Research Article

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# Oil and pectin extraction from *citrus paradise* (grape) peels: A case of response surface optimization


## Abstract

This work reflects the extraction process of oil and pectin from grape peels using a response surface method in which a central composite rotatable design of 2×5 and 3×5 was used for the two extractions. Output temperatures (80–100°C) and heating times (5–9 hours) were used for oil extraction, while (80 -100°C) and heating times (20 – 60 minutes) and a pH of extract (1.0 – 3.0) were selected for pectin removal. Oil yield ranged from 7.90 - 15.30%, while pectin yield ranged from 19.90 – 35.70%. A maximum oil yield of 15.30% was obtained at a temperature of 90°C at a heating time of 9.0 hours, while a maximum yield of pectin of 35.70% was obtained at a pH of 2.5, 95°C temperature and 50 minutes heating time. The optimum value for oil production was 15.63% at an average temperature of 99.64°C and heating time of 8.99 hours, while the average value of pectin yield was 38.01% at an output temperature of 94.00°C, the period release time of 58.00 minutes with a pH of 2.00. The deviation between the experimental and predicted values was low and not significant. All processing conditions have important impacts on the extraction of oil and pectin from grape peels.


**Keywords:** oil, pectin, drying temperature, reaction time, response surface methodology

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## Introduction

The importance of grape plant including its fruit, stem, leave, peel and roots to man in terms of health and economy cannot be overemphasized. Virtually, all parts grape produce is useful to man as well as the ecosystem. The fruits are used for food/wines and medicine, while the stems, leaves and roots are of medicinal values. A study revealed that grape peel inhibits activities of fungal and bacterial infections.[1] Some other studies have revealed that grape peel has the ability to reverse a cancerous system.[2–6] Grapefruits are mainly utilized by juice processing industries while the peels are generally wasted in these industries. Grape peels have higher nutritional values than the flesh itself. However, grapefruits especially the grapes (*Citrus paradise*) is one of if not the most commonly grown tree fruit in the world.[7] Additionally, grapes have commercial value and they are produced mainly for fresh consumption, but they are also addressed to the food industry mainly for the production of fruit juice. Among the grape by-products, essential oils have been produced for more than a thousand years.[1] Essential oils extracted from grape peels are very complex matrices containing numerous- compounds of different chemical classes. These compounds are generally divided into two parts: the volatile part, which is the most representative and ranges between 85 and 99% in the different cold-pressed citrus oils, and the non-volatile part, containing fatty acids, sterols, carotenoids, waxes, coumarins, and polymethoxylated flavonoids (2 – 6% of the oil), which ranges between 1 and 15%.[8] The quality and quantity of grape peel essential oils depend on many factors, such as the nature of the fruit itself, provenance, genotype, soil type, climate and the extraction process. Although, Zy et al.[9] have shown that the by-products from grape juice processing represent a serious problem for the industry, given their limited applications and low added value. Grape peel is a primary by-product from extraction and if not re-used, becomes waste and a possible source of environmental pollution. The food processing industry is among the areas that generate large amounts of waste with possibilities for use.[10] On the other hand, pectin is produced commercially in the form of white to light brown powder mainly extracted from grapefruits. It is a group of polysaccharides that are rich in galacturonic acids.[11] Studies have shown that suitable methods were utilized to convert orange peel into value-added products such as essential oil and pectin. Pectin is a methylated ester of polygalacturonic acid[12] extracted from citrus peels and apple pomace under mildly acidic conditions. In the food industry, pectin has been widely applied as a thickening, gelling, and emulsifying agent for jams, soft drinks, fish and meat products, fruit juice, desserts and dairy products.[13,14] It is useful in medicinal applications, in which it helps in lowering serum cholesterol level, removing heavy metal ions from the body, stabilizing blood pressure and restoring intestinal functions[15] and weight reduction.

Generally, the peels and pomace of fruits are disposed as industrial wastes or being used for animal feeding, yet they have been reported to be a potential source of pectin.[16–19] Therefore, the present study seeks to investigate the effects of processing conditions of pectin and oil expressions from grape peels by optimizing the pectin and oil yield and process parameters using response surface methodology (RSM).

## Materials and methods

### Sample preparation

Fresh grapes were purchased from a local market at Ukam, Mkpat Enin, Akwa Ibom State (see Figure 1a). They were skinned and the outer cover was removed, which was then cut into smaller pieces. It was divided into two parts and pre-heated for 1-2 hours (Figure 1b). The dried outer cover obtained was ground (Figure 1c) to provide a consistent and attractive particle (this was important to prevent clumping during solvent extraction) and stored at room temperature for further use.

**Figure 1** Grapefruit (left); dried peels (center), ground peels (right).

## Experimental design for oil and pectin extraction

The experimental design adopted for oil extraction were 2 factors, 5 levels, and for pectin were 3 factors, 5 levels, factorial Central Composite Rotatable Design (CCRD) from Response Surface Methodology[20] respectively. According to the CCRD method, the total number of treatment combinations is:

$$n = 2^{k}(n_f) + 2k(n_a) + k(n_c) \quad [1]$$

where 'k' is the number of independent variables and n is the number of repetitions of experiments at the center point. Additionally, the total number of design points[21–24] is given as:

$$N = 2^{k} + 2k + n_0 \quad [2]$$

Therefore, the CCRD for oil extraction involved 13 experiments of $2^2$ factorial Central Composite Design (CCD), with 4 axial points (α is 2) and 5 replications at the center points and the CCRD for pectin extraction involved 20 experiments consisting of 23 factorial CCD, with 6 axial points (α is 2) and 6 replications at the center points. For each independent variable, the levels were chosen with respect to preliminary experiments and previous reports literatures. For oil extraction from the grape peel, five drying temperatures (80, 85, 90, 95 and 100°C) and heating times of (5, 6, 7, 8 and 9 hours) were selected (Table 1). Also, for pectin extraction 5 pH levels (1.0, 1.5, 2.0, 2.5 and 3.0), temperatures (80, 85, 90, 95, and 100°C) and extraction times of (20, 30, 40, 50, and 60 minutes) were chosen (Table 1). The coded values of the independent variables (-2, -1, 0, 1, 2) were used; where -2, 0 and 2 represent the lowest, medium and highest levels respectively, as shown in Table 1.

**Table 1** Showing the levels, codes and actual values of independent variables

| Factors | Unit | Code | Level | | | | | Interval |
|---|---|---|---|---|---|---|---|---|
| | | | -2 | -1 | 0 | 1 | 2 | |
| **Oil extraction** | | | | | | | | |
| Drying temperature | °C | $X_1$ | 80 | 85 | 90 | 95 | 100 | 5 |
| Heating time | hr | $X_2$ | 5 | 6 | 7 | 8 | 9 | 1 |
| **Pectin extraction** | | | | | | | | |
| pH | | $X_1$ | 1 | 1.5 | 2 | 2.5 | 3 | 0.5 |
| Extraction temperature | °C | $X_2$ | 80 | 85 | 90 | 95 | 100 | 5 |
| Extraction time | min | $X_3$ | 20 | 30 | 40 | 50 | 60 | 10 |

The dependent variables are the parameters affecting the process of oil and pectin yield.[22,25,26] The empirical expression is represented in Equation (3) as:

$$Y = \beta_0 + \sum_{i=1}^{2}\beta_i X_i + \sum_{i=1}^{2}\beta_{ii} X^2{}_i + \sum_{i=1}^{2}\sum_{j=i+1}^{2}\beta_{ij} X_i X_j \quad [3]$$

where $Y$ is the response; $\beta_0$ is a constant term; $\sum_{i=1}^{2}\beta_i$ is the summation of the coefficient of linear terms;

$\sum_{i=1}^{2}\beta_{ii}$ is summation of quadratic terms; $\sum_{i=1}^{2}\sum_{j=i+1}^{2}\beta_{ij}$ is the summation of the coefficient of interaction terms; $X_i X_j$ are independent variables.

## Extraction of oil from grape peels using Soxhlet method

A round bottom flask was washed thoroughly, oven-dried and cooled in a desiccator. Then, 5g dry mass of the puree was measured and labelled as S1. The weighted sample was muffled in the filter, tied

**Citation:** Olatunji OM, Umani KC, Ekanem JT, et al. Oil and pectin extraction from *citrus paradise* (grape) peels: A case of response surface optimization. *MOJ Food Process Technol.* 2021;9(2):44–55. DOI: 10.15406/mojfpt.2021.09.00259

using a thread and placed in the Soxhlet extractor. Again, n-hexane was added until it siphoned once and more hexane was added until the volume of the extractor was half full. It was ensured that the joints of the condenser were tight and the cooling water was circulating. On extraction of puree, the temperature of heating mantle was set at 85°C and kept to boil in the round bottom flask for a period of 6 hours (see Figure 2). This experimental method has been employed by other researchers. See detail of oil extraction procedure in the works of Fakayode et al.[22]. However, the percentage yield of oil was calculated using Equation (4) as:

$$Y_{oil} = (W_2 - W_1) \times \frac{100}{S} \quad [4]$$

where $Y_{oil}$ is oil yield from grape peels (%), $W_1$ is the weight of empty flask (g), $W_2$ is the weight of flask and extracted oil (g), $S$ is the weight of sample (g).

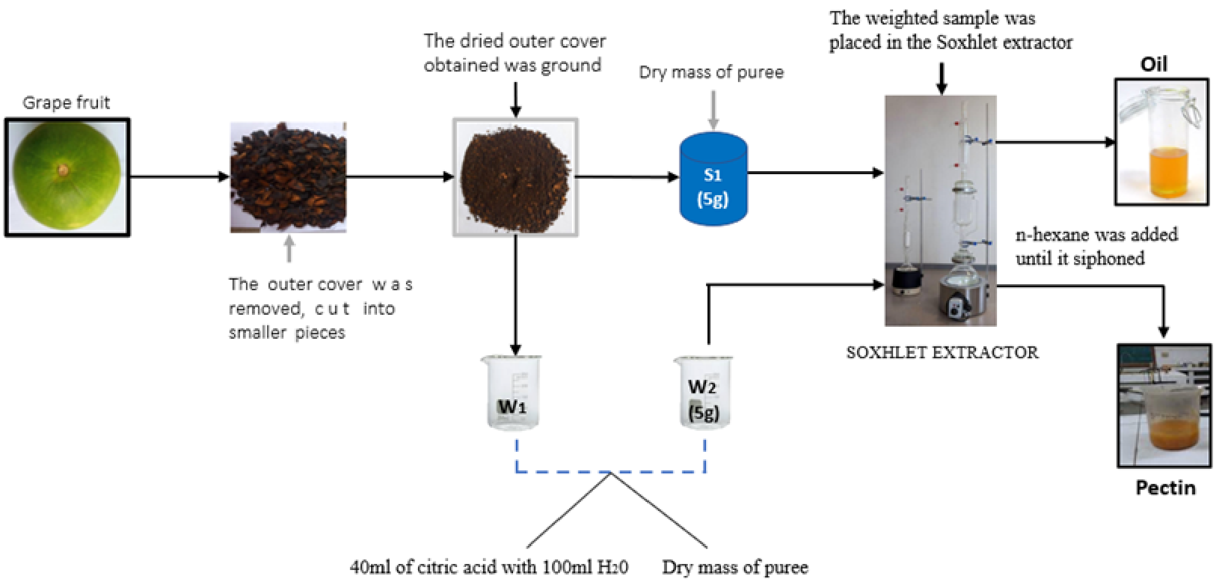



**Figure 2** Flow diagram describing the oil and pectin extraction from grape fruit.

## Pectin extraction from grape peels

A beaker was washed, oven-dried and weighed as $W_1$. Then, 40 *ml* volume of 90% citric acid was diluted with 100 *ml* distilled water in a beaker at a pH of 2.0. A dry mass of *5 g* of the puree was introduced into the beaker and the weight was recorded as $W_2$. This procedure of extraction has also been used in past works for different agro-product. See details in the report of Fakayode et al.[22].

Finally, the precipitate was dried at 55°C in an oven and the weight was recorded. The experiment was done repeatedly using different volumes of citric acid ranging from 60, 70, 80, 90, 100 to 120 *ml* with a constant volume of distilled water of 100 *ml* at varying pH levels, extraction temperature, and extraction time respectively. Also, different volumes of ethanol from 60, 70, 80, 90, 100, to 120 *ml* which corresponds with the volume of citric acid were used to coagulate the filtrate (see Figure 2). The percentage yield of pectin was based on the gram of peel sample taken and was calculated using the expression in Equation (5) as:

$$Y_{pec} = 100 \times \frac{P}{B_i} \quad [5]$$

where, $Y_{pec}$ is the extracted pectin yield (%), $P$ is the amount of dry pectin (g), $B_i$ is initial amount of grape peel (g).

## Statistical analysis

In the present study, design expert version 11 from stat ease was employed to design the experimental procedure for oil and pectin extraction from grape peels using Response Surface Methodology (RSM). Linear two-factorial interaction (2FI), quadratic, and cubic models were developed in the cause of the analyses and these models were fitted to the experimental data. Also, analysis of variance (ANOVA) was utilized to determine the significance and fitness of the model as well as the effect of significant individual terms and their interaction on the response variables. The p-value showed the level of significance for each regression coefficient which also indicated the interaction effect of each cross product. Data obtained from the experiments were statistically analyzed to determine the significant difference in the extraction process and their interactions at 5% probability level using Minitab 17.

# Results and discussion

## Oil and pectin yields

The average summaries of the grape peel oil and pectin yields are presented in Tables 2 & 3 respectively.

**Citation:** Olatunji OM, Umani KC, Ekanem JT, et al. Oil and pectin extraction from *citrus paradise* (grape) peels: A case of response surface optimization. *MOJ Food Process Technol.* 2021;9(2):44‒55. DOI: 10.15406/mojfpt.2021.09.00259

**Table 2** Oil yield from grape peel at various processing conditions

| Runs | Drying temperature (℃) | Heating time (hr) | Oil yield (%) |
|---|---|---|---|
| 1 | 85 | 6 | 9.3 |
| 2 | 100 | 7 | 11.1 |
| 3 | 85 | 8 | 7.9 |
| 4 | 95 | 8 | 15.3 |
| 5 | 90 | 7 | 10.7 |
| 6 | 90 | 7 | 10.9 |
| 7 | 90 | 9 | 10.3 |
| 8 | 90 | 5 | 9.1 |
| 9 | 90 | 7 | 10.8 |
| 10 | 90 | 7 | 11 |
| 11 | 90 | 7 | 9 |
| 12 | 80 | 7 | 10.2 |
| 13 | 95 | 6 | 10.3 |

**Table 3** Pectin yield from grape peel at various processing conditions

| Runs | pH | Extraction temperature (℃) | Extraction time (min.) | Pectin yield (%) |
|---|---|---|---|---|
| 1 | 2 | 90 | 40 | 23.1 |
| 2 | 2 | 90 | 40 | 23.1 |
| 3 | 1.5 | 95 | 50 | 20.3 |
| 4 | 1 | 90 | 40 | 24.7 |
| 5 | 2.5 | 85 | 30 | 21.4 |
| 6 | 2 | 80 | 40 | 29.4 |
| 7 | 2 | 90 | 40 | 25 |
| 8 | 2 | 90 | 40 | 25.6 |
| 9 | 2 | 90 | 40 | 23.2 |
| 10 | 2.5 | 95 | 30 | 21.7 |
| 11 | 1.5 | 85 | 30 | 20.8 |
| 12 | 2 | 90 | 40 | 24.3 |
| 13 | 2 | 90 | 20 | 20 |
| 14 | 1.5 | 85 | 20,00 | 19.9 |
| 15 | 3 | 90 | 40 | 32.3 |
| 16 | 2.5 | 95 | 50 | 35.7 |
| 17 | 2.5 | 85 | 50 | 28 |
| 18 | 1.5 | 95 | 30 | 27.4 |
| 19 | 2 | 100 | 40 | 32.7 |
| 20 | 2 | 90 | 60 | 33.8 |

## Effects of processing conditions on oil yield

The yield of oil from grape peels ranged from 7.90–15.30% (Table 2). It was observed from Table 2 that with an increase of extraction temperature the oil yield increases and it is maximum at 95℃. Furthermore, an increase in time with temperature increases the yield up to an extent and an increase in time has no effect at lower temperatures as observed in Figure 2. As reported by Sharma et al.[27], in simple distillation methods, an increase of distillation time increases the oil yield and it is maximum at a certain point. Also, a further increase in time from 95℃ has no effect on oil yield.

It was observed that as the extraction temperature and time increases, the in essential oil yield also increases. However, at higher temperatures and heating times, beyond the optimum level, the oil yield decreases. This corroborates the findings of Giwa et al.[28] and Fakayode et al.[22], who reported an increasing trend on oil extraction from orange peels using the water distillation method. Again, at low temperatures, steam travels through the grape peels slowly and the built-up pressure is not sufficient enough to extract the oil. Additionally, as the temperature increases for a very long period of time, the oil eventually breaks out of the peel matrix. Therefore, increasing the extraction time at higher temperatures will amount to substantial moisture loss leading to the hardening of peels which consequently leads to a reduction in the oil yield.

## Effects of extraction process conditions on pectin yield

The pectin yield ranged from 19.9 – 35.70% (Table 3). The effects of these factors (pH, temperature, time) on pectin yield showed that the maximum yield from the grape peel sample was found to be 35.70% at pH of 2.5, the temperature of 95°C and time of 50 min (Table 3). Thus, a pH of 2.5 gives the optimum value for the extraction of pectin from the grape peels used. The optimum temperature for pectin extraction was observed to be 95°C and this shows that higher temperature levels influence the yield of pectin. At high temperature, it is highly combustible indicating a greater effect on the yield because a reduction in temperature reduces the yield. Again, it was observed that at very low or moderate temperatures, the yield of pectin was greatly reduced.

At lower temperatures, the time of extraction has less effect on the yield of pectin, but at a higher temperature, the maximum level increases the yield of pectin and shows no form of thermal degradation on the extracted pectin. Kanmani et al.[29] reported that the maximum yield of pectin was obtained from moderate conditions (60 - 75°C). In Figure 3, it was observed that increase extraction time at low pH causes an increase in pectin yield. Similarly, very low pH indicates high level of acidity which increases the pectin extraction yields.[30] Although, as the extraction process proceeds, the pectin concentration in the solution increases as well. However, at increased time duration, the extraction rate gradually reduces because the low concentration gradient which makes the solution more viscous. This is in agreement with the observation of Coulson et al.[31] and Maxwell et al.[32]

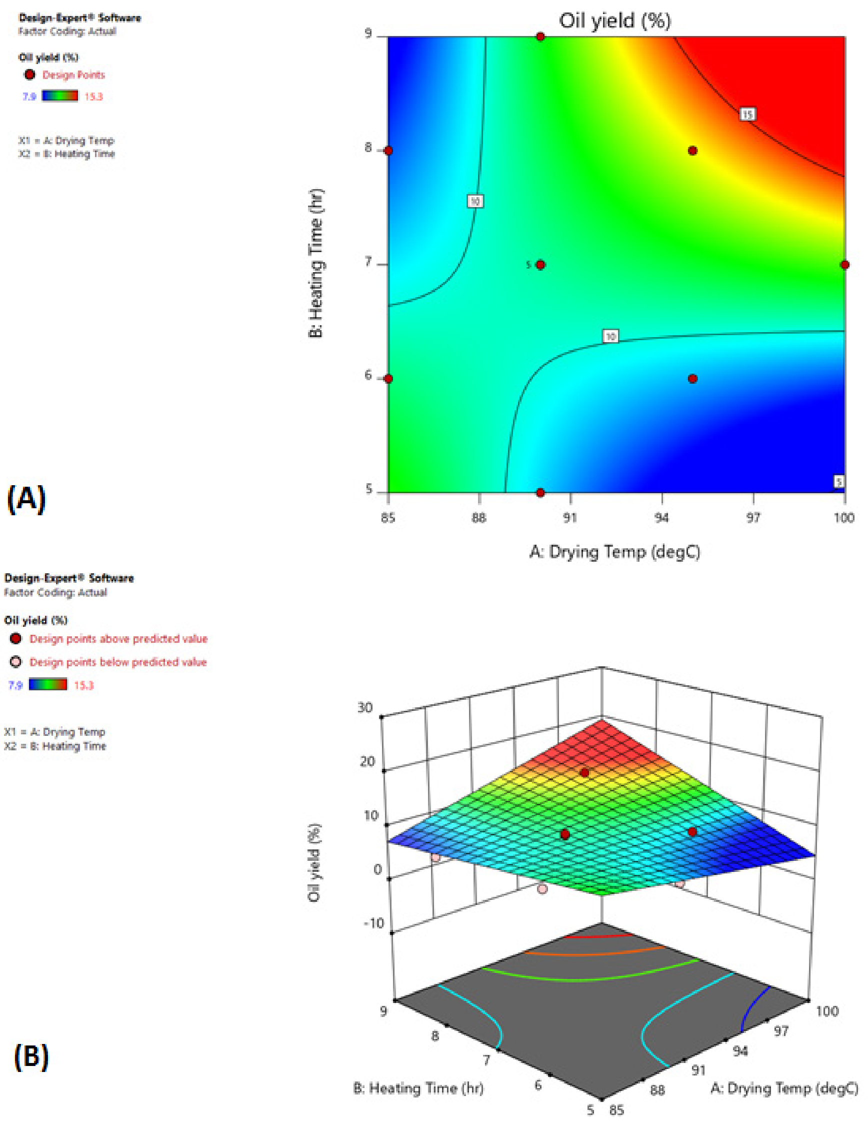



**Figure 3** Response surface contour and 3D plot of the effect of drying temperature and heating time on oil yield.

At high extraction temperature and low pH, the pectin yield increases (see Figure 4). According to Putnik et al.[30], high acidity level causes an increase in pectin extraction yield. This is as a result fractionation of glycosidic bonds in the neutral sugars due to pH sensitivity which corroborates the findings of Pagan et al.[33] and Pagan et al.[34]. From Figure 4, it was observed that an increase in extraction time and temperature leads to an increase in pectin yield, were the optimum temperature and time are 95 °C and 105 mins respectively. Again, this is in agreement with the findings of Pagan et al.[34], Fakayode et al.[22] and Mollea et al.[35] on pectin extraction from peach pomace, cocoa husks, and citric wastes (Figures 5 & 6).

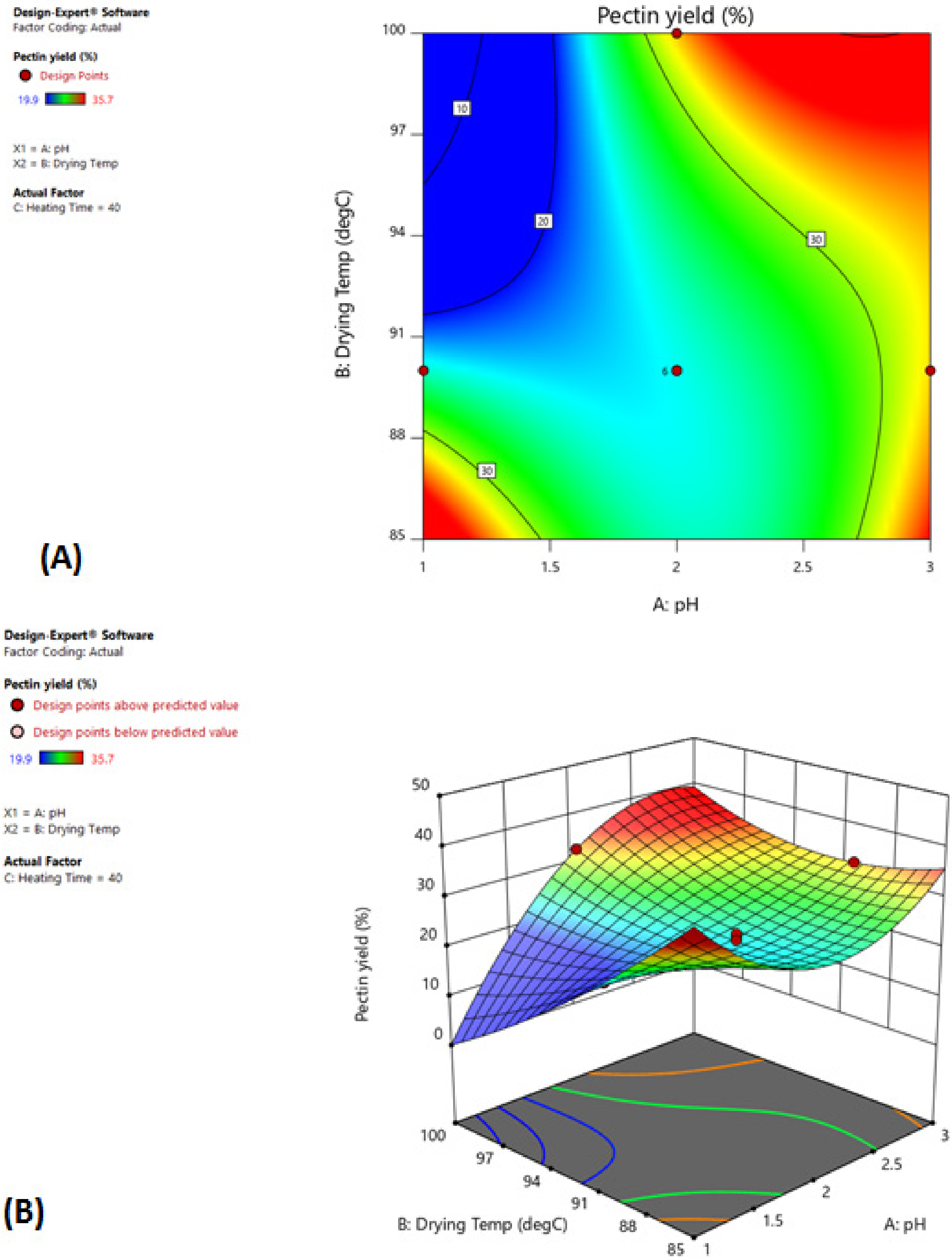



**Figure 4** Response Surface Contour and 3D Plot of the Effect of pH and Extraction Temperature on Pectin Yield.

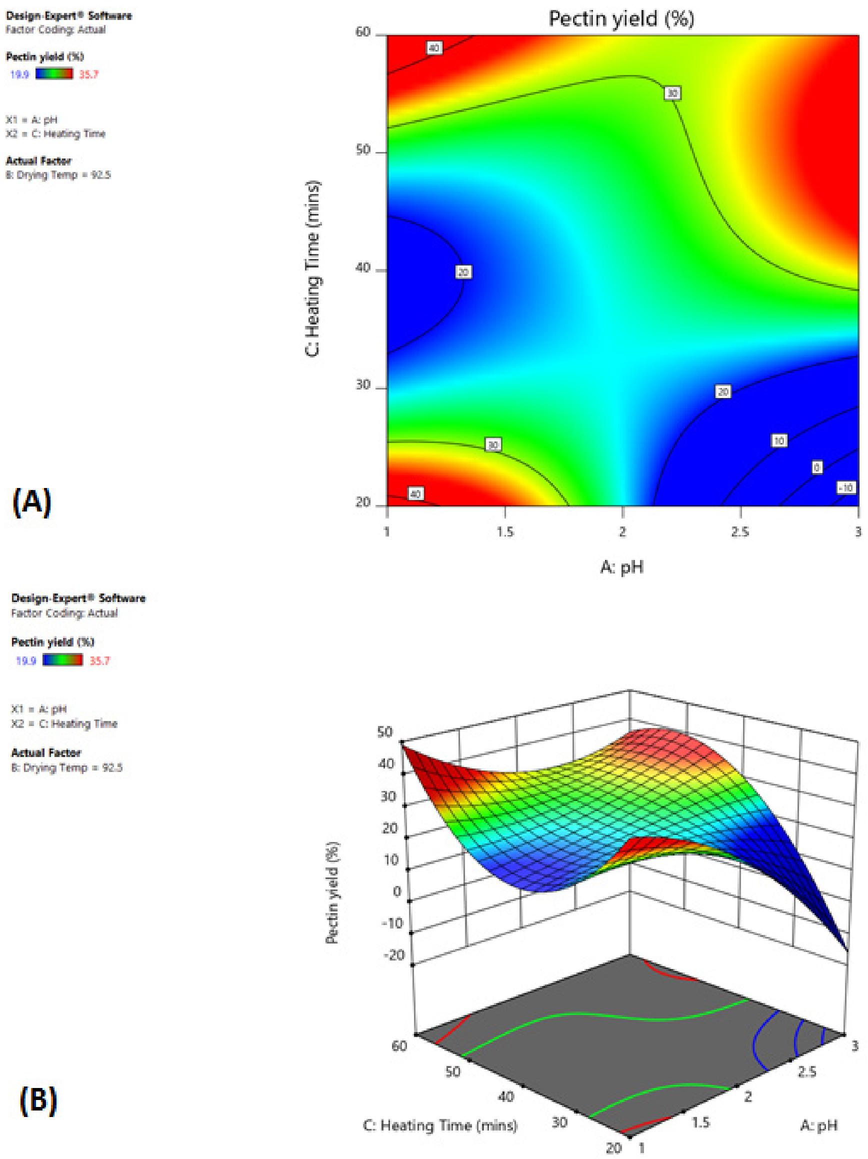
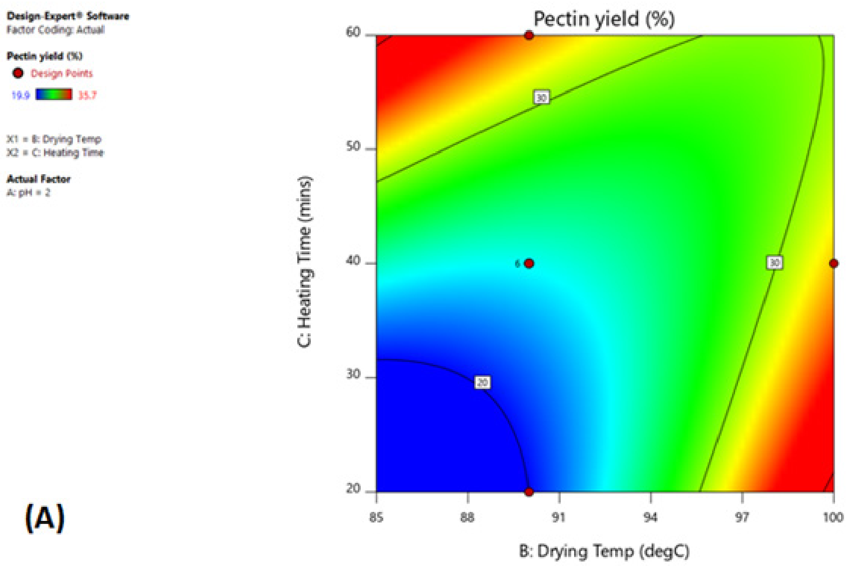



**Figure 5** Response Surface Contour and 3D Plot of the Effect of Extraction Time and pH on Pectin Yield.



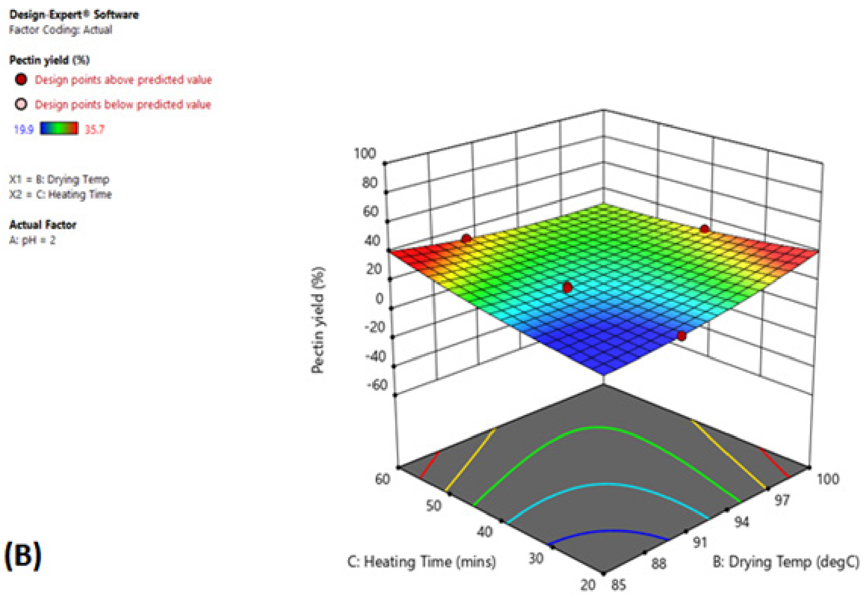



**Figure 6** Response Surface Plot of the Effect of Extraction Time and Extraction Temperature on Pectin Yield.

## Predictive models selection for the oil and pectin yield from grape peels

In the present study, different models were developed from the response surface analysis which include linear, two factorial interaction (2FI), quadratic, and cubic for the prediction of oil and pectin yield. These models were fitted to the experimental data using design expert. The appropriate model was selected based on the highest number of significant terms and the coefficient of correlation. Considering these, quadratic models were chosen for the oil and pectin yield (Tables 4 & 5). The final equations for the oil and pectin yields are given in Equations (3) and (4) respectively.

**Table 4** Model comparison for oil yield (%)

| Sources | Linear | 2FI | Quadratic | Cubic |
|---|---|---|---|---|
| Std. Dev. | 0.7431 | 0.7817 | 0.7966 | 0.759 |
| $R^2$ | 0.7765 | 0.7774 | 0.8202 | 0.8834 |
| Adjusted $R^2$ | 0.7318 | 0.7032 | 0.6919 | 0.7203 |
| Predicted $R^2$ | 0.5012 | 0.2676 | -0.3791 | -11.2846 |
| Press | 12.33 | 18.1 | 34.08 | 303.56 |

**Table 5** Model comparison for pectin yield (%)

| Sources | Linear | 2FI | Quadratic | Cubic |
|---|---|---|---|---|
| Std. Dev. | 3.1 | 2.89 | 2.78 | 0.9988 |
| $R^2$ | 0.6642 | 0.7621 | 0.831 | 0.9891 |
| Adjusted $R^2$ | 0.6012 | 0.6523 | 0.679 | 0.9585 |
| Predicted $R^2$ | 0.4031 | -0.1771 | -0.4443 | N/A |
| Press | 272.74 | 537.87 | 659.96 | N/A |

$$Y_{oil} = -80.89 + 1.46DT + 4.80HT - 0.015DT \times HT - 0.007DT^2 - 0.18HT^2 \quad (3)$$

$$Y_{pec} = +239.11 + 6.67pH - 4.99ET - 3.54Et - 0.028pH \times ET + 0.042pH \times Et + 0.034ET \times Et + 2.90pH^2 + 0.026ET^2 + 0.009Et^2 \quad (4)$$

where $Y_{oil}$ is oil yield from the grape peel (%), $Y_{pec}$ is pectin yield from grape peels (%), $DT$ is drying time (hr), $HT$ is heating temperature (°C), $pH$ is pH, $ET$ is Extraction Temperature or drying temperature (°C), $Et$ is the extraction time (min.) The ANOVA model for the selected for percentage oil and pectin yields from grape peels are presented in Tables 6 & 7 respectively. In equations 3 and 4, the positive terms signify a direct relationship between the oil and pectin extraction process conditions and their interactions with oil yield (OY) and pectin yield (PY), while the negative terms signify an inverse relationship between them. It was observed that all the oil expression process conditions have a direct relationship with OY and PY. This implies that OY and PY exhibited an increase with an increase in the expression process conditions. Heating time was found to be the most significant parameter which affects OY. This agrees with the findings of Mollea et al.[35] on cocoa husks, Pagan et al.[34] on peach pomace, Fakayode et al.[22] on orange peels, Kanmani et al.[29] and Khan et al.[26] on citrus peels.

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**Table 6** ANOVA for the effects of oil extraction conditions on oil yield (%)

| Source | SS | DF | MS | F-value | p-value | Remark |
|---|---|---|---|---|---|---|
| Model | 20.27 | 5 | 4.05 | 6.39 | 0.0153 | Significant |
| A-Drying Temperature | 8.17 | 1 | 8.17 | 12.87 | 0.0089 | Significant |
| B-Heating Time | 11.02 | 1 | 11.02 | 17.37 | 0.0042 | Significant |
| AB | 0.0225 | 1 | 0.0225 | 0.0355 | 0.856 | Not Significant |
| $A^2$ | 0.6294 | 1 | 0.6294 | 0.9918 | 0.3525 | Not Significant |
| $B^2$ | 0.7279 | 1 | 0.7279 | 1.15 | 0.3197 | Not Significant |
| Residual | 4.44 | 7 | 0.6345 | | | |
| Lack of Fit | 4.17 | 3 | 1.39 | 20.5 | 0.0068 | Significant |
| Pure Error | 0.2712 | 4 | 0.0678 | | | |
| Cor Total | 24.71 | 12 | | | | |

**Table 7** ANOVA for the effects of pectin extraction conditions on oil yield (%)

| Source | SS | DF | MS | F-value | p-value | Remark |
|---|---|---|---|---|---|---|
| Model | 379.73 | 9 | 42.19 | 5.46 | 0.0069 | Significant |
| A-pH | 90.46 | 1 | 90.46 | 11.72 | 0.0065 | Significant |
| B-Extraction Temperature | 57.72 | 1 | 57.72 | 7.48 | 0.021 | Significant |
| C-Extraction Time | 149.54 | 1 | 149.54 | 19.37 | 0.0013 | Significant |
| AB | 2.93 | 1 | 2.93 | 0.3793 | 0.5517 | Not Significant |
| AC | 0.3415 | 1 | 0.3415 | 0.0442 | 0.8376 | Not Significant |
| BC | 22.64 | 1 | 22.64 | 2.93 | 0.1176 | Not Significant |
| $A^2$ | 13.19 | 1 | 13.19 | 1.71 | 0.2204 | Not Significant |
| $B^2$ | 10.2 | 1 | 10.2 | 1.32 | 0.2772 | Not Significant |
| $C^2$ | 20.55 | 1 | 20.55 | 2.66 | 0.1338 | Not Significant |
| Residual | 77.2 | 10 | 7.72 | | | |
| Lack of Fit | 72.22 | 5 | 14.44 | 14.48 | 0.0054 | Significant |
| Pure Error | 4.99 | 5 | 0.9977 | | | |
| Cor Total | 456.93 | 19 | | | | |

For oil yield, the *M*odel *p*-value of 0.0153 (Table 6) which is less than the chosen α- a level of 0.05 implies that the model is significant. The Lack of Fit *p*-value of 0.0068 implies the Lack of Fit is significant. The model terms *p*-values (Prob. > F) of 0.0089 and 0.0042 (Table 6) which are less than the chosen α-level of 0.05 indicate model terms are significant. In this case, *A* and *B* are significant model terms (Table 6). This implies that the drying time and the heating temperature have significant effects on oil yield (OY) with the heating temperature having the greatest influence on OY. Therefore, the two oil extraction conditions influenced the oil yield from grape peels. It was also found that the model was significant with a satisfactory coefficient of determination ($R^2$ =0.8202). The high coefficient of determination showed excellent correlations between the independent variables. This value indicates that the response model (OY) can explain 82.02% of the total variability in the response.

For pectin yield, the model *p*-value of 0.0069 (Table 7) which is less than the chosen α-level of 0.05 implies that the model is significant. The Lack of Fit *p*-value of 0.0054 implies the Lack of Fit is significant, insignificant Lack of Fit is good. The model terms *p*-values (Prob > F) are all less than the chosen α-level of 0.05 which implies that the model terms are significant. In this case, *A, B,* and *C* are all significant model terms (Table 7). This implies that the pH, extraction temperature and the extraction time have significant effects on pectin yield (PY) with the extraction time having the greatest influence on PY. Therefore, the three pectin extraction conditions influenced the pectin yield from grape peels. It was also found that the model was significant with a satisfactory coefficient of determination ($R^2$ =0.8310). The high coefficient of determination showed excellent correlations between the independent variables. This value indicates that the response model (PY) can explain 83.10% of the total variability in the response.

## Optimization and model validation

For the oil extraction, in the range of 80 – 100°C for extraction temperature and 5 – 9 hours for a Heating time where the goal for oil yield (OY) was maximum, the predicted oil yield of 15.63% at extraction temperature of 99.64˚C and heating time of 8.99 hours was obtained with the desirability of 0.89. Under these optimal conditions, the experimental value was 15.47%. For the pectin extraction, in the range of 80 – 100˚C for extraction temperature, 20 – 60 min for extraction time, and 1.0 – 3.0 for extraction pH where the goal for pectin yield was maximum, RSM predicted pectin yield of 38.01% at extraction temperature of 94.00˚C, extraction time of 58.00 min, and extraction pH of 2.00 with the desirability of 1.00. This was experimentally validated as 37.84%. There was an excellent agreement between the actual and predicted values for the essential oil and pectin extractions (Figure 7). Deviations between experimental and predicted values were low and statistically insignificant for both extractions. This shows that the models chosen can adequately predict oil and pectin yields.

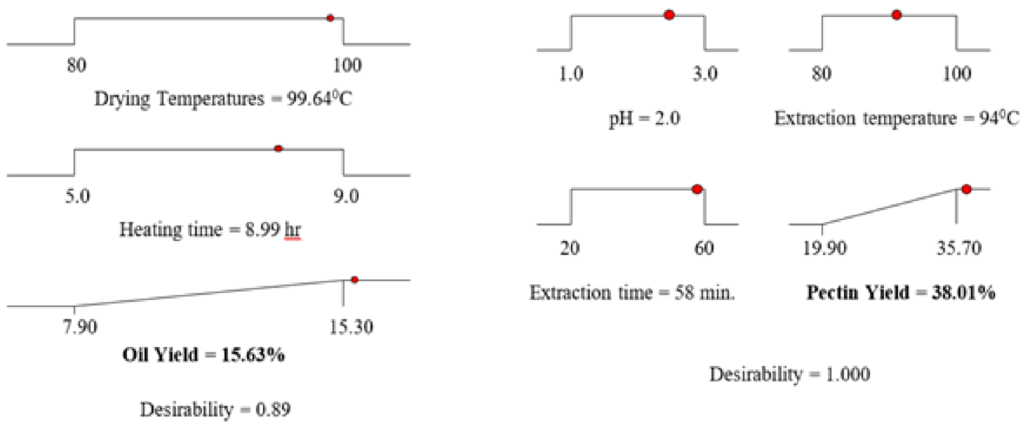


**Figure 7** Ramp for optimization of oil and pectin yield.

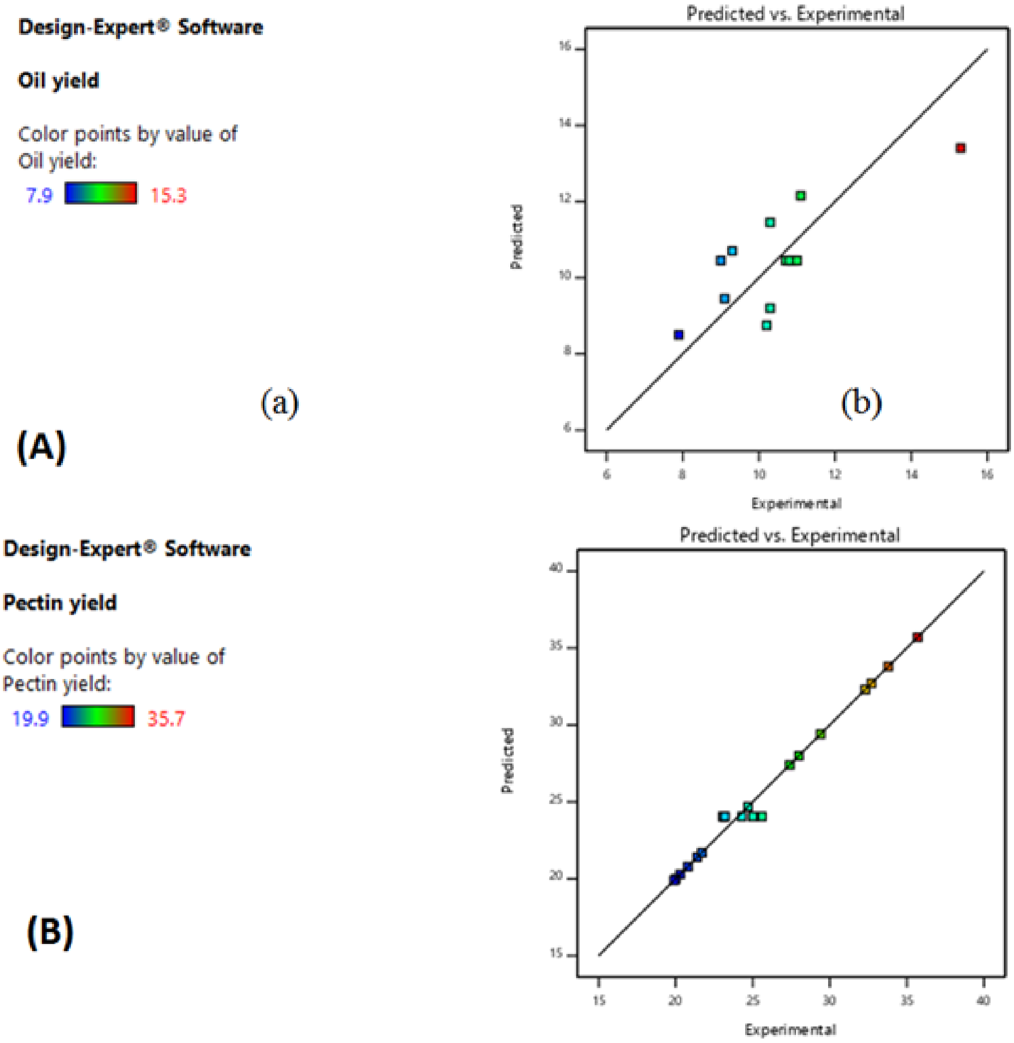


**Figure 8** Comparison of the predicted and experimental values for (a) oil and (b) pectin yield.

## Conclusion

This study has facilitated a detailed investigation of oil and pectin from grape peels, products of the enormous value of food-industry applications. Initially, the maximum yield of pectin was found to be 35.70% from grape peels at pH of 2.5, the temperature of 95˚C and a time of 50 min. Temperature, pH, and extraction time played a significant role in the yield of pectin and the levels of these factors were optimized. The predicted optimum value was 38.01% pectin yield at a heating temperature of 94˚C, the heating time of 58 mins and pH of 2 with the desirability of 1. Under these optimal conditions, the experimental value was 37.84%. The deviations between experimental and predicted values were low and statistically insignificant which implies that the various models selected could actually predict the pectin extraction from grape peels. For the grape peel oil, the maximum oil yield was found to be 15.30% at temperature 90˚C and extraction time of 9 hours. The predicted optimum value was 15.63% oil yield at a heating temperature of 99.64˚C and heating time of 8.99 hours with the desirability of 0.890. Under these optimal conditions, the experimental value was 15.47%. The deviations between experimental and predicted values were low and statistically insignificant which implies that the various models selected could actually predict the pectin extraction from grape peels. The environmental pollution that arises due to the disposal of grape peel can be overcome by using the same for grape peel oil and pectin extractions. Therefore, the disposal problem of the residue of grape peels after extracting the oil can be overcome by vermicomposting.

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## Conflicts of interest

The authors declare that there is in no conflict of interest.

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