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Extracting juice from dates (*Phoenix dactylifera* L.) using response surface methodology: Effect on pH, vitamin c, titratable acidity, free amino nitrogen (FAN) and polyphenols
Optimization of date juice extraction process from "Bournow" cultivar: Effects of temperature, time, volume/mass ratio, and enzyme volume on Bioactive Compounds

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ABSTRACT

The aim of this text is to investigate the effects of temperature, time, volume/mass ratio, and enzyme volume on various properties of date juice extracted from the "Bournow" cultivar, and to optimize the extraction process to maximize the levels of free amino acids, total polyphenols, vitamin C, and pH. The extraction technique was used to obtain date juice for use in the beverage industry from the "Bournow" cultivar. The effects of temperature, time, volume/mass ratio, and enzyme volume on free amino acid, total polyphenols, vitamin C, and pH levels were then investigated using a design centered on four parameters. All multivariate polynomial models of second degree with interactions were discovered and validated. To optimize responses, multiresponse optimization was utilized. The center composite design (CCD) determined the following response ranges: 4.21 to 5.62 pH; 45 to 126.66 mg/L vitamin C; 2.7 to 6.87 g GA/100 g total polyphenols; and 429.94 to 615.55 mg/L free amino acids. The selected factors had varying effects on the pH, vitamin C, total polyphenols, and free amino acid responses, with simple, quadratic, and interaction contributions leading to significant increases or decreases. Multi-response optimization, the objective of which was to maximize all responses besides pH in order to produce an abundant juice, resulted in the following compromise: 95 °C temperature; 10 min duration; 2:1 water/pulp ratio; and 0.5 ml of pectinase. The optimal values simulated yielded the following respective maxima: pH is 4.13; vitamin C is 116.5 mg/L; total polyphenols are 6.25 g GA/100 g; and free amino acids are 587.88 mg/L. This study successfully optimized the extraction technique for obtaining date juice from the "Bournow" cultivar. The results provide valuable insights for the beverage industry. Future prospects include further research on the sensory properties and shelf life of date juice, as well as exploring its potential applications in other food and beverage products.

1. Introduction

Dates (*Phoenix dactylifera* L.) are a globally significant fruit crop due to their nutritional value and economic advantages. Dates are used in high-value products such as candies, syrups, colas, beverages, and chocolates (Younas et al., 2020). They are the most cost-effective source of nutrients for combating food insecurity and rising food demand, especially in developing nations (Ghnimi et al., 2017). Date seeds are nutritious, but the food industry underutilizes them as agricultural refuse. They offer tremendous potential for the development of high-value natural health products. Dates are sacred fruits in all three major religions, but particularly Islam, and must be offered during Ramadan, when the breaking of the fast is both religious and caloric (Alghamdi et al., 2018). Dates alleviate a variety of health issues and

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provide nutritional and pharmacological advantages. Dates provide fast energy from their simple sugars and provide health benefits from their fiber content (Al-Shahib & Marshall, 2002). According to Al-Farsi et al. (2007), the sugar content of dates varies from 35 % to 88 % based on maturation. Fresh or dried, dates are primarily composed of monosaccharides and disaccharides (glucose, fructose, and sucrose), the amounts of which are used to identify them (Al-Hilphy et al., 2023). Dates are an excellent source of vitamins and offer numerous health advantages. Dates are potassium and magnesium abundant. Smaller amounts of calcium, zinc, copper, and selenium are present (Al Hilfi et al., 2019). Minerals are required for the development of bones, dentition, soft tissues, hemoglobin, muscles, and nerve cells (Vayalil, 2012). Underutilized date (Bournow), rich in nutrition and potential for high-value health products, are neglected by the food industry as agricultural waste. This research text highlights the gap in investigating their potential as functional food and nutraceuticals, despite the current focus on plant-based foods and optimal nutrition.

This paper's objective is to identify the optimal conditions for extracting secondary compounds (pH, titratable acidity, vitamin C, total polyphenols, and free amino acids) from date cultivar juice. Consequently, the optimal combination of extraction of minor compounds permits the production of a juice with adequate physicochemical characteristics in minor elements and a close to 1 desirability.

2. Materials and methods

2.1. Biological material

Date (*Phoenix dactylifera* L.) sampling was carried out on the N'Djamena market in collaboration with the Chadian Institute of Agricultural Research for Development (ITRAD). The "Bournow" variety (Fig. 1) was the most suitable because of its availability, good keeping qualities, appreciation by producers and high productivity. This variety accounts for 70 % of production in the Bornou district (Allarangaye et al., 2011).

In this study, the pectinase A, rapidase, was purchased from DSM (Food & Beverage, Netherlands).

2.2. Establishment of mathematical models

Factors affecting the extraction of constituents from dates were used. These factors are: Extraction temperature (x1), extraction time (x2), water/pulp ratio (x3) and pectinase volume (x4). The same 4-factors Centered Composite Design (CCD) were used to run the manipulations. After extraction, the responses considered were pH, free amino nitrogen (FAN) content, total polyphenols and titratable acidity.

For manipulation purposes, the coded variables were transformed into real variables (Wr matrix). The effects of real variables, which are not necessarily expressed in the same units, can be compared using this transformation technique (Desobgo et al., 2010; Mathieu & Phan-tan--luu, 1997):

$$x_j = \frac{U_j - U_j^0}{\Delta U_j} \tag{1}$$

Where: xj : value of coded variable j; Uj = value of natural variable j; Uj0 = value of natural variable j at the center of the domain; ΔUj is called the variation "step".

And;

$$U_j^0 = \frac{U_j^{\max} + U_j^{\min}}{2} \tag{2}$$

Ujmax: maximum value of natural variable j; Ujmin: minimum value of natural variable j.

The model equation was defined and the coefficients of the model equation were predicted after the design of the experiment (the CCD in our case) had been chosen. In response surface method, the model used was generally a full quadratic equation or a diminutive form of this equation. The second-degree model can be written as follows (Mathieu & Phan-tan-luu, 1997):

$$y = \beta_0 + \sum_{j=1}^{k} \beta_j x_j + \sum_{j=1}^{k} \beta_{jj} x_j^2 + \sum \sum_{i<j} \beta_{ij} x_i x_j + \varepsilon \tag{3}$$

With β0 : the constant, ε : the error and βj, βjj and βij were the coefficients of the model.

The following equation provides the matrix expression of the model:

$$y = W\beta + \varepsilon \tag{4}$$

Fig. 1. Date cultivar in its raw state (a,b); date cultivar crushed to different particle sizes (c,d).

Table 1
Experimental matrix and chemical evidence of Bournow date juice.

Run	x1	x2	x3	x4	pH	Vitamin C (mg/L)	Polyphenols (g GA/100 g)	Free Amino Nitrogen (mg/L)
1	0	0	0	0	4.46	78.333	3.72	550.310
2	−1	−1	1	1	4.37	45.000	2.70	440.712
3	1	1	1	1	5.62	88.333	6.72	484.564
4	1	1	−1	−1	4.82	70.000	6.06	605.210
5	−1	−1	−1	−1	4.95	126.667	5.66	524.222
6	0	0	1.607	0	4.63	63.333	3.98	472.649
7	0	0	−1.607	0	4.43	86.667	5.57	617.552
8	1	−1	−1	−1	4.37	116.667	5.45	606.022
9	−1	1	−1	1	4.40	100.000	3.50	503.586
10	0	−1.607	0	0	4.51	71.667	2.81	532.105
11	1.607	0	0	0	4.79	121.667	5.46	566.274
12	1	1	1	−1	5.08	113.333	5.84	497.512
13	0	1.607	0	0	4.66	73.333	4.57	522.800
14	1	1	−1	1	4.64	105.000	5.98	586.278
15	0	0	0	1.607	4.54	70.000	4.11	496.755
16	−1	1	1	−1	4.35	75.000	5.21	435.508
17	0	0	0	0	4.61	66.667	4.57	540.000
18	−1.607	0	0	0	4.44	81.667	3.69	456.660
19	1	−1	1	−1	4.78	103.333	3.52	499.324
20	−1	−1	−1	1	4.37	110.000	4.49	513.354
21	−1	1	−1	−1	4.55	95.000	5.10	515.134
22	0	0	0	0	4.69	63.333	4.06	545.000
23	0	0	0	−1.607	4.61	83.333	5.86	515.891
24	−1	1	1	1	4.39	58.333	3.90	429.944
25	0	0	0	0	4.62	63.333	3.50	540.000
26	1	−1	1	1	4.64	80.000	3.44	487.056
27	−1	−1	1	−1	4.43	95.000	3.84	445.596
28	1	−1	−1	1	4.21	120.000	6.87	587.770

Table 2
Validation criteria for different models based on juice attributes.

Paramètres	R^2	R^2_{adj}	AAD	B_f	A_f
Y_{pH}	0.9352	0.8654	0.029	1.006	1.029
Y_{vitC}	0.9221	0.8381	0.142	0.876	1.170
Y_{Pol}	0.9378	0.8708	0.116	0.925	1.132
Y_{aal}	0.9990	0.9980	0.045	1.034	1.045

It has been translated as follows:

$$\begin{bmatrix} y_1 \\ y_2 \\ \vdots \\ y_n \end{bmatrix} = \begin{bmatrix} 1 & x_{11} & x_{12} & \cdots & x_{1k} \\ 1 & x_{21} & x_{22} & \cdots & x_{2k} \\ \vdots & \vdots & \vdots & \vdots & \vdots \\ 1 & x_{n1} & x_{n2} & \cdots & x_{nk} \end{bmatrix} \times \begin{bmatrix} \beta_0 \\ \beta_1 \\ \vdots \\ \beta_k \end{bmatrix} + \begin{bmatrix} \varepsilon_0 \\ \varepsilon_1 \\ \vdots \\ \varepsilon_k \end{bmatrix} \quad (5)$$

The method of least squares was used to solve the system of equations given above. In this case, the Least Squares Method (LSM) is a multiple regression technique. Here's a summary:

It has been assumed that the random errors in the MMC, are distributed in the same way as the mean zeros and common unknown variances, and that they are independent of each other. The value obtained and the fitted value (y ̂) for the second observation is represented by:

$$\varepsilon_i = y_i - y \quad (6)$$

The residual (error) ε is the evaluation of the corresponding residual (error). This residual takes two aspects into account. The first is a lack of fit, which reflects the potential mismatch between the polynomial model and the real model. The second is experimental errors, which are linked to the random nature of the response.

Evaluations of βj were chosen on the basis that they should minimize

Table 3
ANOVA for the significance of factors used in the extraction of certain constituents from Bournow date juice (*Phoenix dactylifera* L.).

Terms	pH			Vitamin C			Polyphenols			Free amino nitrogen		
	df	MS	P	df	MS	P	df	MS	P	df	MS	P
Const	/	/	**0.000**	/	/	**0.000**	/	/	**0.000**	/	/	**0.000**
x_1	1	0.4007	**0.000**	1	1149.04	**0.002**	1	7.17653	**0.000**	1	24,618.0	**0.000**
x_2	1	0.1835	**0.001**	1	374.15	**0.046**	1	3.97158	**0.000**	1	177.4	**0.000**
x_3	1	0.1319	**0.004**	1	2339.00	**0.000**	1	5.20427	**0.000**	1	43,021.0	**0.000**
x_4	1	0.0304	0.112	1	569.21	**0.018**	1	1.64039	**0.009**	1	750.3	**0.000**
x_1^2	1	0.0079	0.398	1	2812.99	**0.000**	1	0.92178	**0.038**	1	2175.1	**0.000**
x_2^2	1	0.0022	0.654	1	138.94	0.202	1	0.08501	0.496	1	577.4	**0.000**
x_3^2	1	0.0009	0.768	1	234.79	0.104	1	1.54493	**0.011**	1	0.9	0.703
x_4^2	1	0.0010	0.753	1	312.58	0.065	1	2.37146	**0.003**	1	2906.6	**0.000**
x_1x_2	1	0.4192	**0.000**	1	1.56	0.889	1	1.15562	**0.023**	1	68.5	**0.004**
x_1x_3	1	0.4935	**0.000**	1	1083.51	**0.002**	1	0.18922	0.315	1	788.0	**0.000**
x_1x_4	1	0.0410	0.069	1	291.84	0.073	1	3.38560	**0.001**	1	54.5	**0.009**
x_2x_3	1	0.0315	0.106	1	826.56	**0.006**	1	6.25000	**0.000**	1	1.0	0.682
x_2x_4	1	0.0885	**0.012**	1	451.56	**0.031**	1	0.08122	0.506	1	0.5	0.780
x_3x_4	1	0.1314	**0.004**	1	1254.34	**0.001**	1	0.00303	0.897	1	35.8	**0.026**
Error	13	0.0104	/	13	76.98	/	13	0.17337	/	13	5.7	/
Lack of fit	10	0.0108	0.510	10	84.86	0.369	10	0.16026	0.688	10	0.2	1.000
Pure error	3	0.0093	/	3	50.69	/	3	0.21709	/	3	24.2	/

Const: constant; df: degree of freedom; MS: mean square; P: probability.

the sum of squared residuals, also known as the sum of squared errors and has been noted SSE.

$$SSE = \sum_{i=1}^{n} \varepsilon_i^2 = \sum (y_i - y)^2 \quad (7)$$

Residuals were evaluated using the following equation:

$$\varepsilon = y - W\beta \quad (8)$$

And SSE used the following expression:

$$SSE = \varepsilon^T \varepsilon = (y - W\beta)^T (y - W\beta) \quad (9)$$

Dividing the SSE as a function of β, a vector of partial derivatives was found as follows:

$$\frac{\partial}{\partial \beta}(SSE) = -2W^T(y - W\beta) \quad (10)$$

By setting this derivative equal to 0, it was obtained:

$$y = W\beta \quad (11)$$

It was possible to solve this system of equations directly to obtain the β coefficients:

$$W^T W\beta = W^T y \quad (12)$$

After that, the formal solution of these equations was given by:

$$\beta = \left(W^T W\right)^{-1} W^T y = CM^T y \quad (13)$$

With:

$$C = \left(W^T W\right)^{-1} \quad (14)$$

With C, the square matrix.

The model equation took its final form thanks to the values of the coefficients. The Minitab 21 program was used to perform matrix operations to evaluate the β vector. OriginLab 2022 was used to plot the graphs. It is important to note that the experimenter, who is aware of the stakes and risks of the study, determines the final model.

2.3. Validation of mathematical models

The coefficient of determination R^2 represented the goodness of fit of the second-degree equations. The models were validated using two techniques. The first strategy was the Absolute Average Deviation analysis (AAD) (Baş & Boyaci, 2007), while the second strategy used the accuracy factor and the polarized factor.

Method 1: The aim of the statistical analysis was to give the model's representativeness scientific legitimacy. The model equation was used to easily calculate the predicted response after obtaining the regression coefficients. Since system behavior is generally unknown, it was necessary to check whether the models corresponded correctly to the experimental data. Several methods are used to determine whether the model is adequate. Residual analysis measuring the residuals, the sum of the prediction errors of the residuals and the lack-of-fit test are some of these methods. The coefficient of determination (R^2) has generally been used to explain the predictive potential of the model as a whole. It should be noted that the coefficient of determination (R^2) is not the only measure of model accuracy. It is a measure of the amount of reduced response variability that has been achieved using the model's repressor variables. However, a high R^2 value does not mean that the regression model is good. Regardless of whether the additional variable is statistically significant or not, adding an additional variable to the model will always increase R^2. Consequently, models with large R^2 values may provide poor predictions for new observations or evaluations of the mean response. If we compare experimental results with model results, we should obtain a straight line with a 45° angle passing through the origin. However, it is possible to obtain such a line using the formula [y=ax+b]. Absolute average deviation analysis (AAD), a direct method for describing deviations, was used to eliminate these types of errors.

The following equations were used to calculate the coefficient of determination R^2 and AAD:

$$R^2 = \frac{\sum_{i=1}^{n} \left(y_{i,cal} - \overline{y}\right)^2}{\sum_{i=1}^{n} \left(y_{i,\exp} - \overline{y}\right)^2} \quad (15)$$

$$AAD = \frac{\left[\sum_{i=1}^{n} \left(\frac{\left|y_{i,\exp} - y_{i,cal}\right|}{y_{i,\exp}}\right)\right]}{n} \quad (16)$$

Where n is the number of experiments performed, $\bar{y}$ is the mean of the experimental responses and yi,exp and yi,cal are the experimental and calculated responses respectively.

To check the accuracy of the model, the combined evaluation of R^2 and AAD values should be more effective. The AAD between predicted and observed data should be as small as possible and R^2 should be close to 1 (Baş & Boyaci, 2007). The model equation defines the true behavior of the system and can be used for interpolation in the experimental domain, depending on the acceptable values of R^2 and AAD. It is important to consider the issue of extrapolation outside the area where the initial observations were made. It is quite possible that a model that works well with the initial data will no longer work with the outside data.

Method 2: Observed and theoretical values were compared to assess model validation. Equations for the polarized factor, Bf, and the polarized accuracy factor, Af1, were provided (Ross, 1996):

$$B_f = 10^{\frac{1}{n}\sum_{i=1}^{n} \log\left(\frac{y_{i,cal}}{y_{i,\exp}}\right)} \quad (17)$$

$$A_{f1} = 10^{\frac{1}{n}\sum_{i=1}^{n} \left|\log\left(\frac{y_{i,cal}}{y_{i,\exp}}\right)\right|} \quad (18)$$

Yi,theo, response obtained using the model; Yi,exp, response obtained by experiment; n, number of trials.

In the perfect predictive model, Af1 = Bf = 1. The acceptable predictive model is defined as 0.75 < Bf or Af1 < 1.25 (Dalgaard & Jørgensen, 1998).

2.4. Optimization

Optimization was carried out using a multi-response approach that included maximization of pH, vitamin C, polyphenols and titratable acidity as specifications. Minitab 21.3.1 was used to find the best combination meeting all specifications. Response optimization refers to a set of variable parameters that work together to optimize a single response or a set of responses. This method is useful for determining the effect of several variables on a response. Minitab assigns an individual desirability to each response and determines it according to the importance attributed to it. These values were added together to determine the overall desirability of the multiple-response system. When the composite desirability reached its maximum, an optimal solution was found. Individual and composite desirability were used to determine how well a combination of variables met the objectives of the response. Individual desirability measures the extent to which parameters optimize a single response, while composite desirability measures the extent to which parameters optimize a group of responses. The desirability scale runs from 0 to 1. A value of 1 would be ideal, while a value of 0 would indicate that one or more responses are outside the acceptable range. The weighted geometric mean of the individual desirability of the different responses is the composite desirability. Minitab determined the optimal parameters for the input variables by maximizing the composite desirability.

2.5. Date juice extraction

Solid-liquid extraction was carried out here. Extractions were carried out in accordance with the parameters of the four-factor centered composite design (CCD). The date juice extraction process was carried out according to Kadlezir et al. (2023).

Dates were sorted, washed and pitted. They were then crushed to increase the exchange surface and facilitate juice extraction. Extraction was performed by immersing beakers containing crushed dates and water in a water bath, using a centered composite design with the variables temperature, time, water/pulp ratio, and pectinase (enzyme) volume ranging from 25 °C to 95 °C, 10 min to 120 min, 2 to 5, and 0 mL to 0.5 mL, respectively. This yielded 28 assays, each of which was filtered through a filter cloth, then pasteurized for 15 s at 72 ± 2 °C (Burapalit, 2019). Pasteurized juices were stored in the refrigerator at 4 °C.

2.6. Determination of free amino acid content in juices

The ninhydrin technique was used to determine the concentration of free amino acids in extracts by colorimetry (EBC-Analysis-Committee, 1998).

To obtain a 1/100 dilution, 99 ml distilled water was mixed with 1 ml extract. The sample was diluted and separated into three test tubes. Each test tube received 1 ml of color reagent (100 g/L Na_2HPO_4, 60 g/L KH_2PO_4, 5 g/L ninhydrin and 3 g/L fructose). Tubes were immersed in boiling water for 16 min. They were then cooled in a water bath to 20–25 °C. Each received 5 ml dilution solution (2 g KIO_3, 1 L H_2O/ Ethanol 96 % (600:400, v/v)). A Jenway 6405 UV/Visible spectrophotometer was used to measure absorbance at 570 nm (Jenway Ltd Felsted, Dunmow, Essex CM6 3LB, UK). The results obtained were compared with those of the control and the standard. To create the blank, 2 ml of distilled water was used in place of the diluted extract. The standard was 2 ml glycine (10.72 mg/L) instead of the diluted extract. The following relationship was used to determine the proportion of free amino acids:

$$FAN(mg/L) = \frac{2 \times A_1}{A_2} \times d \quad (19)$$

FAN: free amino nitrogen (mg/L); A1: absorbance of test solution at 570 nm; A2: mean absorbance of standard solution; d: dilution factor.

2.7. pH measurement of date juice

The pH meter electrode (Jual HANNA HI9813–6 Portable pH/ EC/ TDS Meter Harga Murah) was dipped into the beaker containing 20 ml of sample at 25 °C; the pH value was read. The operation was repeated three times.

2.8. Determination of vitamin C content in date juice

An Erlenmeyer flask was filled with a volume V' equal to 5 ml of sample measured with a graduated pipette, followed by 5 ml of the metaphosphoric acid-acetic acid solution (v/v) and 10 ml of distilled water. A second empty Erlenmeyer flask was filled with standard ascorbic acid solution (250 mg/L). Vitamin C (Vit C) was titrated with dichlorophenolindophenol (DCPIP) solution (8.61×10–3 mol/L) for 30 s until a pink tint persisted. The procedure was repeated three times. The following formula was used to calculate vitamin C content.

$$VitC(mg/L) = \frac{[DCPIP] \times V \times M}{V_0} \quad (20)$$

M: molar mass of vitamin C (176 g/mol); V: volume of DCPIP (ml); Vo: volume of sample (mL)

2.9. Determination of total phenolic content in date juice

Polyphenols in date juice were measured using the Folin-Ciocalteu reagent (Matloob & Balakita, 2016), which produces a blue phosphotungstic-phosphomolybdenum complex. Two milliliters distilled water and 1.0 mL Folin-Ciocalteu reagent (diluted 1:10) were added to 100 µL sample extract. After allowing the mixture to stand for 5 min, 0.75 mL Na2CO3 solution (60 g/L) was added. After 90 min, absorbance was measured at 765 nm using a UV–visible spectrophotometer (Jenway Ltd Felstd, Dunmow, Essex CM6 3LB, UK) against water as a blank. For three replicates, total phenol concentration was expressed as g gallic acid equivalent (GAE) per 100 g fresh sample.

3. Results and discussion

3.1. Modeling pH, vitamin C, free amino acids and polyphenols

The influence of process parameters (temperature, time, enzyme ratio and volume) on the extraction of some responses (pH, vitamin C, free amino acids and polyphenols) from date juice was determined. The results are shown in Table 1.

CCD models relate singular factors, interactions and quadratic effects to response variables. These models consisted of:

$$Y_{pH} = 4.5771 + 0.0856x_1 + 0.058x_2 + 0.0491x_3 - 0.0236x_4 + 0.0095x_1^2 + 0.005x_2^2 - 0.0033x_3^2 + 0.0035x_4^2 + 0.06268x_1x_2 + 0.06801x_1x_3 + 0.0196x_1x_4 + 0.01718x_2x_3 + 0.0288x_2x_4 + 0.03509x_3x_4 \quad (21)$$

$$Y_{vitC} = 66.36 + 4.59x_1 - 2.62x_2 - 6.54x_3 - 3.23x_4 + 5.623x_1^2 + 1.25x_2^2 + 1.625x_3^2 + 1.874x_4^2 + 0.121x_1x_2 + 3.187x_1x_3 + 1.654x_1x_4 + 2.783x_2x_3 + 2.057x_2x_4 - 3.429x_3x_4 \quad (22)$$

$$Y_{Pol} = 3.935 + 0.3624x_1 + 0.2696x_2 - 0.3086x_3 - 0.1732x_4 + 0.1018x_1^2 - 0.0309x_2^2 + 0.1318x_3^2 + 0.1633x_4^2 + 0.1041x_1x_2 - 0.0421x_1x_3 + 0.1781x_1x_4 + 0.242x_2x_3 - 0.0276x_2x_4 - 0.0053x_3x_4 \quad (23)$$

$$Y_{aal} = 544.08 + 21.223x_1 - 1.801x_2 - 28.055x_3 - 3.705x_4 - 4.945x_1^2 - 2.548x_2^2 + 0.099x_3^2 - 5.716x_4^2 + 0.801x_1x_2 - 2.718x_1x_3 - 0.715x_1x_4 - 0.097x_2x_3 - 0,066x_2x_4 + 0,579x_3x_4 \quad (24)$$

With, Y_{pH}: pH; Y_{vitC}: Vitamin C; Y_{Pol}: Polyphenols; Y_{aal}: Free amino acids; x1: Temperature; x2: Time; x3: Water/pulp ratio; x4: Pectinase volume.

These second-order models are useful if some of the input variables are precise. Table 2 shows that all the models are valid and allow a thorough evaluation of the components.

The ANOVA in Table 3 only takes into account variables with a probability of less than 0.05. These are therefore the only relevant

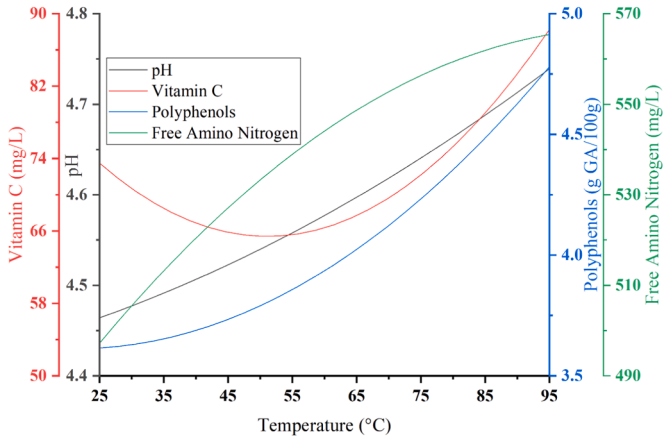

Fig. 2. Changes in vitamin C, pH, polyphenols and free amino acids as a function of temperature (time, water/pulp ratio and pectinase volume fixed at 65 min, 3.5 and 0.25 mL respectively).

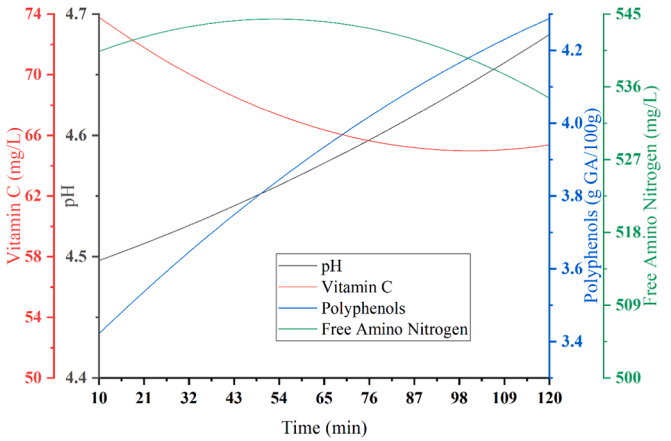

Fig. 3. Changes in vitamin C, pH, polyphenols and free amino acids as a function of time (temperature, water/pulp ratio and pectinase volume fixed at 60 °C, 3.5 and 0.25 mL respectively).

elements. These are therefore the only relevant elements.

3.1.1. *Impact of singular factors on responses*

3.1.1.1. Impact of temperature. The factor corresponding to extraction temperature, as a singular factor (extraction time (x2), water/pulp ratio (x3), and enzyme volume (x4) being fixed at their central values of 65 min; 3.5 and 0.25 mL respectively), has a significant impact on vitamin C, pH, polyphenols and free amino acids (Table 3).

All these responses increase with increasing temperature. Indeed, pH, vitamin C, polyphenols and free amino acids increase significantly (Table 3) from 4.46; 73.5 mg/L; 3.61 g GA/100 g and 497.2 mg/L respectively at 25 °C, to values of 4.73; 88.26 mg/L; 4.78 g GA/100 g and 565.42 mg/L at 95 °C (Fig. 2).

In the case of vitamin C and the free amino acids present in dates (Ashraf & Hamidi-Esfahani, 2011), the temperature would weaken the pulp, allowing greater release of these two components into the juice. In fact, for vitamin C, the extraction kinetics would be superior to those of denaturation.

For pH, this could be explained by the fact that potassium, one of the most abundant mineral compounds in dates (Ibrahim et al., 2001; Mohamed, 2000), could increase the pH. Secondly, high-temperature extraction would rapidly deactivate pectinase, resulting in an extract with a higher pH. Because of the loss of volatile acids and carbon dioxide due to increased temperature, the acidity of the juice would decrease, resulting in a higher pH.

The higher temperature would enable polyphenols to be extracted more efficiently. In this case, the rate of polyphenol extraction would be higher than the rate of degradation. This may be explained by the presence of hydrolyzable tannins, which are thermodegradable. In reality, hydrolyzable tannins were degraded at high temperature (100 °C), resulting in an increase in non-tannin content. Al-Farsi et al. (2005) reported an increase in the total phenolic content of sun-dried dates due to temperature-induced tannin degradation during the drying process. In addition, Jeong et al. (2004) reported a significantly higher concentration of polyphenols in heated citrus peels than in unheated peels.

3.1.1.2. Impact of extraction time. The factor corresponding to extraction time, as a singular factor (extraction temperature (x1), water/pulp ratio (x3), and enzyme volume (x4) being fixed at their central values of 60 °C; 3.5 and 0.25 mL respectively), has a significant impact on vitamin C, pH, polyphenols and free amino acids (Table 3). pH and polyphenol content increase significantly (Table 3) with increasing extraction time. On the other hand, vitamin C and free amino acids decrease significantly (Table 3) with increasing extraction time (Fig. 3).

Indeed, for pH and polyphenols, we start from 4.49 to 3.42 g GA/100 g respectively for a time of 10 min, to increase to 4.68 and 4.28 g GA/100 g respectively for a time of 120 min Fig. 3. In addition to the above-mentioned reasons concerning temperature (here it is fixed at 60 °C), a longer extraction time would allow greater accumulation in the juice, justifying the increase. It's important to note that different types of polyphenols have different effects. According to Kaack and Austed (1998), anthocyanidins are protected from oxidative destruction by vitamin C, which acts as an inhibitor. This protection is provided by vitamin C. According to Aka et al. (2013), the protective effect described is most likely due to the decrease in the oxidized form of the polyphenol caused by ascorbic acid. This form of the polyphenol is then oxidized, as has been discovered for chlorogenic acid and epicatechin.

The decrease in vitamin C and free amino acids can be seen in Fig. 3. For vitamin C and free amino acids, we start from 73.79 mg/100 g and 540.39 mg/L for a time of 10 min, and decrease to 65.37 mg/100 g and 534.61 mg/L respectively, for a time of 120 min Fig. 3.

The main route of vitamin C degradation in aqueous liquid systems involves the oxidation of ascorbic acid to dehydroascorbic acid, which rapidly degrades to 2,3-diketogulonic acid (Washko et al., 1992). The hydrolysis of dehydroascorbic acid results in the loss of the vitamin property of the molecule. Ascorbic acid degradation increases with increasing water activity or moisture content (Lee & Labuza, 1975). The reaction of ascorbic acid with its oxidized form, dehydroascorbic acid, and subsequent hydrolysis to 2,3-diketogulonic acid occur simultaneously in water in the absence of oxidizing or reducing compounds (Serpen & Gökmen, 2007).

The carbonyl groups of proteins, peptides and amino acids condense with the carbonyl groups of sugars to trigger Maillard reactions, forming Schiff bases that can be rearranged into Amadori or Heyns products (Hellwig & Henle, 2014). These macromolecules are decomposed or modified to generate reactive dicarbonyl species, which can react readily with other nucleophiles such as amines, guanidines and thiols. By reacting with free amino acids to produce imines, these intermediates can undergo Strecker degradation, leading to the formation of Strecker aldehydes (Lund & Ray, 2017). This would justify the decrease in free amino acid content over time.

3.1.1.3. Impact of the water/pulp ratio. The factor that corresponds to the water/pulp ratio, as a singular factor (with extraction temperature (x1), extraction time (x2), and enzyme volume (x4) fixed at their central values of 60 °C; 65 min; and 0.25 mL respectively), has a significant impact on vitamin C, pH, polyphenols and free amino acids (Table 3).

Vitamin C, polyphenols and free amino acids decreased with increasing water/pulp ratio, while pH increased with increasing water/

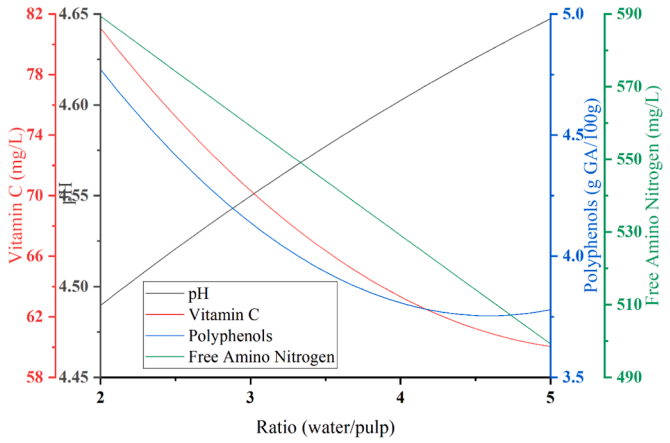

Fig. 4. Changes in vitamin C, pH, polyphenols and free amino acids as a function of water/pulp ratio (temperature, time and pectinase volume fixed at 60 °C, 65 min and 0.25 mL respectively).

pulp ratio (Fig. 4).

In fact, vitamin C, polyphenols and free amino acid contents go from 81.06 mg/100 g; 4.77 g GA/100 g and 589.42 mg/L respectively for a water/pulp ratio of 2 (Fig. 4), to 60.04 mg/100 g; 3.77 g GA/100 g and 499.25 mg/L respectively for a water/pulp ratio of 5 (Fig. 4). On the other hand, pH rose from 4.48 for a water/pulp ratio of 2 to 4.64 for a water/pulp ratio of 5. All these observations can be explained simply by the dilution effect, which reduces the concentration of vitamin C, polyphenols and free amino acids, while increasing the pH.

3.1.1.4. Impact of enzyme volume. The factor corresponding to enzyme volume, as a singular factor (extraction temperature (x1), extraction time (x2), and water/pulp ratio (x3) being fixed at their central values of 60 °C; 65 min; and 3.5 respectively), has a significant impact on vitamin C, pH, polyphenols and free amino acids (Table 3).

Vitamin C and polyphenol contents undergo a significant decrease, followed by a non-significant increase with increasing enzyme volume (Fig. 5), while free amino acid content undergoes a non-significant increase on the one hand, followed by a significant decrease with increasing enzyme volume (Fig. 5).

In fact, for vitamin C, we go from 76.39 mg/100 g without the addition of pectinase, followed by a decrease to a minimum value of 64.96 mg/100 g at a pectinase volume of 0.38 mL and then a non-significant increase to 66 mg/100 g at a pectinase volume of 0.5 mL (Fig. 5). The polyphenol content ranges from 4.63 g GA/100 g with no pectinase added, to a minimum value of 3.89 g GA/100 g with 0.33 mL pectinase added, followed by a non-significant increase to 4.07 g GA/100 g with 0.5 mL pectinase added (Fig. 5). The free amino acid content starts at 535.27 mg/L without the addition of pectinase, followed by a non-significant increase to 544.68 mg/L with the addition of 0.2 mL pectinase, and then a significant decrease to 523.36 mg/L with the addition of 0.5 mL pectinase (Fig. 5).

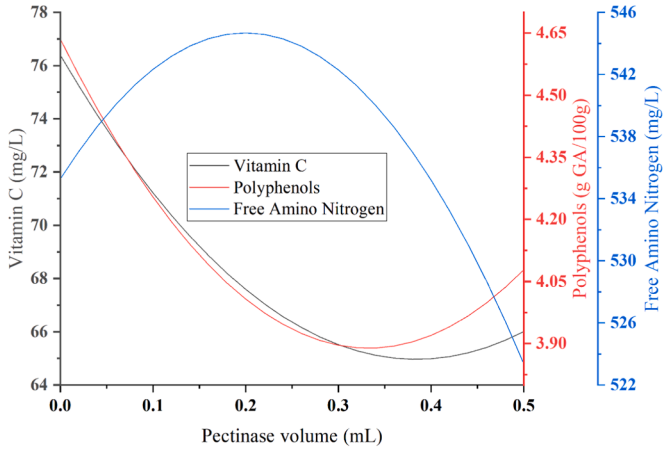

Fig. 5. Changes in vitamin C, polyphenols and free amino acids as a function of pectinase volume (temperature, time and water/pulp ratio set at 60 °C, 65 min and 3.5 respectively).

The reason for the drop in polyphenols and amino acids with increasing pectinase volume is hydrolysis of the date matrix, releasing these two constituents, which are involved in the disorder formation process. The most common cause of cloudiness in beverages is the interaction between proteins and polyphenols. Proline is present in proteins that bind polyphenols, and the more proline present, the greater the disorder-forming activity. Proline-rich proteins with high binding affinity to polyphenols via hydrogen bonding and hydrophobic interactions have been shown to be responsible for haze formation (Schulte et al., 2016). At least two sites of haze-causing polyphenols can bind to proteins, enabling them to crosslink proteins and produce insoluble, light-scattering particles. At least initially, the interaction between protein and polyphenol is non-covalent and reversible. The ratio of haze-active polyphenols (HA) to HA protein affects both haze particle size and haze intensity (Schulte et al., 2016).

3.1.2. Impact of interactions on responses

The x1×2 interaction (temperature/extraction time) contributes to a significant increase in pH, polyphenols and free amino acids (Table 3). Indeed, for pH, polyphenols and free amino acids, this increase is observed with a simultaneous increase in extraction temperature and time (Fig. 6).

Increased temperature and extraction time contribute to pulp embrittlement and greater release of minerals such as potassium and calcium (Ibrahim et al., 2001; Mohamed, 2000), which in aqueous solution form bases that raise the pH.

The increase in polyphenols with a simultaneous increase in temperature and extraction time can be explained by the fact that polyphenol extraction is better at higher temperatures (Jeong et al., 2004), and with longer extraction times there is an accumulation of these polyphenols in the date juice, resulting in an increase.

The increase in free amino acid content with increasing temperature and extraction time is explained by the fact that these compounds are extracted much more quickly, so that the extraction kinetics are greater than those for the formation of Maillard reaction compounds (Hellwig & Henle, 2014) and haze.

The x1×3 (temperature/pulp water ratio) interaction contributes to a significant increase in pH and vitamin C (Table 3), while it contributes to a significant decrease in free amino acid content (Table 3).

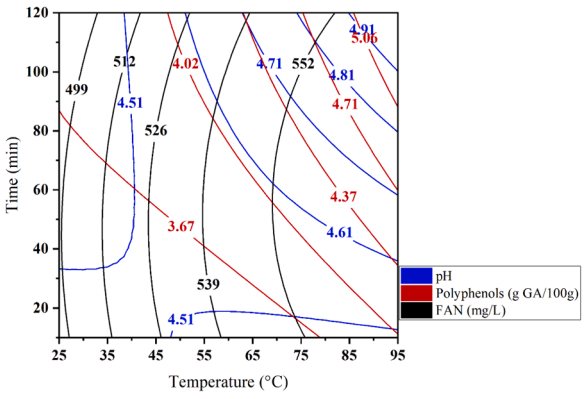

Fig. 6. Changes in pH, polyphenols and free amino acids as a function of temperature/time interaction. The pulp/water ratio and pectinase volume are fixed at 3.5 and 0.25 mL respectively.

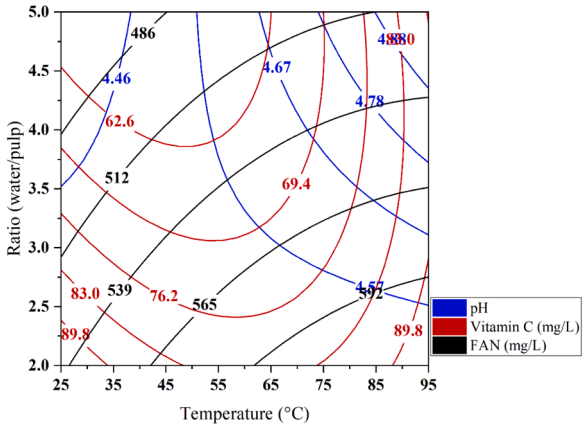

Fig. 7. Changes in pH, vitamin C and free amino acids as a function of temperature/water/pulp ratio interaction. The time and volume of pectinase are fixed at 65 min and 0.25 mL respectively.

The increase in the pH is achieved through a simultaneous increase in temperature and water/pulp ratio (Fig. 7). The water/pulp ratio, which favors dilution of the juice, contributes to raising its pH, while the increase in temperature contributes to better extraction of minerals, such as potassium, which forms bases in solution.

The increase in vitamin C is observed by reducing the extraction temperature simultaneously with an increase in the pulp water ratio (Fig. 7). This would lead to an accumulation of extracted vitamin C, and with the temperature reduced, vitamin C would not be destroyed by the latter.

The drop in free amino acids is observed when the temperature is reduced simultaneously with an increase in the water/pulp ratio (Fig. 7). This decrease is explained more by the dilution effect than by an increase in the ratio.

The x1×4 interaction contributes to a significant increase in polyphenols and a simultaneous significant decrease in free amino acids (Table 3).

In fact, the increase in polyphenols is obtained via a simultaneous increase in temperature and pectinase volume (Fig. 8). This increase is explained by the fact that the extraction temperature weakens the pulp and, combined with the volume of pectinase, hydrolyzes the pectins, thus enabling greater extraction of polyphenols.

The decrease in free amino acid content can be observed by lowering the extraction temperature simultaneously with an increase in pectinase volume (Fig. 8). In fact, this effect can be explained by the fact that not only would the lower temperature not allow better extraction, it would also not allow hydrolysis of the pulp by pectinase.

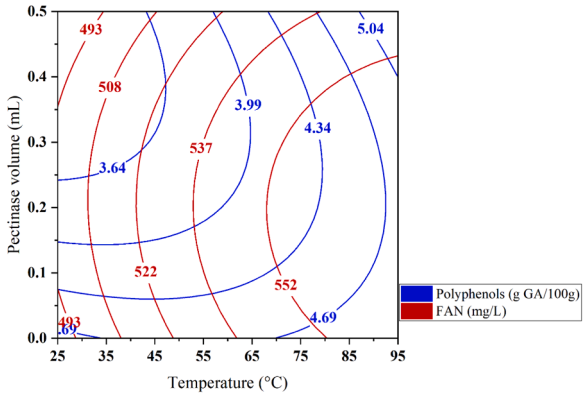

Fig. 8. Changes in polyphenols and free amino acids as a function of pectinase temperature/volume interaction. The pulp/water ratio and time are set at 3.5 and 65 min respectively.

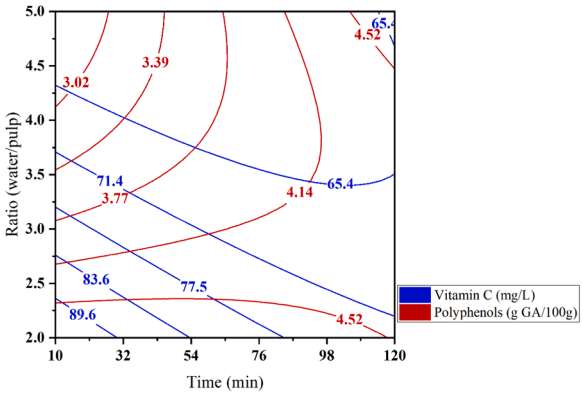

Fig. 9. Evolution of vitamin C and polyphenols as a function of time/water/pulp ratio interaction. Temperature and pectinase volume set at 60 °C and 0.25 mL respectively.

The x2×3 interaction contributes to a significant increase in vitamin C and polyphenols (Table 3). The increase in vitamin C content is observed via a simultaneous decrease in time and water/pulp ratio (Fig. 9). This could be explained by the fact that, on the one hand, a decrease in the water/pulp ratio leads to an increase in concentration, and a reduction in time contributes to a decrease in the effect of temperature on vitamin C degradation, and thus to an increase in vitamin C content.

The increase in polyphenol content is observed with an increase in extraction time simultaneously with a decrease in the water/pulp ratio (Fig. 9). Indeed, increasing the extraction time contributes to an accumulation of polyphenols in the juice, and concomitantly a decrease in the water/pulp ratio reduces the effect of dilution and, in turn, creates an increase in concentration.

The x2×4 interaction contributes to a significant increase in pH and vitamin C content (Table 3). This increase in pH is achieved by increasing extraction time and decreasing pectinase volume (Fig. 10). The increase in pH would be due simultaneously to an accumulation of ions responsible for the basic character (such as potassium, sodium and others), while the reduction in pectinase volume would not favor hydrolysis of the pulp and would therefore reduce extraction of the organic acids present in the date.

The increase in vitamin C can be observed via a simultaneous reduction in extraction time and pectinase volume. In fact, the thermal degradation of vitamin C is slowed down as the time of heat application is reduced. At the same time, according to studies, the stability of vitamin C depends largely on the temperatures applied and the presence of oxygen, and its degradation is largely due to oxidation reactions

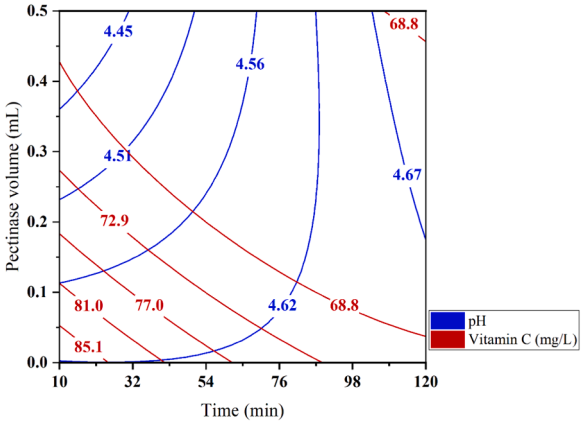

Fig. 10. Evolution of pH and vitamin C as a function of pectinase time/volume interaction. Temperature and water/pulp ratio set at 60 °C and 3.5 respectively.

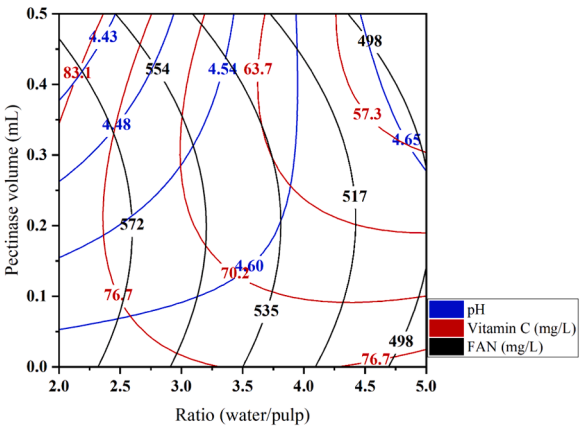

Fig. 11. Changes in pH, vitamin C and free amino acids as a function of the water/pulp ratio/pectinase volume interaction. Temperature and time set at 60 °C and 65 min respectively.

occurring during the adiabatic heating phase (Oey et al., 2008). The applied temperature inactivates endogenous enzymes, notably ascorbic acid oxidase, which can cause vitamin C degradation (Leong & Oey, 2012; Munyaka et al., 2010).

The x3×4 interaction contributes to an increase in pH, free amino acid content and a decrease in vitamin C content (Table 3).

The increase in pH is achieved by increasing the water/pulp ratio simultaneously with a decrease in pectinase volume. Indeed, the increase in the water/pulp ratio would contribute to this pH increase via the dilution effect, and the simultaneous decrease in pectinase volume would contribute to the reduction of the hydrolytic effect of this enzyme, which could have enabled the release of organic acids (Fig. 11).

The increase in free amino acid content would occur via a reduction in the water/pulp ratio and simultaneously with the increase in pectinase volume (Fig. 11). In fact, reducing the water/pulp ratio would bring about a concentration effect, while at the same time increasing the pectinase volume would contribute to better pulp hydrolysis and, in turn, an increase in free amino acid content.

The decrease in vitamin C content can be seen in the simultaneous increase in the water/pulp ratio and enzyme volume (Fig. 11). Although the increase in pectinase volume would contribute to an increase in vitamin C content through hydrolysis of the pulp, the gradient of the water/pulp ratio would be greater, and consequently lead to a decrease in the vitamin C content of the juice.

3.2. Optimization of minor constituents (pH, vitamin C, polyphenols and FAN)

Multi-response optimization was used to maximize the minor physicochemical properties of date juice. To this end, vitamin C, polyphenols and free amino acids (FAN) were all taken into account. pH, on the other hand, was left unchanged. The final compromise for Minitab 21.3 optimization was as follows: extraction at 95 °C for 10 min, water/pulp ratio 2 and pectinase volume 0.5 mL. Using this combination, free amino acids of 587.88 mg/L, polyphenols of 6.25 g GA/100 g, vitamins C of 116.5 mg/100 g and a pH of 4.13 were obtained. Individual desirabilities for free amino acids, polyphenols and vitamin C were 0.841, 0.852 and 0.875 respectively. Composite desirability was 0.856. In the study, composite desirability (0.856) was close to 1, suggesting that the parameters appeared to have a positive impact on all responses. However, the individual desirability data showed that the parameters were more effective at maximizing vitamin C (0.875) than polyphenols (0.852) and FANs (0.841).

4. Conclusion

In this study, the response surface methodology was used to analyze the characteristics of extracted date juices. The juice extraction method affected the pH, vitamin C, free amino acids, and polyphenols in the juice. The extraction process was optimized to maximize desired responses and improve the formulation. The results showed that the extraction method effectively extracted both major and minor compounds from the date fruit. This suggests that the parameters used in the study were successful in achieving favorable outcomes. The study had a high composite desirability value of 0.8560, indicating that the response surface methodology could be a suitable approach for date juice extraction when optimal conditions are established. The findings of this study have broader implications, as the production of date juice could contribute to the development of functional food products with added health benefits. Date juice has the potential to serve as a prebiotic for fermentation using probiotics, highlighting its nutritional value.

CRediT authorship contribution statement

Fiacre Kadlezir: Conceptualization, Data curation, Investigation, Writing – original draft. **Ahmed Mohammed Mohagir:** Validation, Visualization, Writing – review & editing. **Steve Carly Zangué Desobgo:** Conceptualization, Data curation, Formal analysis, Methodology, Software, Supervision, Validation, Visualization, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no conflict of interest

Data availability

Data will be made available on request.

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