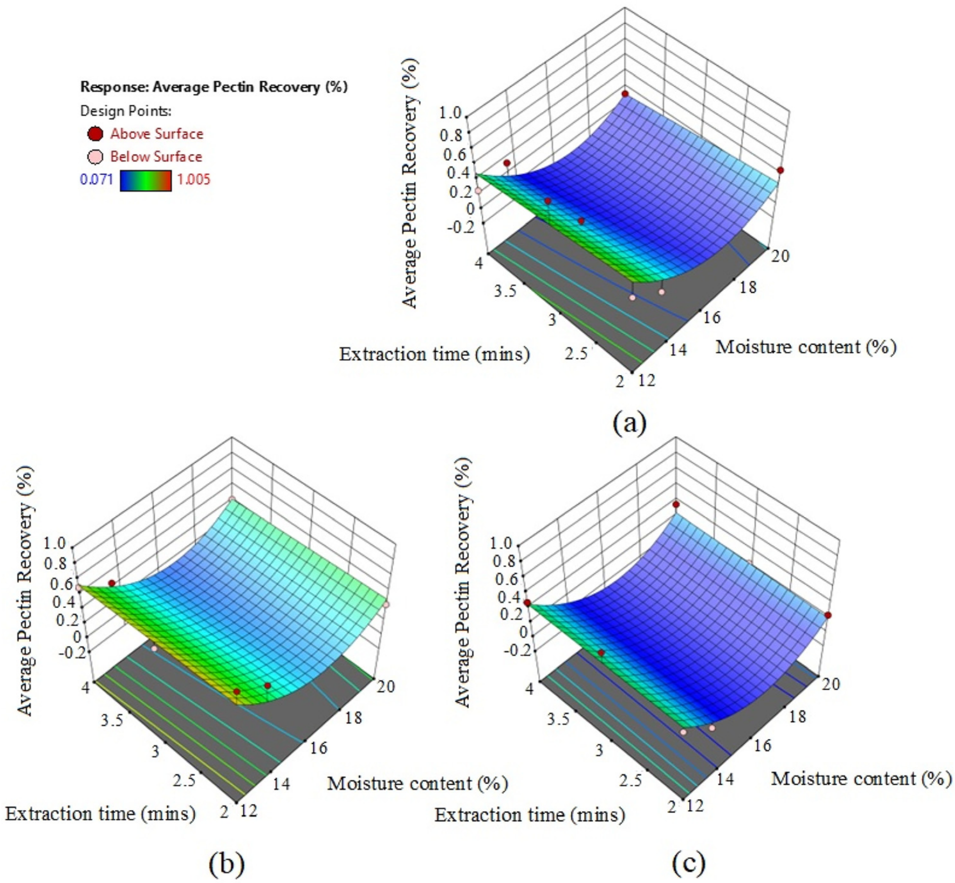

Fig. 5. Contour plots showing average pectin yield as affected by the interaction of moisture content and extraction time at pH level (a) 1 (b) 2 and (c) 3

and 3 where contour plots were shown to be dominated by blue color, indicating low pectin recoveries.

Figure 6a shows the relationship between extraction time and pH level when the moisture content was kept constant at 12%. In the response surface plot, a higher yield of pectin was observed at pH level 2 and extraction time of 2–3 min while at the pH level of 3 with extraction time of 4 min, the yield of pectin was lower. Figure 6b shows the relationship between the extraction time and pH level when the moisture content was kept constant at 13.5%. In this response surface plot, a higher yield of pectin was observed at pH level 2 and extraction time of 2–3 min while at the pH level of 1 and 3 with extraction time of 4 min, the yield of pectin was lower. Similarly, Fig. 6c shows the relationship between the extraction time and pH level when the moisture content was kept constant at 20%. In response surface plot, a higher yield of pectin was observed at pH level 2 and extraction time of 2–3 min while at the pH level of 1 with extraction time of 4 min, the yield of pectin was lower.

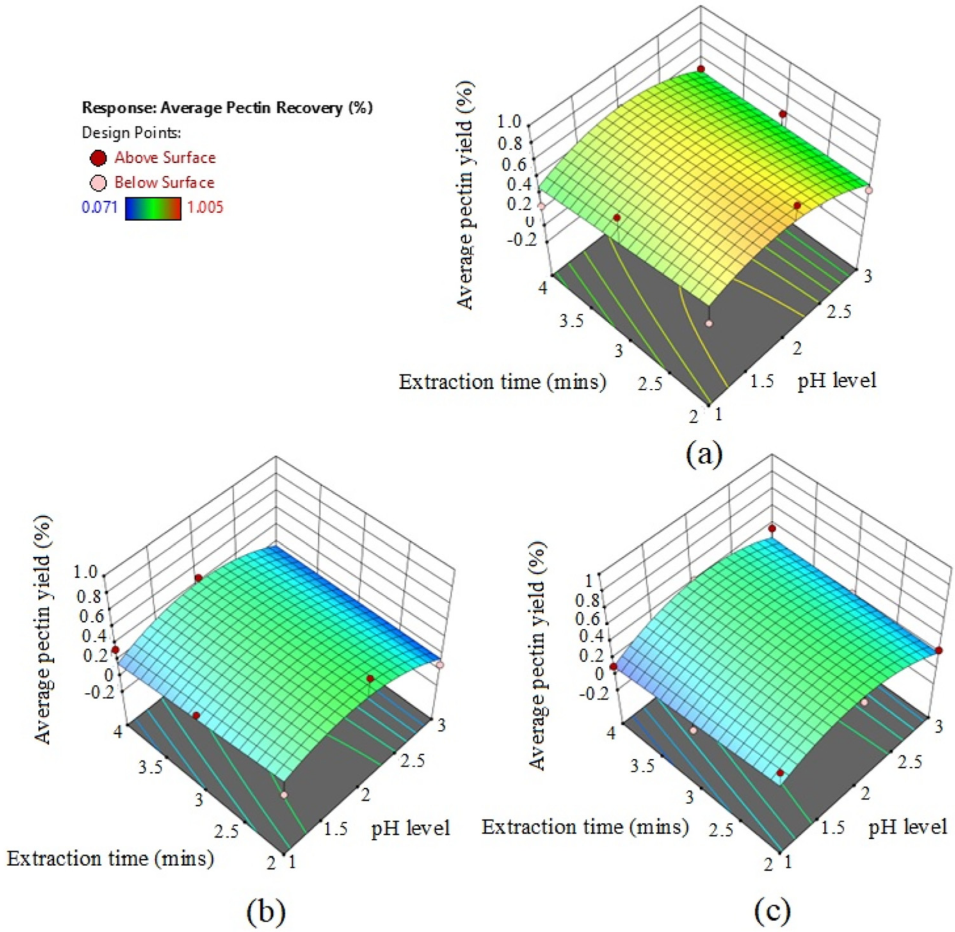

Fig. 6. Contour plots showing the average pectin yield as affected by the interaction of extraction time and pH level at moisture content of (a) 12% (b) 13.5% and (c) 20%

Optimum combination

A factorial experiment was executed to determine the key factors for the microwave-assisted extraction of pectin from queen pineapple peel. Overall, the best combination of the above parameter is a moisture content of 12%, pH level of 2, and 2-min extraction time. Such combination can yield pectin equivalent to approximately 1% of the initial weight of dried queen pineapple peel. This amount of pectin recovery is relatively low as compared to the 2.27–2.70% pectin recovery obtained by Zakaria et al. (2021) who also employed MAE on pineapple peel. The 1% pectin recovery obtained from this study is also significantly lower than the 3.88–13.06% pectin recovery measured by Karim et al. (2014) and 11.24% by Sarangi et al. (2020) who both employed the conventional acid extraction method followed by ethanol precipitation.

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

The study was conducted to optimize pectin extraction from queen pineapple peels using microwave-assisted method of extraction. A three-factor factorial experiment was employed to investigate the effect of moisture content of queen pineapple peel, pH of the citric acid solution and extraction time inside the microwave oven and to determine their optimum combination. Results showed that the interaction of the three factors significantly affected pectin recovery. Analysis showed that the best combination of the parameters is at a moisture content of 12%, pH level 2 using citric acid solution, and extraction duration of 2 min.

Considering the low pectin recovery obtained in this study compared to other pectin extraction studies on pineapple, further optimization using other parameters may be done in future studies. The effect of microwave power can be investigated and other acids can be used for the extraction process. Furthermore, to confirm that the high energy of the microwave caused the extraction of pectin and not the high temperature, control experiments employing high temperature (with no microwave) may be conducted. Moreover, extraction time shorter than 2 min can also be explored in future experiments as it was shown in the current study that the effect of microwave seems to be effective in extracting pectin even at a short time of 2 min. In addition, taking into consideration the favorable effect of lower moisture content in improving the extraction yield of pectin, future work can investigate the effect of drying method on the pectin yield of queen pineapple and other pectin sources from agricultural wastes. Moreover, the researchers recommend that the pectin from *Qp* peel be characterized in terms of its physico-chemical properties.

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