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ULTRASOUND ASSISTED EXTRACTION OF PECTIN FROM BANANA PEEL WASTE AS A POTENTIAL SOURCE FOR PECTIN PRODUCTION

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ABSTRACT

Background. Large quantities of banana peels are discarded as waste or used as cattle feed or fertilisers. Moreover, the use of banana peel waste is considered to be a valuable polysaccharide like pectin that is valued by both pharmaceutical and food industries. It is well known that pectin is a high-value functional food ingredient widely used due to its special functional properties.

Materials and methods. Pectin was extracted from banana peel waste using ultrasound assisted extraction (UAE) and the extraction was optimised by response surface methodology. The independent variables such as extraction temperature, sonication time and pH of a solution were used in the range of 35–45°C, 10–20 min, and 1–3, respectively.

Results. The overall optimal conditions of multiple responses were achieved at an extraction temperature of 33.12°C, sonication time of 17.12 min and pH of 3.68. Under these conditions, the predicted value of pectin yield, the degree of esterification and galacturonic acid content, were 2.62%, 88.26% and 87%, respectively. The experimental values were well correlated with the predicted values and revealed a satisfactory validation through real-time experiments, which was accomplished at optimised conditions. The physico-chemical properties of extracted pectin using optimal conditions were comparable to commercial pectin.

Conclusion. The results obtained revealed that pectin was effectively extracted from the banana peel waste using UAE, which confirmed that it has potential as a source for the food, beverage and pharmaceutical industries. Further studies should be conducted to improve the quality of pectin and explore the bioactivity potential of pectin such as antimicrobial, prebiotic and antioxidant properties.

Keywords: banana peel, extraction, optimisation, pectin, response surface methodology, ultrasound assisted extraction

INTRODUCTION

Banana (*Musa sapientum* L.) is an important traded fruit in tropical countries. The three top countries that produce bananas are India, China and Indonesia, respectively (FAOSTAT, 2020). Banana is consumed not only in unripe form but also converted into various products such as banana crisps, chips, flour, puree, etc. (Oliveira et al., 2016). Large quantities of banana peels were discarded as waste or used as cattle feed or fertilisers. Moreover, the use of banana peel waste is considered to be a valuable polysaccharide like pectin

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that are valued by both pharmaceutical and food industries (Cordenunsi et al., 2008; Rebello et al., 2014).

Pectin is a non-starch heteropolysaccharide found in plant cell walls (Carpita and McCann, 2000; Willats et al., 2001). It is well known that pectin is a high-value functional food ingredient widely used due to its special functional properties such as water binder, stabiliser, thickener and gelling agents which can be used in many food products such as jellies, juices, confectionery and yogurt (Kratchanova et al., 2004; May, 1990; Wai et al., 2010). Furthermore, pectin has been used in cosmetic and pharmaceutical industries (Zitko and Bishop, 1966).

The extraction of pectin is an important process. Previous studies have reported that pectin extraction by using conventional methods was time-consuming, exhausted a lot of energy and that it could produce pectin degradation (De Oliveira et al., 2015; Seixas et al., 2014). Therefore, the need to replace conventional extraction with a better method could be a potential extraction to minimise the aforementioned adverse effects. The extraction of pectin yield and pectin composition depends on the extraction conditions used during the pectin extraction procedure and purification steps (Levigne et al., 2002).

Ultrasound assisted extraction (UAE) is widely used in the food industry and has been accepted as a proficient, clean and green extraction method. Furthermore, it has many advantages, such as: it reduces the solvent used, quickens liberation of recoverable substances, increases the mass transfer, improves better-quality products with less degradation (Al-Dhabi et al., 2017; Moorthy et al., 2017) and improves functional properties of food (Šic Zlabur et al., 2015). In recent years, the UAE has been reported in the extraction of pectin from natural sources, such as tomato waste (Ninčević Grassino et al., 2016), grape pomace (Minjares-Fuentes et al., 2014), grapefruit peel (Wang et al., 2015) and jack fruit peel (Moorthy et al., 2017), for use in the food systems.

Maran et al. (2017) reported the extraction of pectin from *Musa balbisiana* by the UAE with citric acid. The result exhibited that the UAE with a weak acid was effective in pectin yield (8.99%) with an optimal extraction time of 27 min, pH of 3.2 and power of 323 W. Furthermore, the extraction variables, time and temperature had an effect on the pectin yield and chemical compositions or quality of extracted pectin from the banana peels (Phaiphan, 2019; Phaiphan et al., 2020). Qiu et al. (2010) showed that a higher salting out temperature and extensive extraction time had affected the pectin degradation, thus affecting the pectin extraction yield. Similarly, Oliveira et al. (2016) highlighted that a harsh temperature and pH value increased extraction yield but decreased a degree of methoxylation (from 79% to 43%). Happi Emaga et al. (2008) also reported that using lower pH values had affected pectin composition, such as galacturonic acid content, but it promoted the yield of pectin.

However, only limited reports were available on the conditions of the UAE method for pectin extraction from banana peel waste. In this research, the pectin from the banana peel (*Musa sapientum* L.) was extracted under UAE. Therefore, the main aim of this study was to develop and optimise the influence of pectin extraction parameters (extraction temperature, sonication time and pH of a solution) from banana peels on maximal extraction yield and high quality of pectin compositions (degree of esterification and galacturonic acid) by using three levels of the three-factor central composite design under the response surface methodology (RSM).

MATERIALS AND METHODS

Materials and sample preparation

Samples of banana peels (*Musa sapientum* L.) were purchased from a local vendor (Ubon Ratchathani, Thailand). The samples were washed, cut into small slices (1.5 cm × 3 cm) and boiled in 95% of ethanol solution (banana peel/solvent ratio 1:1) at a temperature of 80°C for 10 min followed by washing three times with distilled water. Then, the banana peels were dried in a hot air oven at 65°C and were ground in a mechanical grinder (the final moisture content of banana powder was 3.54 ±0.18%). Thereafter, the dried peels were passed through an 80 mesh pore size sieve. The powdered banana peel was kept in a glass bottle and was stored in a freezer (–18 ±1°C) until further experiments.

Pectin extraction using ultrasound assisted extraction (UAE)

Pectin extraction from the banana peel waste was performed using the ultrasonic bath (Branson, model 5510, USA), which was operated at a frequency of 40 kHz

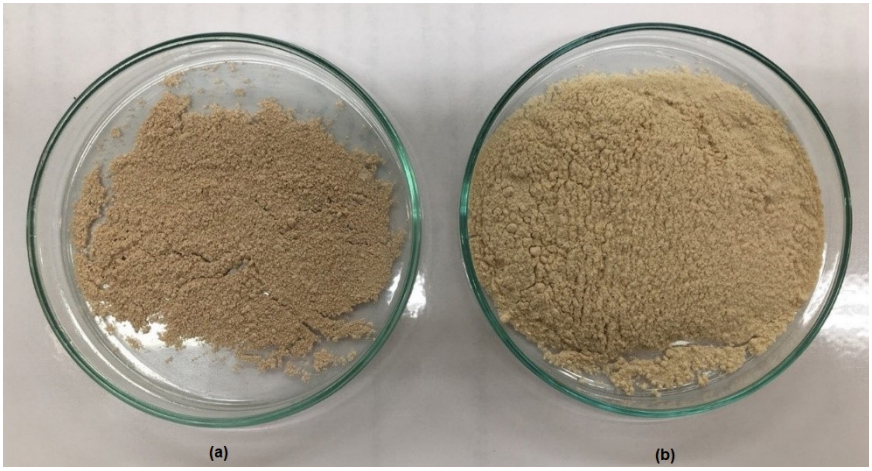

Fig. 1. Dried pectin: **a** – dried pectin (banana peel) extracted by UAE, **b** – dried commercial (citrus) pectin; ΔE value or total colour difference between **a** and **b** was 6.68

and maximum input power of 185 W. The pectin extraction was carried out according to the method described by Li et al. (2012), with slight modifications. Ten grams of dried peel samples of *Musa sapientum* L. were added into 0.05 M hydrochloric acid (dried peel sample/solvent ratio 1:12, W/V). The mixture was adjusted to the desired pH values (pH 1, 2 and 3). Thereafter, the mixture was extracted with three extraction temperatures of 35°C, 40°C and 45°C for three sonication times of 10 min, 15 min and 20 min (based on the experimental conditions of pectin extraction as presented in Table 1), whereby all conditions were selected after preliminary experiments. After extraction, the mixture was centrifuged at a speed of 4,500 rpm for 30 min. Then, 95% of ethanol was added into the supernatant (solvent/filtrate ratio 1:1, V/V) for sample precipitation and the mixture was stored at room temperature for 15 hr. Thereafter, the precipitated pectin was separated by using a filter and cleansed three times with 95% of ethanol followed by 50% of acetone, respectively. Next, the extracted pectin was dried in a temperature-controlled oven at 60°C until it obtained a stable weight. The dried sample was ground into powder and the percentage of pectin extraction yield (%) was calculated as described by Phaiphan et al. (2020).

Degree of esterification (DE) and methoxyl content

The degree of esterification (DE) of extracted pectin from the banana peels was done by the titrimetric method as described by Hosseini et al. (2016), with slight modifications. Briefly, 300 mg of dried pectin were moistened with 2 mL of ethanol and thereafter

Table 1. Experimental factors and their levels used in the central composite design

Independent variable	Code unit	Coded level				
		α (–1.682)	–1	0	1	α (+1.682)
Extraction temperature, °C	X_1	31.59	35	40	45	48.41
Sonication time, min	X_2	6.59	10	15	20	23.41
pH of solution	X_3	0.32	1	2	3	3.68

100 mL of distilled water was added to dissolve the sample completely. The mixture was mixed thoroughly and four drops of phenolphthalein were added. Then, the mixture was titrated with 0.5 M of NaOH (the volume of NaOH, Volume 1). Subsequently, 10 mL of 0.5 M NaOH was added and stored for 15 min. Thereafter, 10 mL of 0.5 M HCl was added to the mixture to neutralise the NaOH and four drops of phenolphthalein were added to the mixture. The mixture was titrated again with 0.5 M NaOH (the volume of NaOH, Volume 2) until it appeared pale pink in colour. The DE (%) of extracted pectin was calculated, as indicated by the formula below and the value of methoxyl was done by using the DE (%) content.

$$\text{Degree of esterification, \%} = \frac{\text{NaOH (Volume 2)}}{\text{NaOH (Volume 1)} + \text{NaOH (Volume 2)}} \times 100$$

Galacturonic acid (GalA) determination

The galacturonic acid (GalA) content was determined according to the colorimetric method as described by Yoo et al. (2006). In brief, 0.1 g of dried pectin was dissolved with 100 mL of 0.05 M NaOH and was stirred for 30 min. Furthermore, 10 mL of sample solution was mixed with 100 mL of distilled water. Then, 2 mL of diluted solution was mixed with 1 mL of 0.1% of Cabazole, followed by adding 12 mL of concentrated H_2SO_4. In the next step, the mixture solution was continuously stirred at room temperature for 25 min for subsequent analysis. Thereafter, the sample and standard solution were recorded for absorbance at 525 nm using UV/Visible spectrophotometer (Biochrom Libra S70, Cambridge, UK). The total GalA content of the sample was calculated by using D-GalA calibration curve. The concentration of standard D-GalA solutions was at 10–100 $\mu g \cdot mL^{-1}$ ($Y = 0.0082X - 0.0071$, $R^2 = 0.9839$).

Determination of total moisture and ash content

The total moisture and ash contents of dried pectin extracts were determined by the gravimetrical procedure of the Association of Official Agricultural Chemists (AOAC) methods (AOAC, 2000). Moisture content was carried out by drying the samples at 105°C until a constant weight was achieved. Ash content was determined by heating the sample in a furnace at 550°C for 6 hr.

Determination of dried pectin colour

The colour of the dried pectin was measured by a colour measurement (Hunter Lab, Colour Flex, VIRG, USA). The parameters of colour measured were L^*, a^* and b^*, where L^* was expressed as lightness (100 is white and 0 is black), a^* expressed as greenness/redness ($+a$ is redness and $-a$ is greenness) and b^* expressed as yellowness/blueness ($+b$ is yellowness and $-b$ is blueness) (Hennessey-Ramos et al., 2021). The following equation was used to calculate the total colour difference (ΔE^*) of pectin extracted by UAE versus the commercial pectin:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

where: ΔL^*, Δa^*, and Δb^* were the differentials between colour parameters of the sample and the standard.

Experimental design

In this study, a central composite design (CCD) was used to construct three experimental factors and three levels of the experimental design. This was to investigate and optimise the effect of experimental factors of extraction temperature (X_1), sonication time (X_2) and pH of a solution (X_3) on the extracted pectin yield, where DE and GalA were the dependent variables. The experimental factors and their levels used in the CCD are shown in Table 1. The value levels for the extraction temperature were 35°C, 40°C and 45°C; for sonication time were 10 min, 15 min, and 20 min; for pH values were 1, 2, and 3. The Minitab software 16.1.1.0 (Minitab Inc., State College, PA, USA) was used to run the statistical tests. The result of this research was analysed with multiple regressions and numerical optimisation.

Model verification

Optimal conditions for UAE on the responses (pectin yield, DE and GalA content) from banana peels, which depended on experimental factors (extraction temperature, sonication time and pH of a solution) were derived from the RSM. Experimental data were carried out for predicted values of optimum extraction conditions. The experimental and predicted values were analysed to affirm the validity of the models by

applying a two-sample t-test. A difference was considered statistically significant when $p < 0.05$.

RESULTS AND DISCUSSION

Statistical analysis

The effects of UAE on the extraction of pectin yield, DE and GalA content were assessed by the RSM using CCD. The CCD is useful in RSM for creating a quadratic model or second order for the responses (dependent variables) without using a full factorial design 3-level (Choon-Hui et al., 2009). Factors coded and decoded levels and responses for the 20 runs of pectin extraction are presented in Table 2. The CCD was used to examine the effect of sonication time, extraction temperature and pH values on the pectin yield, GalA and DE of banana peels. Statistical data analyses were carried out using analysis of

Table 2. Experimental design and results of extraction of pectin from banana peel by UAE

Runs	Independent variables			Responses		
	temperature	time	pH	yield	DE	GalA
1	35 (–1)	20 (1)	1 (–1)	2.15	80.65	90.85
2	35 (–1)	20 (1)	3 (1)	1.97	85.42	91.56
3	40 (0)	15 (0)	2 (0)	1.68	85.73	91.78
4	40 (0)	15 (0)	2 (0)	1.67	84.91	94.52
5	40 (0)	6.59 (–1.68)	2 (0)	1.23	78.57	83.04
6	45 (1)	20 (1)	1 (–1)	3.69	73.33	92.33
7	48.41 (1.68)	15 (0)	2 (0)	2.03	81.48	70.81
8	35 (–1)	10 (–1)	3 (1)	2.02	86.00	79.89
9	35 (–1)	10 (–1)	1 (–1)	2.22	73.17	91.04
10	40 (0)	15 (0)	2 (0)	2.19	80.00	94.93
11	31.59 (–1.68)	15 (0)	2 (0)	2.38	84.21	87.30
12	40 (0)	15 (0)	2 (0)	1.72	82.69	95.81
13	45 (1)	10 (–1)	3 (1)	1.41	78.95	76.00
14	45 (1)	20 (1)	3 (1)	1.82	83.33	77.67
15	40 (0)	15 (0)	2 (0)	1.92	84.62	92.67
16	40 (0)	23.41 (1.68)	2 (0)	2.96	77.59	85.52
17	40 (0)	15 (0)	0.32 (–1.68)	3.74	66.10	97.11
18	45 (1)	10 (–1)	1 (–1)	2.12	79.31	90.85
19	40 (0)	15 (0)	2 (0)	1.85	83.05	98.96
20	40 (0)	15 (0)	3.68 (1.68)	1.89	85.00	85.44

Values in parentheses for independent variables are coded values according to the CCD design.
Yield – pectin yield, %; DE – degree of esterification, %; GalA – galacturonic acid content, %.

variance (ANOVA) and the second order model results are presented in Table 3. The results examined showed that the *p*-values of the model for pectin yield, DE and GalA content were 0.002, 0.009 and 0.000, respectively, which revealed that the fitness of the model was highly significant ($p < 0.05$). Furthermore, the lack-of-fit test did not show any significance ($p < 0.05$) (the *p*-values of lack-of-fit for pectin yield, DE and GalA content were 0.071, 0.11 and 0.213, respectively), which proved that these models could be used to predict the responses. The values of R^2 were 0.87, 0.82 and 0.91 for pectin yield, DE and GalA content, respectively. These R^2 values were higher than 0.750, indicating that these models could explain 87%, 82% and 91% of the responses' variability (Wai et al., 2010). The statistical parameters of the quadratic models for pectin yield, DE and GalA content are presented in Table 4.

The effect of extraction condition on pectin yield

The values of pectin yield ranged from 1.23% to 3.74% (w/w) based on a dry basis as illustrated in Table 2. Both sonication time and pH displayed significantly ($p < 0.05$) linear effects on the yield. The interaction effects of extraction temperature and sonication time or pH had a statistically significant ($p < 0.05$) effect on the yield (Table 3). The effects of the sonication time, pH and extraction temperature on the yield are presented in Figure 2a–2c). The results showed that the yield was found to be increased when the extraction temperature was increased from 35°C to 45°C, whereby the pH (pH 1 to 3) was lowered, as shown in Figure 2. The increase

Table 3. Analysis of variance (ANOVA) for regression model of pectin yield, DE and GalA content from banana peel

Term	Pectin yield					DE					GalA				
	SS	DF	MS	*F*-value	*P*-value	SS	DF	MS	*F*-value	*P*-value	SS	DF	MS	*F*-value	*P*-value
Model	6.99516	9	0.77724	7.56	0.002	409.716	9	45.524	5.02	0.009	993.83	9	110.425	11.01	0.000
X_1 – temperature	0.00061	1	0.00061	0.01	0.940	16.281	1	16.281	1.79	0.210	143.20	1	143.199	14.27	0.004
X_2 – sonication time	1.66569	1	1.66569	16.20	0.002	0.977	1	0.9770	0.11	0.750	25.88	1	25.882	2.58	0.139
X_3 – pH	2.69908	1	2.69908	26.24	0.000	255.114	1	255.114	28.12	0.000	259.90	1	259.896	25.90	0.000
X_1X_1	0.03765	1	0.09527	0.93	0.359	2.985	1	0.093	0.01	0.921	333.22	1	380.145	37.89	0.000
X_2X_2	0.00243	1	0.02593	0.25	0.626	26.768	1	37.089	4.09	0.071	149.76	1	155.862	15.53	0.003
X_3X_3	1.27099	1	1.27099	12.36	0.006	89.979	1	89.979	9.92	0.010	9.59	1	9.585	0.96	0.351
X_1X_2	0.55125	1	0.55125	5.36	0.043	9.031	1	9.031	1.00	0.342	8.67	1	8.674	0.86	0.347
X_1X_3	0.60500	1	0.60500	5.88	0.036	7.920	1	7.920	0.87	0.372	45.46	1	45.458	4.53	0.059
X_2X_3	0.16245	1	0.16245	1.58	0.237	0.661	1	0.661	0.07	0.793	18.15	1	18.150	1.81	0.208
Residual error	1.02846	10	0.10285			90.735	10	9.073			100.33	10	10.033		
Lack-of-fit	0.83058	5	0.16612	4.20	0.071	69.411	5	13.882	3.26	0.11	68.26	5	13.651	2.13	0.213
Pure error	0.19788	5	0.03958			21.324	5	4.265			32.08	5	6.415		
Cor total	8.02362	19				500.451	19				1094.16	19			
R-squared			0.87					0.82					0.91		
Adj. *R*-squared			0.76					0.66					0.83		

SS – sum of squares, DF – degree of freedom, MS – mean square, yield – pectin yield, DE – degree of esterification, GalA – galacturonic acid content.

Table 4. Regression equations and statistical parameters of the models

Equations	F	R^2
$EY = 1.84573 + 0.00669X_1 + 0.34924X_2 - 0.44456X_3 + 0.08131X_1^2 + 0.04242X_2^2 + 0.29698X_3^3 + 0.26250X_1X_2 - 0.27500X_1X_3 - 0.14250X_2X_3$	7.56 ($p = 0.002$)	0.87
$DE = 83.4523 - 1.0919X_1 + 0.2674X_2 + 4.3221X_3 + 0.0804X_1^2 - 1.6042X_2^2 - 2.4987X_3^2 - 1.0625X_1X_2 - 0.9950X_1X_3 + 0.2875X_2X_3$	5.02 ($p = 0.009$)	0.82
$GalA = 94.7136 - 3.2381X_1 + 1.3767X_2 - 4.3624X_3 - 5.1360X_1^2 - 3.2887X_2^2 - 0.8156X_3^2 - 1.0412X_1X_2 - 2.3838X_1X_3 + 1.5063X_2X_3$	11.01 ($p = 0.000$)	0.91

EY – extraction yield, DE – degree of esterification, GalA – galacturonic acid content, X_1 – temperature, X_2 – sonication time, X_3 – pH of solution.

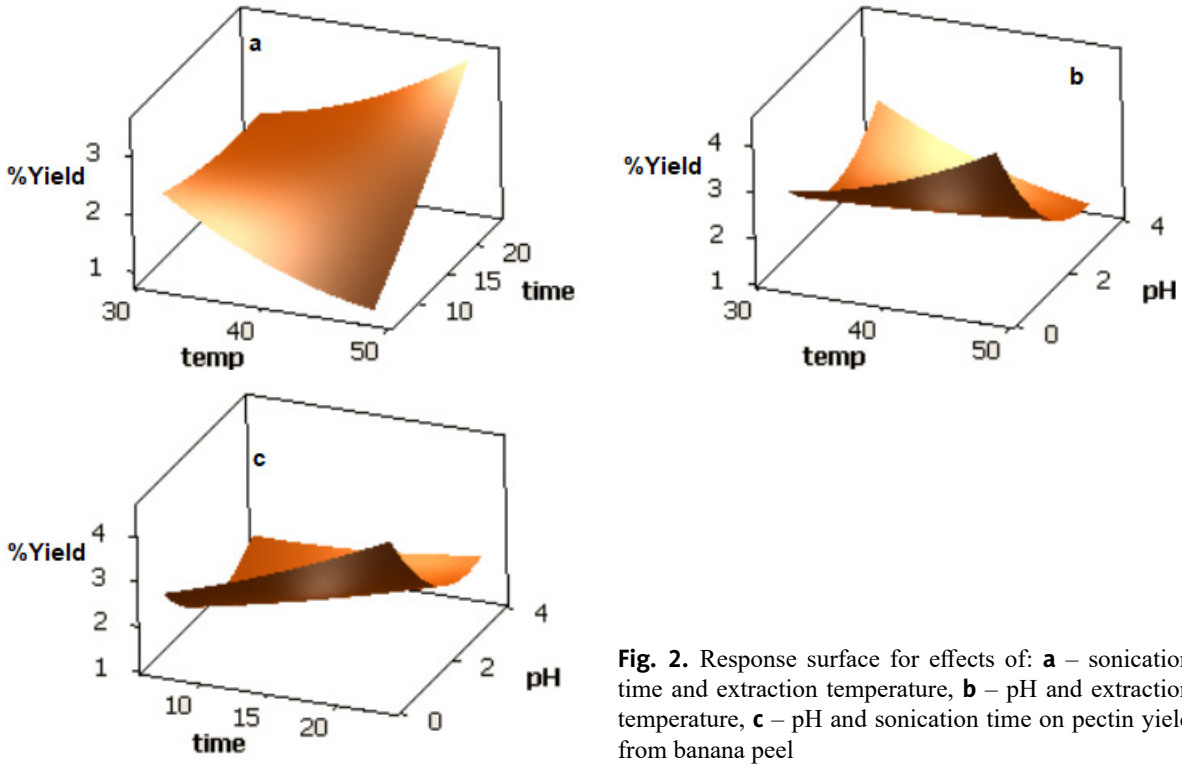

Fig. 2. Response surface for effects of: **a** – sonication time and extraction temperature, **b** – pH and extraction temperature, **c** – pH and sonication time on pectin yield from banana peel

in extraction temperature could enhance the rate of pectin extraction since the ultrasound waves generated the cavitation bubbles causing the cell wall disruption to improve the efficiency of pectin yield (Bhaskaracharya et al., 2009; Luque-Garcia and De Castro, 2003; Vinatoru, 2001). A longer sonication time from 10 min to 20 min exhibited a positive effect on the pectin yield (Fig. 2). This result was similar to a report by Phaiphan et al. (2020) who noted that higher sonication time (5–15 min) increased the pectin yield and was in accordance with the reports by Bagherian et al. (2011), Pagán et al. (2001) and Li et al. (2012). The reason was that the extraction of pectin yield required appropriate extraction time and temperature to expose the pectin out of the banana peels to the extraction solvent (Maran et al., 2014; Samavati, 2013; Qiu et al., 2010). However, long extraction time and high temperature might accelerate to degrade the pectin chain structure

(Zheng et al., 2011). The pH of a solution is one of the factor variables that affect the pectin extraction. The percentage of yield was highest when the pH values of the solution decreased to lower than 2.0 (Fig. 2b and 2c) and the combination effects between pH of a solution and extraction temperature encouraged positive effects on the pectin yield and could help the efficiency of the UAE. The results exhibited that a high concentration of acidity could improve the efficiency of pectin extraction. In this condition, it has a high ability to dissolve and hydrolyse insoluble pectin into soluble pectin, whereby the yield of pectin could significantly increase (Ma et al., 2013; Maran and Prakash, 2015).

The effect of extraction condition on DE of pectin

The DE values of pectin were obtained at a range of 66.1% to 86% (Table 2). The results exhibited that the pH had significantly ($p < 0.05$) linear effects on the DE and the interaction of pH affected the DE in quadratic manners (Table 3). The effect of the sonication time, pH and extraction temperature on the DE are presented in Figure 3a–3c. The DE of pectin increased with the increase in extraction temperature and sonication time, before it began to decrease. A higher amount of DE could be obtained at an extraction temperature of 30–40°C, pH between 2–4 and sonication time between 15–20 min as shown in Figure 3. The hydrochloric acid of 0.05 M was used as a solvent extraction that could extract highly methylated pectin from the cell wall of the banana peels. Nevertheless, the use of harsh extraction conditions, such as extremely low pH, very high extraction temperature and very long extraction time, caused the DE to decrease due to the increase in the de-esterified polygalacturonic acid backbone (Wai et al., 2010). Based on the results, a range of DE values (66.1–86%) was similar to other banana peel sources that were reported by Happi Emaga et al. (2008), Phaiphan (2019) and Phaiphan et al. (2020). All the pectin extracted from this study had DE values higher than 50%. Thus, the DE values of pectin can be suggested to be highly esterified and can

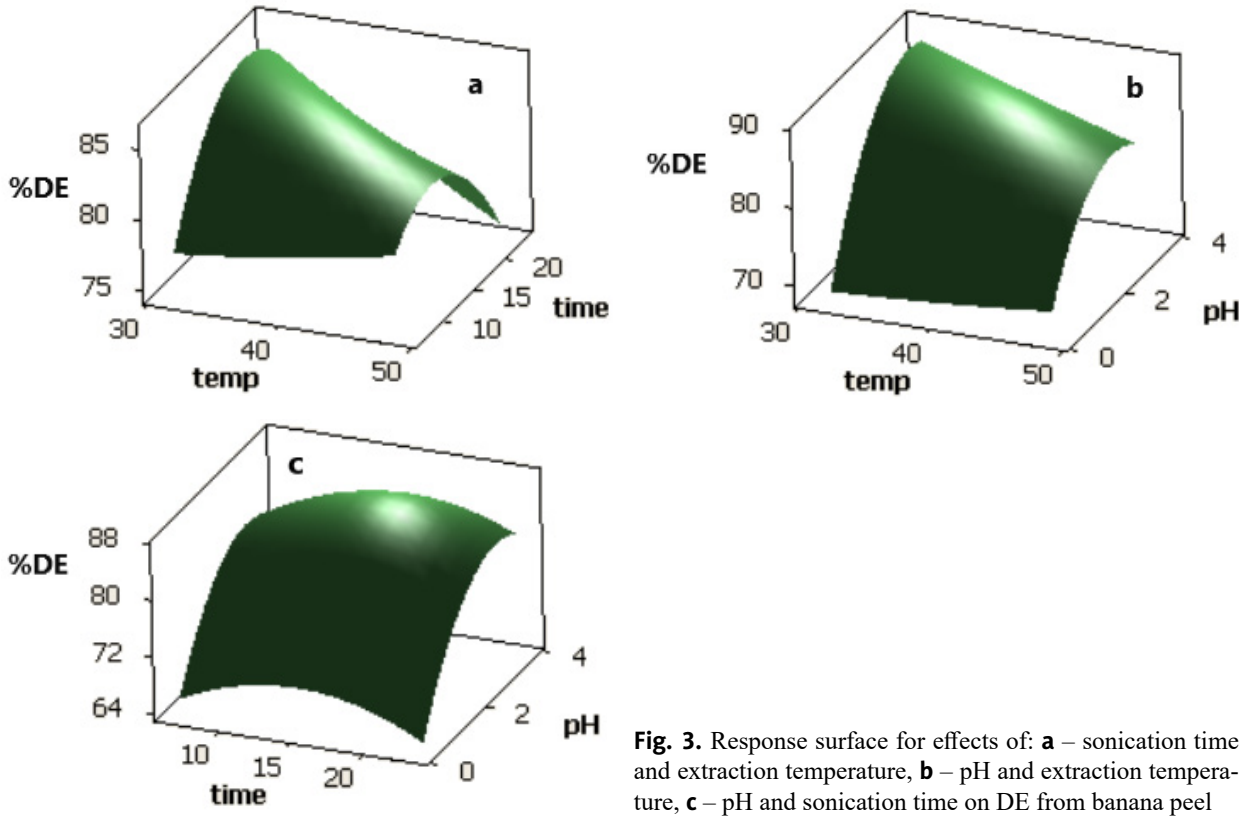

Fig. 3. Response surface for effects of: **a** – sonication time and extraction temperature, **b** – pH and extraction temperature, **c** – pH and sonication time on DE from banana peel

be classified as high-methoxyl pectin (HMP) which forms a gel in a high concentration of sugar (>65%) and low pH of a solution (Videcoq et al., 2011).

The effect of extraction condition on GalA of pectin

The GalA values of pectin ranged from 70.81% to 98.96% (Table 2). The results demonstrated that the extraction temperature and pH had significantly ($p < 0.05$) linear effects on the GalA. The interactions of extraction temperature and sonication time affected the GalA in quadratic manners (Table 3). The GalA of pectin increased with the increase in sonication time and extraction temperature before it began to decline. The GalA of pectin increased with the increase in sonication time of 10–20 min, extraction temperature of 33–46°C and pH lower than 3. The response surface plots are presented in Figure 4a–4c. All the GalA values had a range of 70.81% to 98.96%, which was higher than 65%, which is the Food and Agricultural Organisation's (FAO) approval for the minimum GalA value of pectin property (Bagherian et al., 2011). It could be considered that the highest GalA value was 98.96%, which was obtained at sonication time of 15 min, pH of 2.0 and extraction temperature of 40°C (Run 19). From the results, using a high extraction temperature would decrease the GalA value and it could possibly cause the pectin chain degradation or depolymerisation (Garna et al., 2004; Zhang et al., 2013). From this point of view, a range of GalA values (70.81–98.96%) was similar to other studies on banana peel sources conducted by Oliveira et al. (2016), Phaiphan (2019) and Phaiphan et al. (2020), whereby the GalA values were reported at 53.08–85.99%, 26–88% and 90.07%, respectively.

Optimisation and validation of optimised conditions

The individual numerical optimisation of extraction parameters is presented in Table 5. The highest extraction yield of pectin (6.31%) was obtained at the extraction temperature of 48.41°C, sonication time of

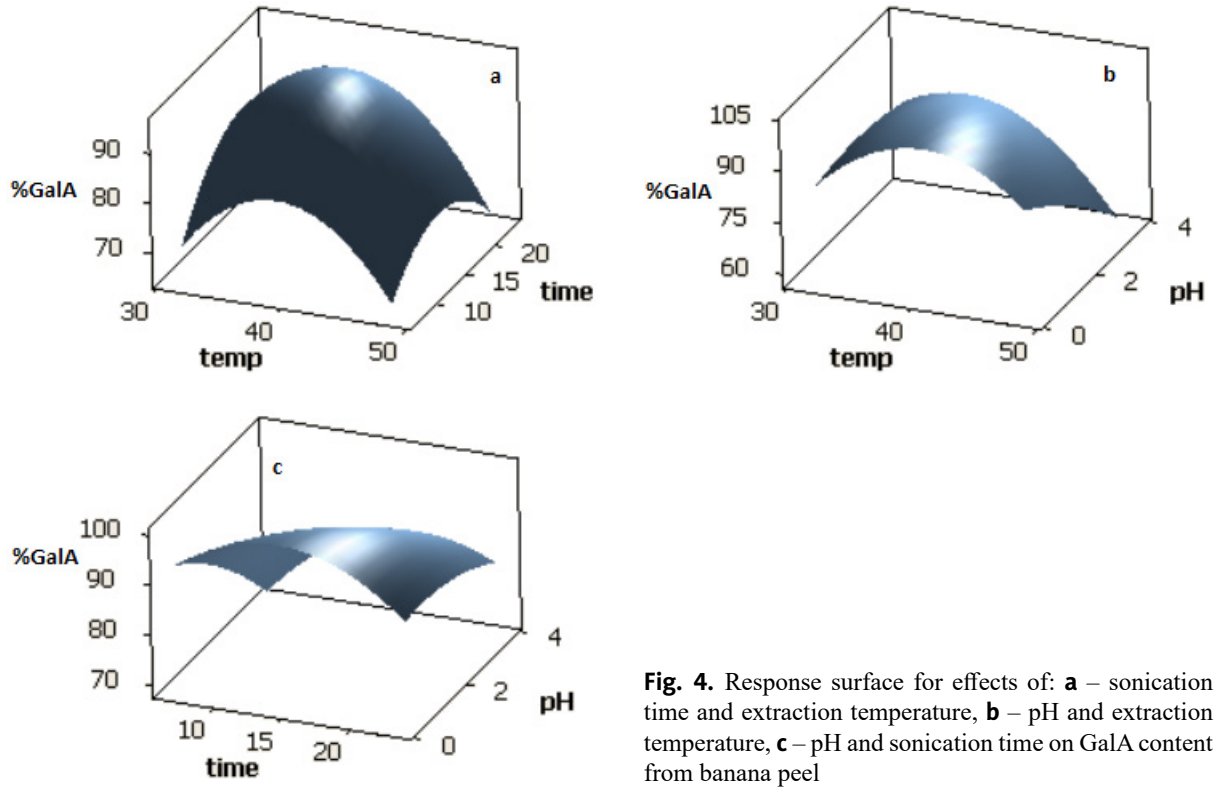

Fig. 4. Response surface for effects of: **a** – sonication time and extraction temperature, **b** – pH and extraction temperature, **c** – pH and sonication time on GalA content from banana peel

Table 5. Predicted and experimental response values at optimum conditions of each responses using the equation models under these conditions of UAE

Responses	Optimum UAE condition			Maximum value of response	
	temperature °C	time min	pH	predicted	experimental*
Extraction yield, %	48.41	23.41	0.32	6.31	6.48 ±0.10
Degree of esterification, %	31.59	18.82	3.24	90.01	89.53 ±1.27
Galacturonic acid content, %	40.42	14.07	0.32	99.89	97.42 ±1.03

*Mean ±standard deviation for three replication experiments.

23.41 min and pH of 0.32. The DE value could be archived at a maximum of 90.01% if the UAE were processed at an extraction temperature of 31.59°C, sonication time of 18.82 min and pH of 3.24. The optimal conditions of GalA values with sonication time of 14.07 min, pH of 0.32 and extraction temperature of 40.42°C could obtain the maximum GalA content of 99.89%.

The multiple response optimisation using UAE is presented in Table 6. All independent variable combinations could archive the maximum GalA content, DE and yield. The overall optimal conditions were achieved at an extraction temperature of 33.12°C, sonication time of 17.12 min and pH of 3.68. Under these conditions, the predicted value of pectin yield, DE and GalA content were 2.62%, 88.26% and 87%, respectively. The experiments were done in triplicate by using optimised conditions. The mean values of yield, DE and GalA content were 2.83 ±0.25%, 89.36 ±0.60%, 86.96 ±0.18%, respectively, and it was well correlated with the predicted value and revealed a satisfactory validation through real-time experiments, which was accomplished at optimised conditions.

Physico-chemical properties of pectin extracted by UAE compared to commercial pectin

The physico-chemical properties of pectin extracts are presented in Table 7. The pectin yield of optimal conditions was 2.83%, which was higher than the pectin yield of 2.32%, which was reported by Phaiphan et al. (2020), when extracting pectin from the banana peels using the UAE method (50°C, 10 min). The colour of commercial pectin was still brighter than the pectin extracted by UAE and both were significantly different ($p < 0.05$). The colour of commercial pectin was $L^* = 79.60$, $a^* = 3.57$, and $b^* = 15.78$ and the colour of the extracted pectin under UAE was $L^* = 73.41$, $a^* = 6.08$, and $b^* = 15.51$. ΔE value was 6.68. Values over 5 indicate that there is a noticeable variation to the human eye (Obón et al., 2009). The dried pectin extracted by UAE and dried commercial citrus pectin are presented in Figure 1.

Table 6. Multiple response optimisation of overall optimum conditions (33.12°C, 17.12 min, pH 3.68) using UAE

Responses	Predicted value	Experimental value*
Extraction yield, %	2.62	2.83 ±0.25
Degree of esterification, %	88.26	89.36 ±0.60
Galacturonic acid content, %	87	86.96 ±0.18

*Mean ±standard deviation for three replication experiments.

Table 7. Physico-chemical properties of pectin extracted by UAE compared to commercial pectin

Characteristics	Pectin extracted by UAE (33.12°C, 17.12 min, pH 3.68)	Commercial (citrus) pectin
Yield of pectin, %	2.83 ±0.16	–
*L**	73.41 ±0.01[b]	79.60 ±0.02[a]
*a**	6.08 ±0.02[a]	3.57 ±0.02[b]
*b**	15.51 ±0.03[b]	15.78 ±0.02[a]
Moisture content, %	4.71 ±0.06[b]	6.54 ±0.43[a]
Ash content, %	3.06 ±0.12[a]	1.80 ±0.04[b]
Degree of esterification, %	88.76 ±0.52[a]	81.25 ±1.16[b]
Methoxyl, %	14.48 ±0.40	13.26 ±0.25
Galacturonic content, %	87.73 ±0.28[b]	98.00 ±0.48[a]

Values are expressed as mean ±standard deviation for three replication experiments. Values with different superscript letters in the row are significantly different at $p < 0.05$ by Tukey's test.

The extracted pectin under UAE had a lower moisture content (4.71%) than commercial pectin (6.54%). Pectin with a low moisture content had long-term storage benefits and prevented bacterial growth (Muhmad-Zadeh et al., 2010). Pectin with a lower ash content indicates that it has better gelation (Norazelina et al., 2011), while the commercial pectin has lower ash content (1.80%) than the extracted pectin under UAE (3.06%).

The extracted pectin under UAE had higher DE (88.76%) and methoxyl (14.48%) than commercial pectin, which had DE of 81.25% and methoxyl of 13.26%. Both of them could be classified as a high-methoxyl pectin, which could be used as rapid-set pectin types. It forms a gel with a high sugar content (>65%) with a low pH solution (Shaha et al., 2013; Yapo et al., 2007). It is mostly used for jelly and jam products with high suspension of matter or other food products with high amounts of sugar (De Oliveira et al., 2016). Moreover, high-methoxyl pectin can easily form a high sugar gel (Rouse et al., 1962). The GalA content, which was obtained from the commercial pectin of 98%, was higher compared with the pectin under UAE (87.73%). The GalA content indicated physical properties and the purity of pectin, whereby a high GalA and low ash content of pectin could be classified as highly pure pectin (Hwang et al., 1992).

CONCLUSIONS

In this study, pectin was extracted from banana peels by using UAE under different process conditions such as extraction temperature, sonication time and pH. The obtained optimal conditions were an extraction temperature of 33.12°C, sonication time of 17.12 min, and pH of 3.68, which could achieve the maximum pectin yield of 2.62%, DE of 88.26% and GalA content of 87%. The experimental values were well correlated with the predicted values and revealed a satisfactory validation through real-time experiments which were accomplished at optimised conditions. The physico-chemical properties of extracted pectin were comparable to commercial citrus pectin. Therefore, the results obtained revealed that pectin was effectively extracted from the banana peel waste using UAE, which confirmed that it has potential as a source for the food, beverage and pharmaceutical industries. Further studies should be conducted to improve the quality of pectin and explore the bioactivity potential of pectin such as prebiotic, antioxidant properties, dynamic rheology, and sugar composition of pectin (galactose, rhamnose, xylose, arabinose, etc.) by chromatography.

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