Print ISSN 2682-3993
Online ISSN 2682-4000

Production and Characterization of Pectin by Acid Extraction Method from Orange Peels Waste Using Response Surface Methodology (RSM)

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***Abstract*:** Egypt produces orange fruits about 72.3% of the total production of citrus fruits with approximately 3.42 million tons. Orange peels are the major source of commercial pectin. Pectin is a polysaccharide compound present in the rind of citrus fruits. Pectin is used in different industries such as pharmaceuticals and food processing. This study aims to extract pectin from orange peels using the acid extraction method. The effect of pH (1-3), temperature (60-90℃) and time (30 to 120 min) was investigated on the yield using a Box-Behnken design and response surface methodology. The highest extraction yield of (21.9%) was obtained at a temperature of 90℃, time of 120 min and pH of 1. The degree of esterification for extracted pectin was found 68.28% indicating that pectin is high methoxyl pectin. Extracted pectin has an equivalent weight, a methoxyl, and an anhydrouronic acid content of 704.46 g/mole, 10.7%, and 89.58% respectively. It was found that moisture and ash content was 12%, and 2.2% respectively. The results showed that the characteristics of extracted pectin are within the limits permitted by the International Pectin Producers Association (IPPA).

Keywords:- Pectin, Orange Peels, Acid Extraction, Response Surface Methodology, Physico-chemical Properties

I. INTRODUCTION

Egypt's annual production of citrus fruits reached 4.45 million tonnes in 2020. About 76.4% of citrus fruits represent production oranges reached 3.42 million tonnes [1], which covers domestic consumption and exports the surplus production. Egypt ranked first in the world among orange exporting countries globally, its exports reached about 1.5 million tons, representing 38% of the world's orange exports [2,3]. Due to the mass production of oranges, a very large amount of orange peel waste is produced annually, with unsafe disposal of this waste will lead to long-term environmental risks. While this waste can be used in the manufacture of uncountable useful products in many fields such as orange pectin [4]. Pectin is produced commercially in the form of white to light brown powder mainly extracted from citrus fruits. Pectin can be extracted from orange peels using several methods such as conventional methods (acid extraction) and non-conventional methods (Ultrasound, Electromagnetic induction, Microwave extraction and Enzymatic extraction) [5]. Pectin is a complex mixture of polysaccharides composed of a linear backbone of α (1-4)-D-galacturonic acid residues. Molecules of pectin include the linear parts of 1-4 linked α-D galacturonic acid units with some of the carboxyl groups and hydroxyl groups which are esterified by methanol. The amount of pectin contained in orange peels is estimated to be 30% [5].

Pectin is widely used in the food industry as a gelling, stabilizing, and thickening agent. Furthermore, pectin is employed in diverse pharmaceutical activities such as wound healing, and lipase inhibition [6]. Several factors affect the process which include the pH, temperature, solvent used for extraction, time of extraction, agitation rate, and liquid-solid ratio (LSR) amongst others. Many studies have been done to discuss the effect of different parameters on the extraction of pectin from orange peels and identify the factors that most influence the results [7-9]. According to Tiwari et al., the main affected parameters in the extraction of pectin are temperature, pH, and time. They investigated the possibility of pectin extraction from orange peels at different parameters that affected the yield such as pH and different particle sizes of the peel sample. They found that the best yield was extracted at pH 1 and 60-mech size of the sample. Further, they noticed that by decreasing the sample particle size the yield is increased. This happens as the mass transfer for the sample surface area increases with decreasing the particle size thus increasing the yield [10]. Nitin and Shah et.al discussed the extraction process of pectin using both fresh orange peels and dried cake produced from steam distillation. They found at higher extraction temperature, that the yield produced from the dried cake is higher than the

Print ISSN 2682-3993
Online ISSN 2682-4000

fresh peels which are not economic due to the high heating requirements for the extraction process [11]. Bhavya et.al. discussed the extraction of pectin from orange peel using ethanol, and hydrochloric acid, which is affected by pH, temperature, and time. They found the optimum parameter that gets an optimum yield of 7.9% at pH 1, temperature 70°C and the time of extracting was 30 minutes [12]. Khan et.al discussed the extraction process of pectin from sweet orange peels at different parameters. They found that the affected parameters were (temperature, pH, and time), and their values on the extraction of pectin were at a temperature of 85°C, pH ranging from (1- 3), and 1 hr respectively. They also noted that increasing pH of more than 2 causes decreasing the extracted pectin at 85°C [13]. Hosseini et.al. studied the effect of LSR, time, the temperature on the extracted pectin yield from sour orange peels. They found that maximum yield (18.35%) can be obtained at 95°C, 90 min and LSR of 25% [14]. On the other hand, Banu et.al. discussed the ability to extract pectin from orange peels using different acids to get a maximum yield of pectin and they found the maximum yield of 30% can be obtained using nitric acid as a solvent [15]. Finally, Mota et al. studied the extraction of pectin using conventional and microwave heating methods from opuntia robusta. The pectin was analysed by calculating the degree of esterification, methoxyl content, yield, molar mass and galacturonic acid content. The results indicated that pectin extracted using the conventional method has a degree of esterification lower than 50, with a yield percentage of 15.71 and a molecular weight of 20339 $g.mol^{-1}$. While the pectin extracted from the microwave method had a degree of esterification higher than 50%, with a yield percentage of 14.64 and molecular weight of 33110 $g.mol^{-1}$ [16].
The degree of esterification (DE) is an effective property in gel production by pectin. According to DE, pectin can be classified into two categories: High methoxyl pectin (HMP) with DE above 50% and low methoxyl pectin (LMP) with DE less than 50% [17]
This study aims to extract pectin from orange peels using the acid extraction method with the help of response surface methodology to optimize the main pectin extraction parameters (time, temperature, pH) to obtain the optimum pectin yield and investigate the characteristics of pectin produced.

II. Materials and Methods

A. Materials

The main raw material used in the production of pectin is orange peel, which was collected from the local juice shop. All chemicals and solvents were purchased from (Al Gomhouria Company for Chemicals and Pharmaceuticals, Egypt).

B. Sample Preparation

The peels were washed and cut into small pieces. After that, the peels were left to dry under the sunlight for complete drying. The dried peels were ground to pass through 40 mesh sieves to obtain powdered peels. Then stored in a dry place for the next steps.

C. Experimental Design

The main factors that affected the pectin extraction process were extraction temperature, extraction time and extraction pH which showed a massive effect on pectin yield [11,12]. Box-Behnken design with the 3 optimal processing independent variables was used to calculate ideal conditions for the extracted pectin from orange peels. These variables were temperature ranging from 60 to 90°C, pH values from 1 to 3 and time from 30 to 120 minutes (see Table 1). These factor levels were coded as –1 (low), 0 (medium), and 1 (high), respectively as shown in Table 1. The experimental design comprised a total of 17 experiments with 5 repeating experiments at centre points and 12 factorial points [18].
The total number of experiments was obtained from equation (1).

$$No.of\ Experiment = 2k * (k-1) + Co \quad (1)$$

Where the number of variables is k (pH, time, and temperature). There are 12 experiments with 5 replications (Co) that have been enhanced to evaluate that pure error.

Table 1: Codded and Actual Values of Pectin Extraction

Variable	Symbol	Coded and actual levels		
		-1	0	+1
pH	X_1	1	2	3
Temperature	X_2	60	75	90
Time	X_3	30	75	120

Print ISSN 2682-3993
Online ISSN 2682-4000

D. Extraction of Pectin

Acidic extraction of pectin was carried out according to the method described by Pasandide et.al with some modifications [19]. 5 grams of orange peels powder were added to 150 ml of distilled water with a liquid-solid ratio (LSR) of 30 (v/w), the pH of the mixture was adjusted by using drops of hydrochloric acid to get the desired pH (1,2 and 3), the mixture was heated using water bath at different temperatures (60, 75 and 90℃) with a mechanical stirrer at 50 rpm at a different time of extraction (30, 75 and 120 min). The pH was adjusted every 15 min and the lost water was replaced. Then the mixture was cooled rapidly to less than 40℃ and filtered using filter paper. An equal volume of ethanol 95% was added to the filtrate solution and left for 30 minutes at 4°C to allow the floatation of pectin on the surface. Gelatinous pectin was filtered using cheesecloth and then washed using 70% and 95% ethanol respectively to remove disaccharides and impurities. The formed gel was then dried at (40- 45°C) until a constant weight was achieved. "Fig.1" shows the experimental procedure of pectin extraction. The yield (%) can be calculated using equation (2) [19].

$$Yield\ \% = \frac{W_d}{W_p} * 100 \quad (2)$$

Where Wd= weight of dried pectin obtained (g), and Wp= initial weight of orange peel powder used for extraction (g).

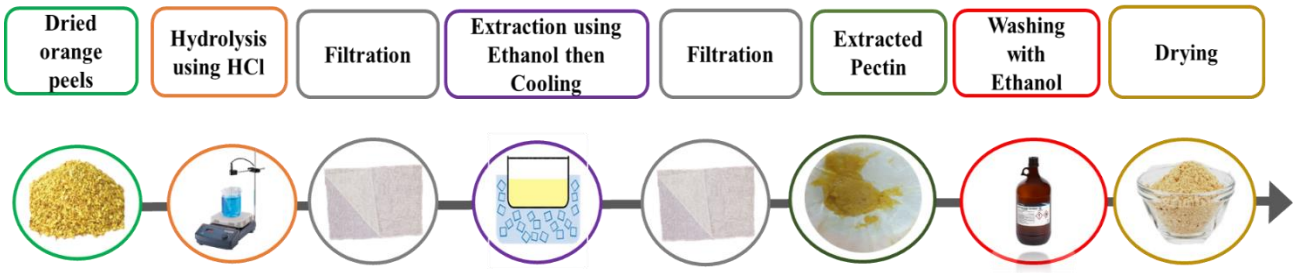

Figure 1: Experimental Procedure

E. Characterization of Extracted Pectin from Orange Peels

The dried pectin extracted from orange peels at the optimum conditions was characterized by determining the Physico-chemical properties through the following quantitative tests.

1. Fourier Transform Infrared (FTIR) Spectroscopy Analysis

The extracted pectin was dried and stored for FTIR analysis. FTIR was recorded for polysaccharide pectin using Class 1 Laser Product IEC/EN 60825-1/A2:2001 Avatar Series (USA) in The Egyptian Academy for Engineering and Advanced Technology [20].

2. X-Ray Diffraction (XRD) Analysis

X-Ray Diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The mineralogical composition is assessed using X-ray diffraction Brukur D8 advanced computerized X-ray Diffractometer apparatus with monochromatic Cu Kα radiation which operates at 40KV and 40mA [21].

3. Determination of Equivalent Weight

Equivalent weight was used for calculating the Anhydrouronic Acid Content (AUA) and the degree of esterification. It is determined by titration with sodium hydroxide (NaOH) using phenolphthalein. 0.5 g of the pectin was mixed with 5 ml of ethanol with 1 gm NaCl with 100 ml of distillate water with 6 drops of phenolphthalein with rapid stirring till

Print ISSN 2682-3993
Online ISSN 2682-4000

all pectin is dissolved. Titration takes place slowly with 0.1N of NaOH till the colour change to pink. The equivalent weight can be calculated from equation (3) [22].

$$\text{Equivalent Weight} = \frac{1000 * \text{weight of the sample (g)}}{\text{volume of NaOH (mL)} * \text{Normality of NaOH}} \quad (3)$$

4. Determination of Methoxyl Content (MeO)

Methoxyl content of pectin is important to control the gel strength, the setting time and the ability of the pectin to form gels. Methoxyl content was determined by adding 25 ml of 0.25N NaOH to the neutralized solution obtained during the equivalent weight determination and then placing it at room temperature for 30 min. Then add 25ml of 0.25N HCl to the solution. Finally, it is titrated as previous using 0.1N NaOH until the colour changes to pink. The methoxyl content was determined from equation (4) [22].

$$\text{MEO\%} = \frac{\text{volume of NaOH (ml)} * \text{Normality of NaOH} * 31}{\text{Weight of the sample (mg)}} \quad (4)$$

5. Determination of Anhydrouronic Acid (AUA)

Anhydrouronic acid is used to determine the purity, degree of esterification, and to evaluate the physical properties of pectin. By using the values of the equivalent weight and the methoxyl content. Anhydrouronic acid content was calculated from equation (5) [9].

$$\text{(AUA)\%} = \frac{176 * 100 * \text{Volume of NaOH(ml) from (2)} * \text{Normality of NaOH}}{\text{Weight of the sample(g)} * 1000} + \frac{176 * 100 * \text{Volume of NaOH(ml) from "(3)"} * \text{Normality of NaOH}}{\text{Weight of the sample(g)} * 1000} \quad (5)$$

6. Determination of Degree of Esterification (DE)

The degree of methyl esterification of pectin can be calculated using values of methoxyl content (MeO) and total anhydrouronic acid content (AUA). The degree of esterification was calculated from equation (6) [22].

$$\text{DE}(\%) = \frac{176 * \text{MEO\%}}{31 * \text{AUA\%}} * 100 \quad (6)$$

Where 176 and 31 are the formula weights of AUA and MeO respectively.

7. Determination of Moisture Content

The moisture content of the pectin sample was determined by weighing 1g of the sample into a crucible. The crucible and sample were heated at 130°C for 2 hours. The moisture content was calculated from equation (7) [23].

$$\text{Moisture Content\%} = \frac{\text{Initial weight (g)} - \text{Final weight (g)}}{\text{Initial weight (g)}} \text{ x } 100 \quad (7)$$

8. Determination of Ash Content

The ash content of the extracted pectin sample was determined by weighing 1g of pectin in a crucible and then heated in a muffle furnace at (550 - 600°C) for 4 hours, then cooled to room temperature. The produced ash was weighed and the percentage was calculated depending on the sample weight taken. The percentage of ash content of the sample was calculated from equation (8) [23].

$$\text{Ash Content \%} = \frac{\text{Weight of the ash (g)}}{\text{Weight of pectin (g)}} \text{ x } 100 \quad (8)$$

III. Results and Discussion

A. Model Fitting and Statistical Analysis

In this study, three factors and three levels Box–Behnken response surface design (BBD) were applied to evaluate and optimize the effect of process variables such as time (30–120 min), temperature (60–90°C) and pH (1–3) on the yield of pectin extracted from orange peels. The experimental design of RSM in coded and actual values of variables and the obtained yield% are shown in Table (2).

In the selection of the appropriate model that describes the relationship between the independent variables (time, temperature and pH) and the response (yield), several considerations were made to select the highest order polynomial

Print ISSN 2682-3993
Online ISSN 2682-4000

where the additional terms are significant and there is no error in the model. It was found that the model was a second-order [8].

The second-order model equation is shown below in equation (9)

$$Y = \beta 0 + \sum \beta_j X_i + \sum \beta_{jj} X_{ii} + \sum \beta_{ij} X_{ij} + \varepsilon \quad (9)$$

Where Y represents the predicted response, β0 is the model intercept, and βj, βjj, and βij are the regression coefficients for the linear, quadratic, and interactive effects of the model, respectively. Xi and Xj are the factors and ε is the error of the model.

The result from the analysis of variance (ANOVA) for the quadratic regression model used for pectin production shows that the lack of fit for this prediction model was not significant. This indicates that the prediction models are suitable for predicting the optimum conditions to extract pectin from orange peels. In addition, the R-squared value for the model used to predict the yield of pectin that found to be 0.9936 which indicates that this model can explain satisfactorily the relationship between the independent variables (Time, Temperature, and pH) and the response (pectin yield). P-value less than 0.05 indicates that the model terms are significant in this case according to Table (3). (A, B, C, AB, BC, A^2, B^2, C^2) are significant model terms, and values greater than 0.05 indicate the model terms are not significant. In this case (AC) is an insignificant model term

The initial second-order model equation obtained for pectin yield from orange peels after eliminating the insignificant terms with $p < 0.05$ is shown below in equation (10).

$$\text{Yield} = 17.42 + 3.2073\text{A} - 3.16\text{B} + 2.8675\ \text{C} + 0.3950\ \text{AB} + 1.325\ \text{BC} + 1.37A^2 - 4.395\ B^2 - 2.95\ C^2 \quad (10)$$

Table 2: Coded Levels and Actual Values of the Variables in Box–Behnken Design and Pectin Yield (%)

Run	Variables						Yield %
	pH		Temperature		Time		
1	0	(2)	0	(75)	0	(75)	17
2	0	(2)	0	(75)	0	(75)	17.2
3	-1	*(1)*	0	(75)	+1	(120)	18
4	1	*(3)*	1	(90)	0	(75)	14.8
5	0	*(2)*	1	(90)	-1	(30)	16
6	1	*(3)*	0	(75)	+1	(120)	8.4
7	1	*(3)*	-1	(60)	0	(75)	9
8	0	*(2)*	1	(90)	+1	(120)	22.5
9	0	*(2)*	0	(75)	0	(75)	17.2
10	0	*(2)*	0	(75)	0	(75)	17.5
11	0	*(2)*	0	(75)	0	(75)	17.9
12	0	*(2)*	-1	(-1)	+1	(120)	14.1
13	0	*(2)*	-1	(-1)	-1	(30)	10.76
14	-1	(1)	0	(75)	-1	(30)	8.4
15	-1	(1)	-1	(60)	0	(75)	14.78
16	+1	(3)	0	(75)	-1	(30)	3.24
17	-1	(1)	+1	(90)	0	(75)	20

B. Effects of Processing Factors on Pectin Yield

The yield of pectin ranged from 3.24 to 22.5% as shown in (Table 2). These differences are due to different conditions of the extraction process. Therefore, the effect of parameters on each other should be studied to optimize pectin yield.

1. Effect of Temperature on Pectin Yield

It was observed that increasing extraction temperature leads to an increase in pectin yield, with the maximum obtained yield at 90℃ as shown in "Fig. 2" which agrees with Pagan et al. and Gama et al. As temperature increases, the solubility of pectin is increased causing an increase in the rate of extraction. Nevertheless, beyond the optimum value

Print ISSN 2682-3993
Online ISSN 2682-4000

of the temperature (90℃), the yield of pectin is reduced as a degradative action which results in pectin of minor molecular size not precipitable with alcohol [24,25].

Table 3:Results of Analysis of Variance (ANOVA) for Regression Model of Pectin Yield

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	366.0	9	40.68	121.5	< 0.0001	Significant
A-Temp	82.30	1	82.30	245.9	< 0.0001	
B-pH	79.88	1	79.88	238.7	< 0.0001	
C-Time	65.78	1	65.78	196.5	< 0.0001	
AB	0.624	1	0.624	1.87	0.0293	
AC	2.50	1	2.50	7.46	0.2143	
BC	7.02	1	7.02	20.99	0.0025	
A^2	7.90	1	7.90	23.62	0.0018	
B^2	81.33	1	81.33	243.0	< 0.0001	
C^2	36.64	1	36.64	109.5	< 0.0001	
lack of Fit	1.87	3	0.624	5.34	0.0697	Not significant

2. Effect of Time on Pectin Yield

It was observed that the longer the extraction duration, the higher the pectin yield, with the maximum obtained yield at 120 min as shown in “Fig.3”. As the duration increases, the concentration of the pectin in the solution increases, leading to an increased yield. However, beyond the optimum time at higher temperatures, thermal degradation occurs which leads to a decrease in pectin yield. This complies with El-Nawawi et al., Kliemann et al., Tang et al., and Gama et al. while working on pectin extraction from orange peels [25-28].

3. Effect of pH on Pectin Yield

It was observed that increasing extraction pH leads to an increase in the pectin yield, with the maximum obtained yield at 1 pH as shown in “Fig. 4”. As pH decreases, the solubility of pectin is increased causing an increase in the rate of extraction. This is because a high pH level leads to less degradation of the neutral sugar side chains, and at relatively low temperatures, the solubility of the extracted pectin decreases, hence decreasing extraction rate with consequent low pectin yield, while increasing extraction time and temperature at low pH leads to a corresponding increase in pectin yield [5].

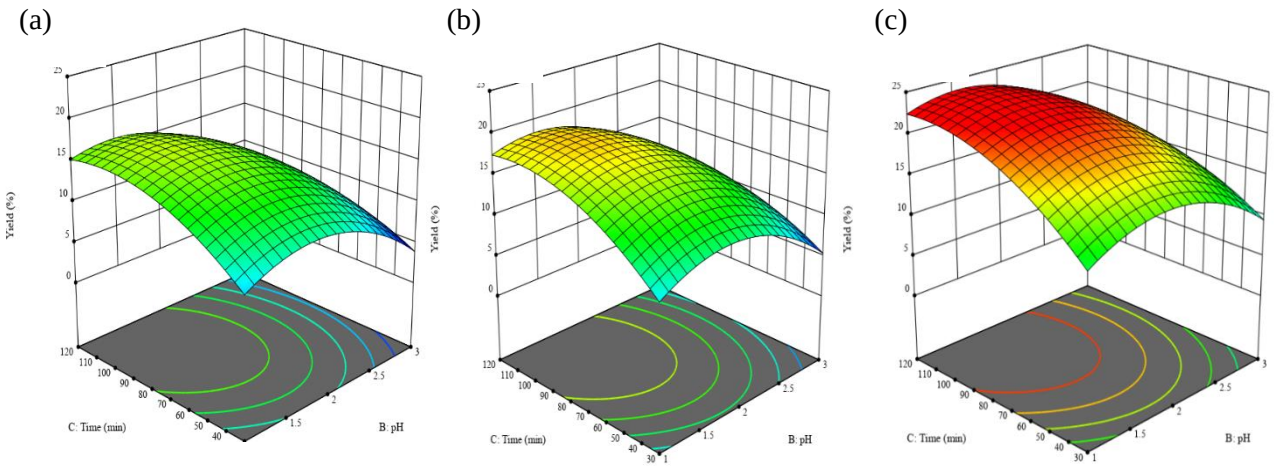

Figure 2: Response Surface Plot of the Effect of Extraction Time and Extraction pH on Dried Pectin Yield at Different Extraction Temperature (a: minimum, b: centerpoint, c: maximum)

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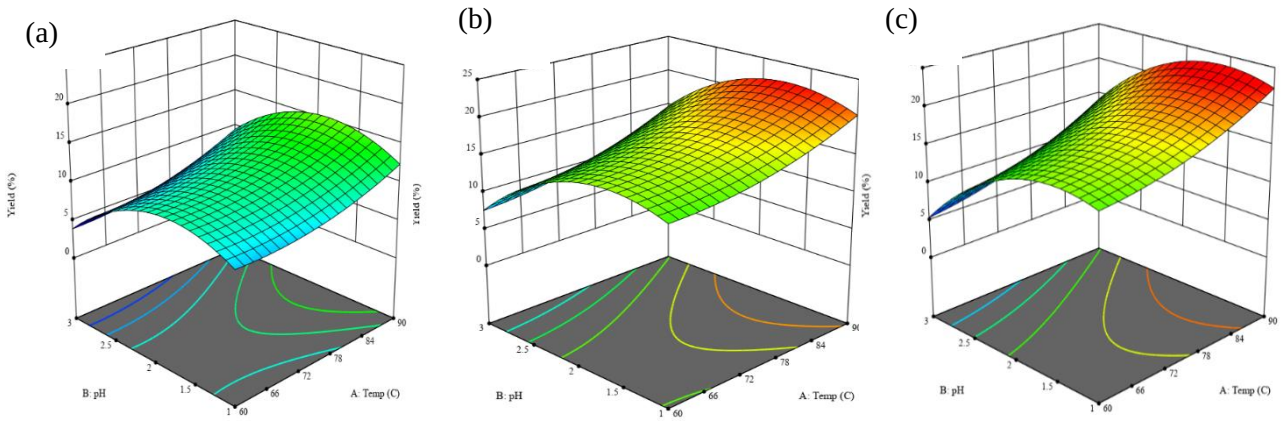

Figure 3: Response Surface Plot of the Effect of Extraction pH and Extraction Temperature on Dried Pectin Yield at Different Extraction Time (a: minimum, b: centerpoint, c: maximum)

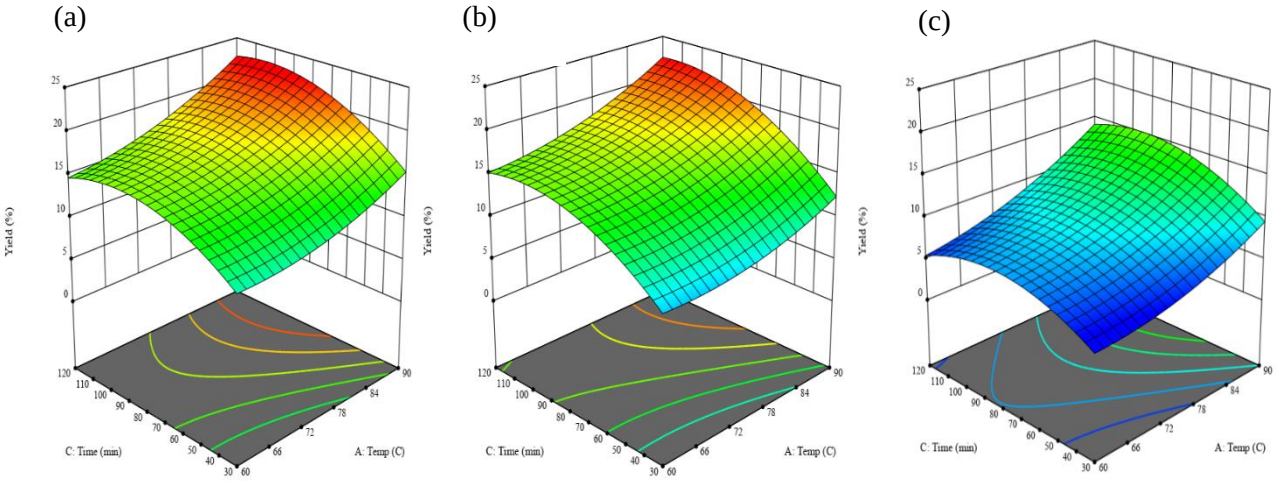

Figure 4: Response Surface Plot of the Effect of Extraction Time and Extraction Temperature on Dried Pectin Yield at Different Extraction pH (a: minimum, b: centerpoint, c: maximum)

C. Optimization of Process Variables

Determination of the optimum process conditions for maximizing pectin yield is very crucial in this present study. The Box-Behnken design cube model indicates that the highest pectin yield was observed to be 22.4% at a temperature of 90°C, 120 min, and pH 1 as shown in the upper left rear edge of the cubic model in "Fig.5" and this value in good agreement with the experimental value of 21.9 (%) performed at the same optimum values of the process variables. To assess the validity of these findings, 5 runs were performed at the optimum conditions. The mean percentage of pectin

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Online ISSN 2682-4000

yield was 21.52(%) with a standard deviation of 0.21679 which proves the validity of the suggested model as the error percent did not exceed 3.9%.

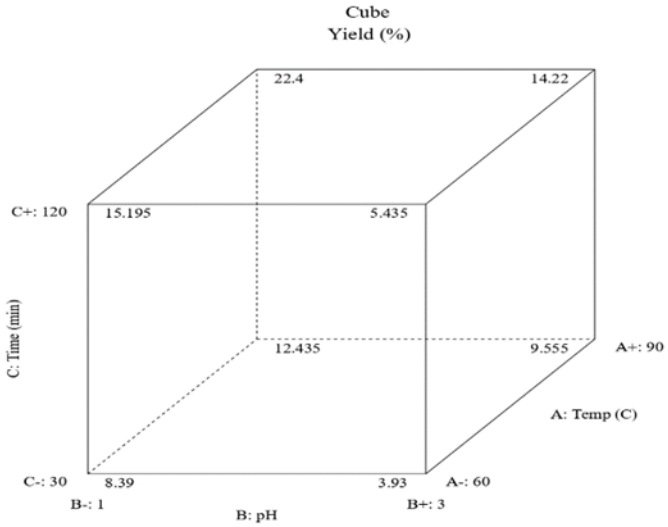

Figure 5: 3D Cube Representation of Pectin Yield (%) at Different Conditions

D. Physicochemical Characterization of Pectin

Characterizations of extracted pectin were carried out for various parameters to evaluate its suitability in food systems. The characteristic of extracted pectin, International Pectin Producers Association (IPPA), and literature values are presented in (Table 4).

1. Fourier Transform Infrared Spectroscopy Analysis of Pectin

In FTIR analysis each peak of wave number on the graph indicates different function groups of a specific polymer due to spectra absorbance. As shown in "Fig.6", the wavelength 3442 cm^{-1} indicates the presence of hydroxyl group (OH). The aliphatic (C-H) and carbonyl (C=O) groups were 2925.36 cm^{-1} and 1749.36 cm^{-1} respectively. The peak at 1446.9 cm^{-1} shows the presence of the methyl group while 1105.48 cm^{-1} indicates the presence of (C-O) in ester, alcohols and carboxylic acid [20].

2. X-Ray Diffraction (XRD) Analysis of Pectin

Pectin shows amorphous behaviour in most of the literature review [29]. XRD analysis results show that the extracted pectin has an amorphous nature as shown in "Fig.7".

3. Equivalent Weight

The equivalent weight of extracted pectin from orange peels was found to be 704.46 g/mol. The previous studies on pectin extraction have stated that their outcomes were ranging from 476-1209 g/mol, which that indicates the higher the equivalent weight the better the gel-forming effect. The equivalent weight of the pectin is the total content of free galacturonic acid in the molecular chains of pectin. Pectin produced at a low pH has a higher equivalent weight because a low pH can cause pectin polymerization into the longer chain, and in turn, reduces the free acid content [30].

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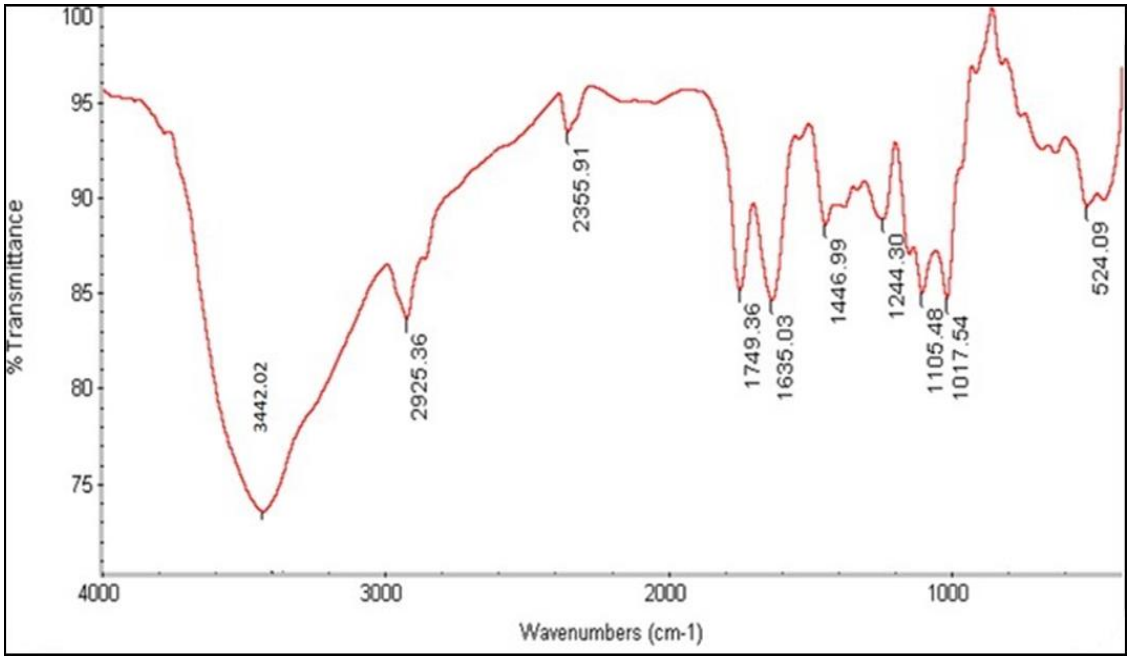

Figure 6: FTIR Analysis of Extracted Pectin

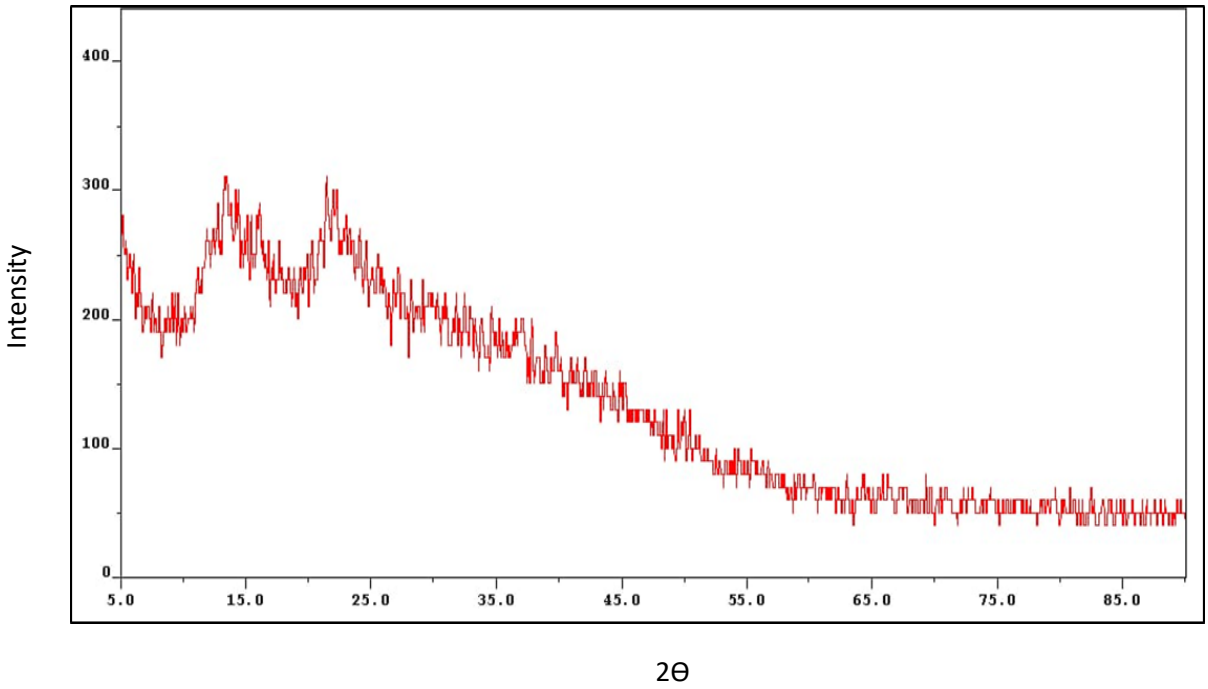

Figure 7: X-ray diffraction (XRD) Analysis of Extracted Pectin

4. Methoxyl Content

Pectin would be classified as high methoxyl pectin if its value was higher or equal to 7%. If methoxyl content was less than 7%, then it would be classified as low methoxyl pectin. The methoxyl content of extracted pectin was found to be 10.7 %. So, the produced pectin obtained from this study was classified as high methoxyl pectin [23].

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5. Anhydrouronic Acid

The anhydrouronic acid of extracted pectin from orange peels was found to be 89.58 %. To keep the purity of extracted pectin, the value of AUA should be more than 65% according to (Food Chemical Codex IV, monograph,1996). The reason for this high value of AUA is due to the double washing technique to avoid any presence of impurities [9].

6. Degree of Esterification

The degree of esterification of extracted pectin using hydrochloric acid was found to be 68.28 %. The pectin can be categorized as high methoxyl pectin because DE is higher than 50%. The degree of esterification decreased with the increase of maturity [30,31].

7. Moisture and Ash Content

The moisture content of the produced pectin using HCl as a solvent was found to be 12% which is the same as the standard specifications of pectin [32]. On the other hand, the ash content of pectin was found to be 2.2% which is less than the values obtained from the literature studies of 5.7%. The reason for this behaviour is due to the double washing technique with ethanol which removes the residual HCl and other impurities such as fibres and sugar [23].

Table 4: Characteristics Values of Extracted Pectin, IPPA Standard and Literature Values

Characteristics	Extracted Pectin	IPPA Standard [32]	Literature Values [32]
Equivalent Weight (g/mol)	704.46	600–800	326.7–1428.57
Methoxyl Content (%)	10.7	2.2–7.8	8.89
Anhydrouronic Acid Content (%)	89.58	min. 35	63.71–77.41
Degree of Esterification (%)	68.28	69.48	61.19–70.79
Ash Content (%)	2.2	max. 10	5.7
Moisture Content (%)	12	max. 12	8.33

IV. Conclusion

A detailed study on the optimization of pectin extraction from orange peels has been successfully carried out. The optimization of the effect of process parameters on pectin yield was studied using Response Surface Methodology. It was found that the optimum conditions for pectin extraction from orange peels were at a temperature of 90ºC, pH 1 and a time of 120 min. The results of the physio-chemical characterization of the extracted were measured and compared with the commercial pectin. The pectin has an equivalent weight of 704.46 g/mol, the methoxyl content of 10.7%, the anhydrouronic acid of 89.58%, degree of esterification of 68.28%, moisture and ash content of 12%, and 2.2% respectively. It was found that these results are within the permissible limits according to the International Pectin Producers Association (IPPA).

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