

# Optimization of Pectin Extraction from Orange Peels Using Response Surface Methodology (RSM)

A. Rady, A. Moawad, H. Hassan, M. Ragaa, M. AbdEl-Kader, M. Farid, M. Hamed and N. Essam

*Egyptian Academy for Engineering and Advanced Technology, Egypt*

*Supervisor: \*Eman M. Abdel Hamid, Assistant Professor*

*Egyptian Academy for Engineering and Advanced Technology, Egypt*

*\*Corresponding author: [eman.mohamed@eaeat.edu.eg](mailto:eman.mohamed@eaeat.edu.eg)*

**Abstract**—Egypt produces about 4.7 million tons of citrus fruits annually. Orange fruit was representing about 72.3% of the total production of citrus fruits in Egypt with approximately 3.42 million tons. Orange peels are one of the major commercial sources of pectin. Pectin is a polysaccharide compound found in the primary cell wall and the middle lamella of orange peels and is used in food processing, biomedical, and pharmaceutical industries. In this study, the acid extraction method was used, as the most suitable method for the extraction of pectin. The effect of temperature (60-90°C), pH (1-3), and time (30 to 120 min), were investigated on yield using a Box-Behnken design. The optimal conditions for the highest production yield of pectin (21.9%) using hydrochloric acid were pH 1, the temperature of 90°C with an extraction time of 120 min. Under these conditions, the produced yield was close to the predicted value of 22.4%. The extracted pectin contained an equivalent weight, a methoxyl, and an anhydrouronic acid content of 704.46 g/mole, 10.7%, and 89.58% respectively. The degree of esterification showed that the extracted pectin was high methoxyl pectin. Ash and moisture content was 12%, and 2.2% respectively. These results are within the limits permitted by the Food and Agriculture Organization (FAO) for commercial pectin.

**Keywords**—Pectin, Orange Peels, Acid Extraction, Response Surface Methodology, Physico-chemical Properties.

## I. INTRODUCTION

Egypt's annual production of oranges in 2020 reached 3.42 million tons, 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 [1][2]. Due to the mass-production of oranges, a very large amount of orange peels waste 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 [3]. 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 as conventional methods such as acid extraction and non-conventional methods such as Ultrasound, electromagnetic induction, microwave extraction and enzymatic extraction [4]. Pectin is a complex mixture of polysaccharides composed of a linear backbone of  $\alpha$  (1-4)-D-galacturonic acid residues. Molecules of pectin include the linear parts of 1-4 linked  $\alpha$ -D galacturonic acid units with some of the carboxyl groups and hydroxyl groups which

esterified by methanol. The amount of pectin contained in orange peels is estimated to be 30% [4].

Pectin is widely used in the food industry as a gelling, stabilizing, thickening agent. Furthermore, pectin is employed in diverse pharmaceutical activities such as wound healing, lipase inhibition [5]. Several factors affect the process which includes 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 [6][7]. According to Olugbenga & Kingsle (2018), the main affected parameters on the extraction of pectin are temperature, pH, and time [8]. Alok & Samarendra et.al. (2017) investigate the possibility of pectin extraction from orange peels at different parameters that affected the yield such as pH and different particle sizes of the peels 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 [9]. Nitin and Shah et.al (2017) 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 fresh peels which are not economic due to the high heating requirements for the extraction process [10]. Dvanooru & Shrilakshmi et.al. (2015) discussed the extraction of pectin from orange peel by 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 [11]. Mazzullah & Nizakat et.al (2015) 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 causing decreasing the extracted pectin at 85°C [12]. While Sayed & Faramarz et.al. (2015) 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% [13]. On the other hand, M. S. Banu et.al. (2012) 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 [14]. Also, Sohair & Fadi (1978) studied the different parameters that affected the pectin yield extracted from orange peels (the particle size and agitation), and they found that increasing the agitation speed has no significant effect on both the quality and the yield of pectin. They also found decreasing particle size, increasing the pectin yield [15].

The degree of esterification (DE) is an effective property in gel production by pectin. According to DE, pectin can be classified into two categories: The high methoxyl pectin (HMP) with DE above 50% and the low methoxyl pectin (LMP) with the DE less than 50% [16].

The aim of this study 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. MATERIAL 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)

### B) Methods

#### 1. Sample Preparation

The peels were washed and cut into small pieces. After that, the peels 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.

#### 2. Experimental design

The main factors that affected the pectin extracted process were extraction temperature, extraction time and extraction pH which shown a massive effect on pectin yield [10][11]. 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 I). These factor levels being coded as -1 (low), 0 (medium), and 1 (high), respectively (see table I). The experimental design comprised a total of 17 experiments with 5 repeating experiments at centre points and 12 factorial points [19].

The total number of experiments obtained from (1).

$$No. of Experiments = 2k * (k - 1) + C_0 \quad (1)$$

Where the number of variables is k (pH, time, and temperature). There are 12 experiments with  $C_0$  which are the 5 replications that have been enhanced to evaluate that pure error.

Table I  
Coded and Actual Values of Pectin Extraction Variables

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

### 3. Extraction of Pectin

Acidic extraction of pectin was carried out according to the method described by Pasandide et.al with some modification [17]. 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°C) 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°C 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 cheese cloth as shown in “Fig.1” 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 as shown in “Fig.2” then calculate yield % from (2)[22].

$$Yield \% = \frac{W_d}{W_p} * 100$$

(2)

Where  $W_d$ = weight of dried pectin obtained (g), and  $W_p$ =



initial weight of orange peel powder used for extraction (g).

Fig. 1: Wet Pectin



Fig. 2: Dried Pectin

### C) Characterization of orange peels pectin

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

#### 1. Fourier Transform Infrared Spectroscopy Analysis (FTIR)

Fourier transform infrared (FTIR) is an analytical methodology used to understand the structure of individual molecules and the composition of molecular mixtures. The FTIR model used is Class 1 Laser Product IEC/EN 60825-1/A2:2001 Avatar Series (USA) in The Egyptian Academy for Engineering and Advanced Technology [23].

#### 2. X-Ray Diffraction Analysis (XRD)

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 Bruker D8 advanced computerized X-ray Diffractometer apparatus with monochromatic Cu K $\alpha$  radiation which operates at 40KV and 40mA [23].

#### 3. Determination of Equivalent Weight

Equivalent weight was used for calculating the Anhydrouronic Acid content 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 are mixed in 100 ml distillate water with 6 drops of phenolphthalein with rapid stirring till 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 (3) [23].

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

#### (3) 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 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 (4) [22].

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

#### 4. 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 (5) [24].

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

#### 5. 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). Degree of Esterification was calculated from (6) [24].

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

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

#### 6. Determination of Galacturonic Acid Content (GAC)

The galacturonic acid value was determined by transferring the neutralized solution obtained during the determination of Methoxyl Content to a 250 ml measuring flask, adding 20 ml of NaOH 0.1 N, and dilute to the mark with distilled water. Then the solution stands for 1 h with agitation at room temperature then 20 ml of 0.1 mol/L hydrochloric acid and a few drops of phenolphthalein were added. The solution titrated against 0.1 N of NaOH and the spent volume of the titration was recorded as S. for the blank determination, 20 ml of HCl was titrated against 0.1 M of NaOH and the spent volume was recorded as B, and the (B-S) difference (ml) was recorded as V3. Galacturonic Acid Content was calculated from (7) [24].

$$\text{GA}\% = (\text{V1} + \text{V2} + \text{V3}) \cdot 19.41 \quad (7)$$

Where V1 is the volume of NaOH from (2), and V2 is the volume of NaOH from (3).

#### 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 105°C for 2 hours. The moisture content was calculated from (8) [23].

$$\text{Moisture Content\%} = \frac{\text{Initial weight (g)} - \text{Final weight (g)}}{\text{Initial weight (g)}} \times 100 \quad (8)$$

### 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 depends on the sample weight taken. The percentage of ash content of the sample was calculated from (9) [23].

$$\text{Ash Content \%} = \frac{\text{Weight of the ash (g)}}{\text{Weight of pectin (g)}} \times 100 \quad (9)$$

## III. RESULTS AND DISCUSSION

### A. Model fitting and statistical analysis

In this study three factors and three levels Box–Behnken response surface design (BBD) was 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 extraction yield of pectin from orange peels. The experimental design of RSM in coded and actual values of variables and the obtained yield% are shown in Table (II).

Table II  
Coded Levels and Actual Values of the Variables in Box–Behnken Design and Obtained 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

In the selection of the appropriate model that describe the relation between the independent variables (time, temperature and pH) and the response (yield) , several considerations were made to select the highest order polynomial where the additional terms are significant and there is no error in the model. It was found that the model was a second-order [26].

The second-order model equation is shown below (10)

$$Y = \beta_0 + \sum \beta_j X_i + \sum \beta_{jj} X_{ii} + \sum \beta_{ij} X_{ij} + \varepsilon \quad (10)$$

Where Y represents the predicted response,  $\beta_0$  is the model intercept, and  $\beta_j$ ,  $\beta_{jj}$ , and  $\beta_{ij}$  are the regression coefficients for the linear, quadratic, and interactive effects of the model, respectively.  $X_i$  and  $X_j$  are the factors and  $\varepsilon$  is the error of the model.

The result from the analysis of variance (ANOVA) for 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 is used to predict the yield of pectin that found to be 0.9936 which indicate 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 (III). (A, B, C, AB, BC, A<sup>2</sup>, B<sup>2</sup>, C<sup>2</sup>) 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 [24].

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

Source	Sum of Square	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 <sup>2</sup>	7.90	1	7.90	23.62	0.0018	
B <sup>2</sup>	81.33	1	81.33	243.0	<0.0001	
C <sup>2</sup>	36.64	1	36.64	109.5	<0.0001	
lack of Fit	1.87	3	0.624	5.34	0.0697	Not significant

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 (12).

$$\text{Yield} = 17.42 + 3.2073A - 3.16B + 2.8675 C + 0.3950 AB + 1.325 BC + 1.37A^2 - 4.395 B^2 - 2.95 C^2 \quad (12)$$

### B. Effects of processing factors on pectin yield

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

#### 1. Effects 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°C as shown in “Fig. 3”. This agrees with Pagan et al. (2001) and Gama et al. (2015), As temperature increases, the solubility of pectin is increased causing an increase in the rate of extraction. Nevertheless, beyond the optimum value of the temperature (90°C), the yield of pectin is reduced as a degradative action which results in pectin of minor molecular size not precipitable with alcohol. [20][23].

### 2. Effects time on pectin yield

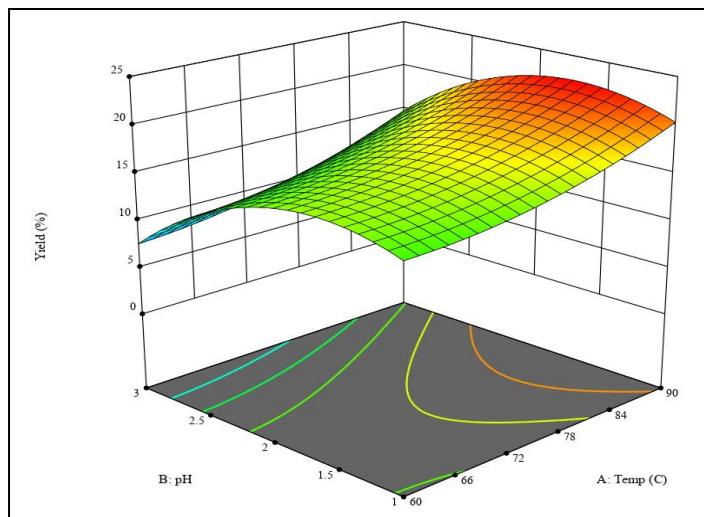
It was observed that the longer the extraction duration, the higher the pectin yield, with the maximum obtained yield at 120 min as shown in “Fig.4”. As the duration increases, the concentration of the pectin in the solution increases, leading to an increased yield. However, beyond the optimum time at higher temperatures, thermal degradation occurs which leads to decrease pectin yield. This supports the findings of El-Nawawi and Shehata (1987), Kliemann et al. (2009), Tang et al. (2011), and Gama et al. (2015) while working on pectin extraction from orange peels [20][27][28].

### 3. Effects 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. 5”. This agrees with Putnik et al. (2017), 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 temperature, the solubility of the extracted pectin decreases, hence decrease extraction rate with consequent low pectin yield, while increasing extraction time at low pH leads to a corresponding increase in pectin yield. [4][29].

#### 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 temperature of 90°C, 120 min, and pH 1 as shown in the upper left rear edge of the cubic model in “Fig.6” 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 yield was 21.52 with a standard deviation = 0.21679 which proves the validity of the suggested model as the error percent did not



exceed 3.9% [21].

Fig. 3: Response Surface Plot of the Effect of Extraction Time and Extraction pH on Dried Pectin Yield

Fig. 4: Response Surface Plot of the Effect of Extraction Temperature and Extraction pH on Dried Pectin Yield

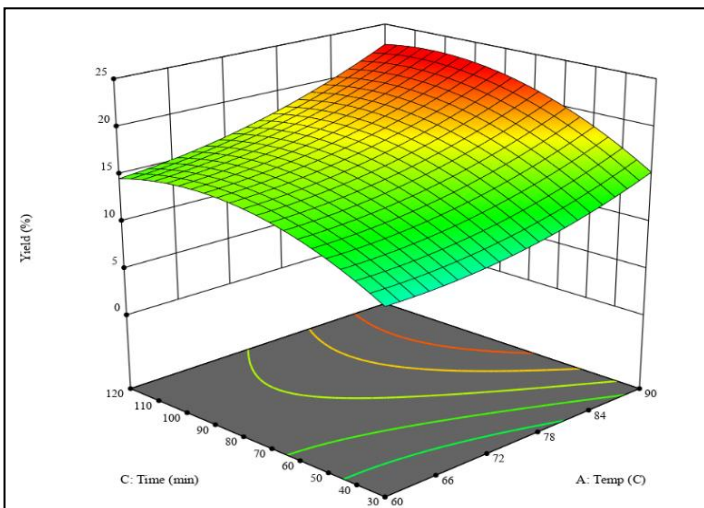
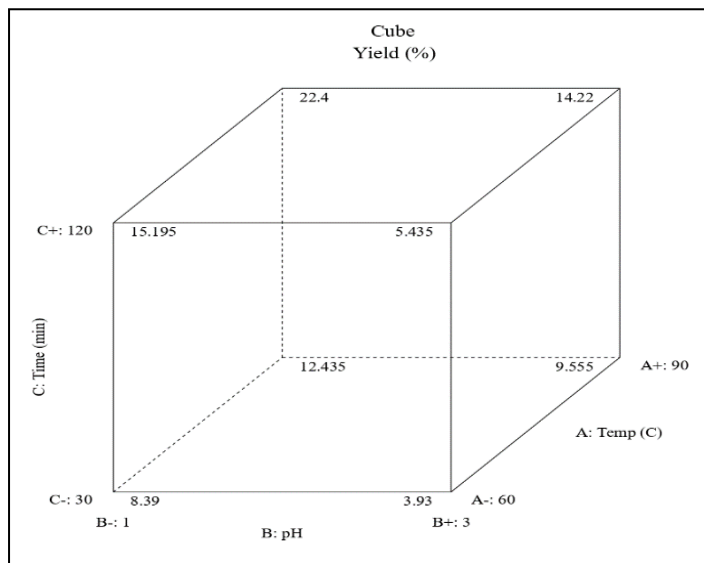
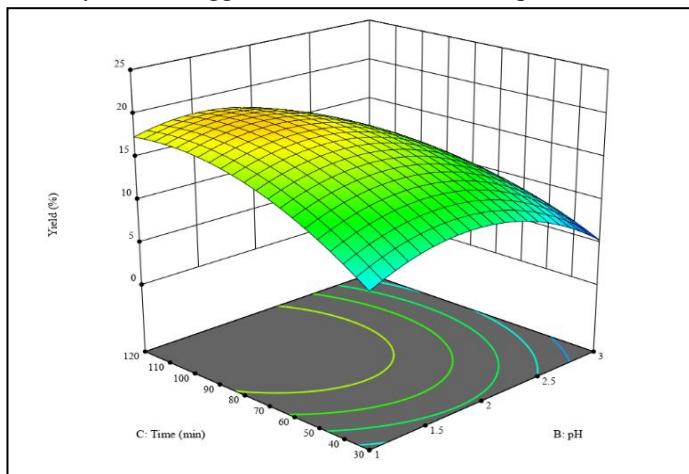


Fig. 5: Response Surface Plot of the Effect of Extraction Temperature and Extraction Time on Dried Pectin Yield



nce,

Fig. 6: 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 result of the characteristic of pectin extracted are presented as the following (see Table IV).

##### 1. Fourier Transform Infrared Spectroscopy Analysis of Pectin

In FTIR analysis each range of wavenumbers on the graph indicates different function groups of a specific polymer due to spectra absorbance. As shown in “Fig.7”, the range from 3600 to 3400  $\text{cm}^{-1}$  indicates a large number of OH groups that form hydroxyl compounds in the structure, near 2900  $\text{cm}^{-1}$  indicates the presence of CH, CH<sub>2</sub>, and CH<sub>3</sub>, while from 1740 to 1750  $\text{cm}^{-1}$  range indicates the presence of C=O groups in the structure [22].

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

Pectin shows amorphous behavior in most of the literature review. XRD analysis results show that the extracted pectin has the amorphous nature as shown in “Fig.8” [22].

##### 3. Equivalent weight

The equivalent weight of extracted pectin from orange peels was found to be 704.46 g/mol comparing to that of commercial pectin which was found to be 893 g/mol. The previous studies on pectin extraction have stated that their outcomes were ranging from 476-1209 g/mol that the weight of pectin-higher value of weight equals to better 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 [25].

##### 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 % and that of commercial pectin was found to be 9.09 %. So, the produced pectin obtained from this study was classified as high methoxyl pectin [25].

##### 5. Anhydrouronic Acid

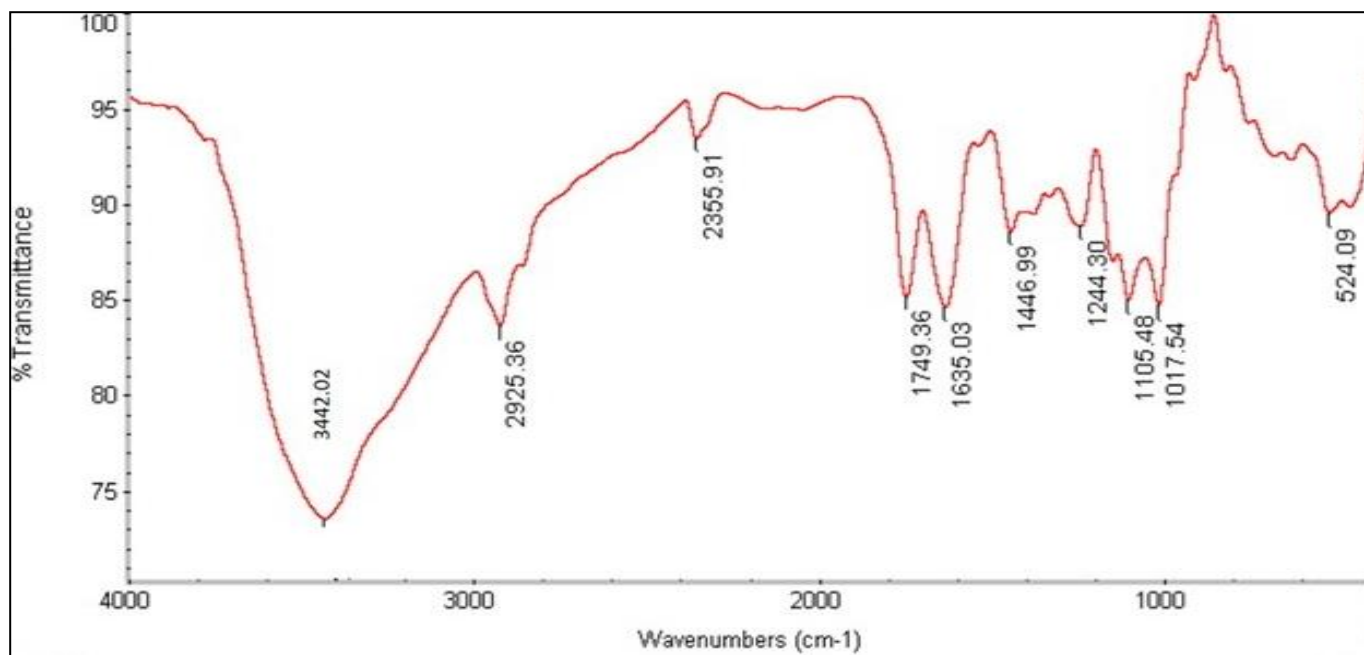
The anhydrouronic acid of extracted pectin from orange peels was found to be 89.58 % and that of commercial pectin was found to be 74.29 %. To keep the purity of extracted pectin, the value of AUA should be more than 65% according to (Food Chemical Codex IV, monograph,1996). The reason of this high value of AUA due to double washing technique to avoid any presence of impurities.[30].

##### 6. Degree of Esterification

The degree of esterification of extracted pectin using hydrochloric acid was found to be 68.28 % while commercial pectin was found to be 69.48 %. 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].

##### 7. Galacturonic Acid Contents of Pectin

Galacturonic acid content of extracted pectin from orange peels was found to be 71.8% and that of commercial pectin was ranged from 78.48% to 94.75%, which is consistent with the Food and Agriculture Organization (FAO) and regulations states that pectin must contain at least of 65% GA [30].



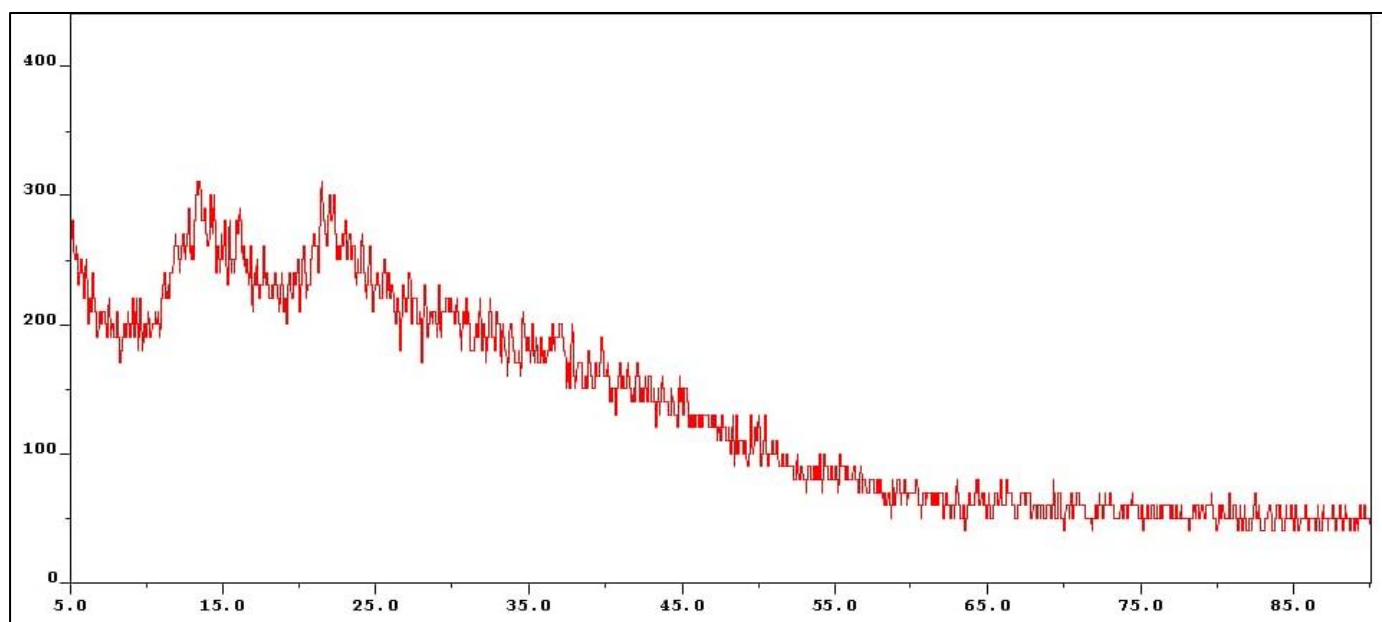


Fig. 8: X-ray diffraction (XRD) Analysis of Pectin

#### 8. 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 for standard specifications of commercial pectin. On the other hand, the ash content of pectin was found to be 2.2% which is less than the value obtained from the commercial pectin with 5%. The reason for this behaviour due to the double washing technique with ethanol which removing the residual HCl and other impurities such as fibres and sugar [30].

Table IV

The Characteristics Values of Commercial Pectin and Extracted Pectin

Characteristics	Extracted Pectin	Commercial Pectin
Equivalent Weight (g/mol)	704.46	893
Methoxyl Content (%)	10.7	9.09
Anhydrouronic Acid Content (%)	89.58	74.29
Degree of Esterification (%)	68.28	69.48
Galacturonic Acid (%)	71.817	78.48
Ash Content (%)	2.2	1.67
Moisture Content (%)	12	12.03

#### IV. Conclusion

A detailed study on the optimization of pectin extraction from orange peels has been successfully carried out. The optimization of the process parameters on pectin yield using was studied using Response Surface Methodology. It was found that the optimum conditions for pectin extraction from

orange peels were at a temperature of 90°C, pH 1 and 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%, the degree of esterification of 68.28%, the galacturonic acid 71.8%, and moisture and ash content of 12%, and 2.2% respectively. It was found that these results are within the permissible limits according to the Food and Agriculture Organization (FAO).

#### ACKNOWLEDGMENT

Special thanks to Assis. Prof. Eman Mohamed Abd El Hamid, at the Chemical Engineering Department, Egyptian Academy for Engineering and Advanced Technology, for her supervision and support. Her patience, motivation, enthusiasm, immense knowledge, her guidance helped us all the time of the research project. She has taught me how to carry out our research and to present it as clear as possible.

#### REFERENCES

- [1] TheAfricaReport, "Egypt's fresh orange exports are squeezing out other citrus crops," 27 5 2021. [Online]. Available: <https://www.theafricareport.com/91735/egypts-fresh-orange-export-is-squeezing-out-other-citrus-crops/>. [Accessed 28 6 2021].
- [2] U. S. D. o. Agriculture, "Citrus Annual : Egypt Maintains its Position as the World," 2020 dec 11. [Online]. Available: [https://citrusbr.com/wp-content/uploads/2021/03/Citrus-Annual\\_Cairo\\_Egypt\\_12-15-2020.pdf](https://citrusbr.com/wp-content/uploads/2021/03/Citrus-Annual_Cairo_Egypt_12-15-2020.pdf). [Accessed 29 July 2021].
- [3] M. Safley, W. Boyd, and R. Schmidt, "Agricultural Waste Management Systems," in *Agricultural Waste Management Field Handbook*, United State Department of Agriculture Natural Resources Conservation Service, pp. 1-37, 2011.
- [4] Putnik P, Kovacevic DB, Jambak AR, Barba FJ, Cravotto G, Binello A, Lorenzo JM, Shpigelman, "Innovative "green" and novel strategies for the extraction of bioactive added value compounds from citrus wastes," - a review. *Molecules.*, p. 22:1–24, 2017.

- [5] C.Rolin, "Pectin," in *Industrial Gums: Polysaccharides and their Derivatives*, London,, pp. 257-293, 1933.
- [6] T. Sakai, T. Sakamoto, J. Hallaert, and E.J. Vandamme, "pectin Pectinase, and Protopectinase: Production, Properties, and Applications," Academic Press, Inc., vol. 39, pp. 213-194, 1993.
- [7] T. MUHAMMED, "Modeling of orange peel oil extraction and optimization of the effect of process parameters using response surface methodology and artificial neural networks," *Elvester*, pp. 1-5, 2019
- [8] A.K. E. A. Olugbenga, "Optimization of oil and pectin extraction from orange (*Citrus sinensis*) peels: a response surface approach," *Journal of Analytical science and Technology*, vol. 9, pp. 1-16, 2018.
- [9] A. K. Tiwari, S. N. Saha, V. P. Yadav, U. K. Upadhyay, D. Katiyar, and T. Mishra, "Extraction and Characterization of Pectin from Orange Peels," *International Journal of Biotechnology and Biochemistry*, vol. 13(1), pp. 5-10, 2017.
- [10] C. S., a. V. S. G. Nitin, "Extraction of Pectin from Orange Peels and its Applications: Review," *International Journal of Innovative Research in Science, Engineering and Technology*, vol. 6, no. 9, pp. 1-6, 2017
- [11] D. K. Bhavya, Shrilakshmi, and R. Suraksha, "Value Added Products from Agriculture: Extraction of Pectin from Agro," *Research Journal of Agriculture and Forestry Sciences*, vol. 3, pp. 1-7, 2015.
- [12] M. Khan, N. Bibi, and A. Zeb, "Optimization of Process Conditions for Pectin Extraction from Citrus Peel," *Maazullah Khan, Nuclear Institute for Food and Agriculture, P.O.*, vol. 34(1), pp. 4-7, 2015.
- [13] S. S. Hosseini, F. Khodaiyan, and M. S. Yarmand, "Aqueous extraction of pectin from sour orange peel and its preliminary physicochemical properties," *Elsevier B.V.*, pp. 3- 7, 2015
- [14] M. S. Banu, G.B. Kannamma, P. Gayatri, H.Nadezhda, and J. Nandhini, "Comparative studies of pectin yield from fruits using different acids," *Elixir International Journal*, vol. 42, pp. 1-3, 2012.
- [15] Braddock, R.J. *Hand book of citrus by-products and processing technology*. John Wiley & Sons, Inc. New York. 191-7,1999.
- [16] S. A. El-Nawawi , and F. R. Shehata, "Extraction of Pectin from Egyptian Orange Peel. Factors Affecting the Extraction," *Biological Waste*, vol. 20(4), pp. 6-8, 1987.
- [17] Pasandide, B., Khodaiyan, F., Mousavi, Z., and Hosseini, S. S. ,Pectin extraction from citron peel: optimization by Box–Behnken response surface design. *Food Science and Biotechnology*, 27(4), 997–1005, (2018) .
- [18] I. V. C. J. I. K. Andre, "Response surface design and analysis," *New York.: Marcel Dekker*, 1987.
- [19] X. Peng, G. Yang, Y. Shi, Y. Zhou, M. Zhang & S. Li, "Box–Behnken design based statistical modelling for the extraction and physicochemical properties of pectin from sunflower heads and the comparison with commercial low-methoxyl pectin," *Scientific Reports*, vol. 10, pp. 1-10, 2020.
- [20] Gama B, De Farias Silva CE, Oliveira Da Silva LM, Abud AKS. Extraction and characterization of pectin from citric waste. *Chem Eng Trans.*; 44:259–64, 2015.
- [21] Chan SY, Choo WS , Effect of extraction conditions on the yield and chemical properties of pectin from cocoa husks. *Food Chem* 141: 3752-3758, (2013).
- [22] Kamnev AA, Ristic M, Antonyuk L, Chernyshev A, Ignatov VV Fourier transform infrared spectroscopic study of intact cells of the nitrogen fixing bacterium *Azospirillum brasilense*. *J Mol Struct* 408: 201-205.1997.
- [23] Pagan J, Ibarz A, Llorca M, Pagan A, Barbosa-Canovas GV. Extraction and characterization of pectin from stored peach pomace. *Food Res Int.*;34: 605–12.2001.
- [24] Yapo BM Biochemical characteristics and gelling capacity of pectin from yellow passion fruit rind as affected by acid extractant nature. *J Agric Food Chem* 57: 1572-1578,2009.
- [25] M.A. Salam, N. Jahan, M. A. Slam, and M. M. Hoque, "Extraction of Pectin from lemon peel: Technology development," *Journal of Chemical Engineering*, vol. 27(2), 2012.
- [26] T. MUHAMMED, "Modeling of orange peel oil extraction and optimization of the effect of process parameters using response surface methodology and artificial neural networks," *Elvester*, pp. 1-5, 2019
- [27] Kliemann E, de Simas KN, Amante ER, Prudencio ES, Teofilo RF, Ferreira MMC, Amboni RD. Optimization of pectin acid extraction from passion fruit peel (*Passiflora edulis flavicarpa*) using response surface methodology. *Int J Food Sci Tech.*;44:476–83.2009.
- [28] El-Nawawi SA, Shehata FR. Extraction of pectin from Egyptian orange peel. Factors affecting the extraction. *Biol Wastes.* 20:281–90,1987.
- [29] Coulson JM, Richardson JF. *Chemical engineering*, Vol. II. New York: Pergamon Press, p. 375, 1978.
- [30] M. Güzel, and Ö. Akpınar, "Valorisation of fruit by-products: Production characterization of pectins from fruit peels," *Food and Bioproducts Processing*, vol. 115, pp. 126-133, 2019.