J. Food. Stab (2023) 6 (2): 66-83 Journal homepage: www.foodstability.com

Received: 28/01/2023/ Revised: 20/02/2023/ Accepted: 22/02/2023 / Available online: 18/03/2023

DOI: 10.36400/J.Food.Stab.6.2.2023-005

Journal of Food Stability

https://www.ajol.info/index.php/jfs

ORIGINAL ARTICLE

Application of response surface methodology in date (*Phoenix dactylifera* L.) juice extraction: Effect of process parameters on Brix, color and sugar/acid ratio

[1]Kadlezir Fiacre / [2]Mohagir Ahmed Mohammed / *[3]Desobgo Zangué Steve Carly/

Authors' Affiliation

[1]University of Ndjamena, Doctoral School of Technical and Environmental Sciences. Doctoral training in Physics and Engineering Sciences

[2]Department of Chemistry, Faculty of Pure and Applied Sciences, University of Ndjamena, BOX: 1027. Ndjamena, Chad

[3]Department of Food Process & Quality Control, Bioprocess Laboratory, University Institute of Technology (UIT), University of Ngaoundere, P.O. Box 455, Adamaoua, Cameroon

Corresponding author

Desobgo Z.S.C.

Email:

desobgo.zangue@gmail.com

Abstract

A technique called "extraction" was used to get juice from the "*Bournow*" date cultivar so that it could be used in the beverage industry. To reach this goal, dates were first studied to find out what their physical traits were. Then, the four-factor centered composite design was used to find out how temperature, time, volume to mass ratio, and enzyme volume affected Brix, color, and sugar/acid ratio. All second-degree multivariate polynomial models with interactions were obtained and validated. To maximize the responses, a multi-response optimization was done. The physical characterization permitted to get values below the criteria for classification. This enabled to say that the *Bournow* dates have bad qualities, which was why they needed to be valued. From center composite design the following responses ranges were obtained: Brix, between 13.2 and 25.9 °B; color, between 125.5 and 206 ASBC; sugar/acid ratio, between 6.24 and 46.90. It was observed that the selected factors have different effects on the Brix, color, and sugar/acid ratio responses, increasing or decreasing them in a significant way in single, quadratic, and interaction contributions. The multi-response optimization, whose goal was to get the maximum of all responses in order to make nutrient-rich juice, came up with the following compromise: temperature, 95 °C; time, 10 min; ratio water/pulp, 2:1, and pectinase volume, 0 mL. The simulated optimum values resulted in the following respective maximums: Brix, 21.89 °B; sugar/acid ratio, 13.99 and color, 197.49 ASBC.

Keywords: *Phoenix dactylifera* L., Juice, Extraction, Optimization

1. Introduction

Consumer demand for functional foods has resulted in the processing of lesser-known fruits such as desert dates, which are known for their medicinal properties rather than their culinary value. These products, such as *Phoenix dactylifera* L., contain antioxidants, vitamins, and other nutritive and functional compounds (Chirinos *et al.,* 2013). It is important for human and animal nutrition because of its productivity, nutritional quality of its fruits, and ability to adapt to Saharan regions. Chad is Africa's eighth-largest producer, with an annual output of about 21,134.59 tons (FAOSTAT, 2022). The cultivar most widely used in almost all production areas in Sahara is "*Bournow*" due to its high productivity, ease of storage, the quality of its fruits for marketing (dry date), and climate adaptation. Date palm products and byproducts are widely used from the Sahara to the Sahel (Mahmoud *et al.,* 2022). The date can be used as a raw material in the development of many products including liquid sugar, date pastes, juices, syrups, soft drinks, confectionery, alcohol, vinegar (Estanove, 1990). It is primarily the dry dates, soft dates, and palms that are used in human or animal food, either locally or through traders throughout

almost the entire Chad (Mahmoud *et al.,* 2022). Date juice could be an excellent substitute. That juice is a beverage with a unique flavor and a lot of nutritional benefits (Mahmoud *et al.,* 2022). Heat treatment causes several biological, physical, and chemical changes in foods, resulting in sensory, textural, and nutritional changes. Heating has been shown to improve food safety by destroying or inhibiting microorganisms and inactivating anti-nutritional factors. It is also involved in the formation of desired compounds such as flavor compounds, antioxidants, and coloring agents. Furthermore, heating improves food digestibility and nutrient bioavailability (Echegaray *et al.,* 2021). Chemical constituents of food products must be affected by processing methods. Understanding the effect of processing on functional components is critical for preserving or improving the original activity of dates during date juice preparation (Burapalit, 2019).

The food and beverage industry still views popular fruits as the only raw materials for juice production; however, the discovery of previously unknown nutritional facts about lesser-known fruits (Amadou & Le, 2017) such as the *Bournow* date presents an alternative source of juice raw material. The study's overarching goal is to contribute to the valorization of the date sector by producing an adequate juice. This will entail specifically developing mathematical models that will allow the prediction of the profile of some physicochemical characteristics of the "Bournow" date extract as well as optimizing the extraction of sugars, color, and the sugar/acid ratio.

2. Materials and Methods

2.1 Biological material

Date sampling was conducted at the N'Djamena market in collaboration with Chadian Institute of Agricultural Research for Development (ITRAD) The variety "*Bournow*" was the best indicated because of its availability, good conservation, producer appreciation, and high productivity. This variety accounts for 70 % of the Bornou district's production (Allarangaye *et al.,* 2011).

2.2 Physical characterization of date fruits

The physical characterization of date fruits was performed according to Sawaya *et al.* (1983). The color of the date fruits was assessed visually; the measurements of the entire fruit and its core (length and width) were taken with a caliper; and the mass (pulp and core) was determined using an analytical balance.

2.3 Determination of the water content of the whole date

The mass lost during drying under typical conditions determines the water content of dates (EBC-Analysis-Committee, 1998). It was calculated using 3 grams of date powder. To achieve a constant mass, the sample was crushed, spread in a porcelain capsule, and dried in a 103±2 °C for 24 h.

$$W(\%) = \frac{loss\ in\ weight}{initial\ weight\ of\ sample} \times 100 \qquad (1)$$

$$loss\ in\ weight = initial\ weight\ of\ sample - final\ weight\ of\ sample\ (after\ drying)$$

With, W (%): water content

2.4 Response Surface Methodology (RSM)

A mathematical model is an equation or set of equations that best describes the reality it depicts. This model will then be used to describe any analogous phenomenon as a whole, as well as to make predictions (interpolation, extrapolation). First, a phenomenon must be observed, followed by the construction of a model that reproduces it as precisely as possible, the identification of the model's limits, and finally, its validation. This mathematical modeling method corresponds well

to the scientific method steps used in the research program, namely observation, analysis, hypothesis, and validation (Gervais, 2007).

Temperature, extraction time, ratio (water/pulp), and pectinase volume were all variables. A centered composite design (CCD) was used to select the experiments values.

N = Nf + N + N0, or 24 + 24 + 4 = 28 trials, is the total number of tests performed.

The value of alpha for CCD was calculated to satisfy the condition of near orthogonality. The proposed model is a second-degree dual-interaction model. Each technique is carried out in triplicate. Minitab 21.3.1 was used to create the experimental design. For the laboratory experiments, the coded variables were converted into real variables (Desobgo *et al.,* 2010; Mathieu & Phan-tan-luu, 1997), resulting in the experimental design shown in Table 1. The transformation operations listed below were used:

$$U_j = U_j^0 + x_j \Delta U_j \quad (2)$$

$$U_j^0 = \frac{U_j^{\min} + U_j^{\max}}{2} \quad (3)$$

With: x_j, value of the coded variable j; U_j, value of the real variable j; U_j^0, value of the real variable j at the center of the domain; ΔU_j, increment; U_j^{max}, value of the real variable at the upper bound of the domain; U_j^{min}, value of the real variable at the lower bound of the domain.

The a priori postulated mathematical model is of the second-degree polynomial type with interactions. It is expressed as follows

$$y = a_0 + \sum a_i x_i + \sum a_{ij} x_i x_j + \sum a_{ii} x_i^2 + \varepsilon \quad (4)$$

Where: y, the response; x_j and x_i, the independent variables; a_0, constant; a_i, a_{ii} and a_{ij}, model coefficients and ε, error or residual.

2.5 Validation and optimization

To express the fit of second-degree equations, the determination coefficient R^2 was used. This coefficient of determination was insufficient for model validation on its own (Desobgo, 2012). The absolute average deviation (AAD) (Baş & Boyaci, 2007) was required to validate a model, as was the use of the bias factor and the accuracy factor (Ross, 1996). As a result, the model validation criterion was calculated using the formulas:

$$AAD = \frac{\left[\sum_{i=1}^{n}\left(\frac{\left|Y_{i,\exp} - Y_{i,theo}\right|}{Y_{i,\exp}}\right)\right]}{n} \quad (5)$$

$$B_f = 10^{\frac{1}{n}\sum_{i=1}^{n}\log\left(\frac{Y_{i,theo}}{Y_{i,\exp}}\right)} \quad (6)$$

$$A_f = 10^{\frac{1}{n}\sum_{i=1}^{n}\left|\log\left(\frac{Y_{i,theo}}{Y_{i,\exp}}\right)\right|} \quad (7)$$

With: AAD, absolute average deviation; B_f, bias factor; A_f, accuracy factor; $Y_{i,theo}$, response obtained using the model; $Y_{i,exp}$, response obtained via experiment; n, number of trials.

The calculated values must fall within the following ranges: AAD, 0-0.3; B_f, 0.75-1.25, and A_f, 0.75-1.25 (Dalgaard & Jørgensen, 1998).

The optimization was carried out using a multi-response approach that included maximizing the brix, color and sugar/acid ratio as specifications. The Minitab 21.3.1 software was used to find the best combination respecting all the specifications. Response optimization referred to a set of variable parameters that worked together to optimize a single response or a set of responses. This was useful for determining the effect of

multiple variables on a response. Minitab assigned an individual desirability to each response and determined it based on the importance assigned to it. These values were added together to determine the overall desirability of the multi-response system. When the composite desirability reached its maximum, an optimal solution emerged. Individual and composite desirability determined how well a combination of variables met the response objectives. Individual desirability measured how well the parameters optimized a single response, whereas composite desirability measured how well the parameters optimized a group of responses. Desirability is scaled from 0 to 1. A value of 1 would be ideal, while a value of 0 would indicate that one or more responses were out of the acceptable range. The weighted geometric mean of individual desirability for various responses would be composite desirability. Minitab determined the optimal parameters for the input variables by maximizing the composite desirability.

2.6 Juice extraction

A solid-liquid extraction was done here. Extractions were carried out in accordance with the parameters of the four-factor centered composite design (CCD). The extraction process of the date juice was carried out as shown in Figure 1.

The dates were sorted, washed, and pitted. They were then crushed to increase the exchange surface and facilitate juice extraction. This extraction was carried out 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 45°C, 10 min to 60 min, 2 to 5, and 0 mL to 0.5 mL, respectively. This resulted in 28 trials (Table 1), each of which was filtered through a filter cloth and then pasteurized for 15 seconds at 72 ± 2°C (Burapalit, 2019). The pasteurized juices were kept in the fridge at 4°C.

2.7 Physicochemical analysis of date extracts

Every analysis was performed in triplicate at each level, according to the experimental design. Brix, color, and sugar/acid ratio were all measured.

2.7.1 Determination of the Brix degree of date extracts

After calibrating the refractometer (Digital Refractometer HI96801) with distilled water (as a zero), a few drops of the sample were dispersed on the refractometer's prism, and the amount of soluble dry residue was recorded from the instrument's digital display. After each assay, the prism plate is cleaned with distilled water and a soft cloth. The operation is repeated three times for each sample.

2.7.2 Measurement of titratable acidity of date extracts

The titratable acidity was determined according to (Martínez *et al.*, 2012) as follows. About 10 mL of sample was introduced in a 250 mL beaker followed by 50 mL of distilled water Three drops of phenolphthalein at 1% concentration were added while stirring. When titrating, a solution of 0.1 N sodium hydroxide (NaOH) was used until a pink color was maintained for 10 seconds. The procedure was performed in triplicate. The formula to determine titratable acidity was given by:

$$A(\%) = \frac{0.0067 V_1}{V_0} \times 100 \qquad (8)$$

With: V_0: volume of sample taken for titration (mL); V_1: volume of 0.1 N sodium hydroxide solution used (mL); 0.0067: acidity conversion factor in malic acid equivalent.

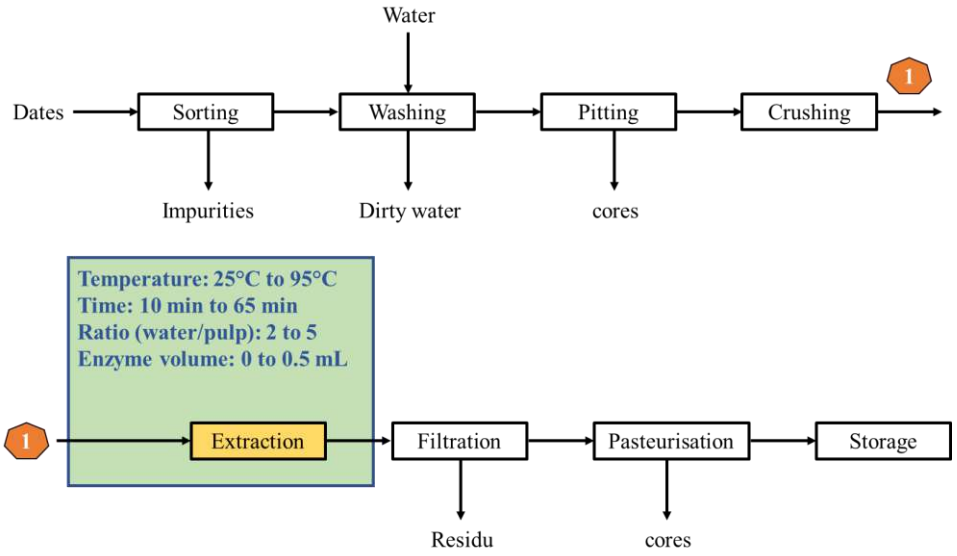

Figure 1: Schematic of date juice extraction process

2.7.3 Determination of the juice color

After collecting the juice sample, an absorbance reading was taken at 430 nm using a Jenway 6405 UV/Visible (Jenway Ltd Felstd, Dunmow, Essex CM6 3LB, UK) spectrophotometer so that the level of color intensity could be determined (ASBC, 2009).

$$Color\ (ASBC) = 12.7 \times Abs\ (430\ nm) \times F \qquad (9)$$

With: Abs, absorbance; F, dilution factor; ASBC, American Society of Brewing Chemists color units.

2.7.4 Determination of the juice color

Sugar/acid ratio (taste) was determined using TSS and TA values, as previously stated (Solomakhin & Blanke, 2010).

2.7.5 Determination of the free amino acid content of extracts

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

To make a 1/100 dilution, 99 ml of distilled water was mixed with 1 ml of extract. The sample was diluted and separated into three test tubes. Each test tube received 1ml of color reagent (100g/L Na_2HPO_4, 60g/L KH_2PO_4, 5g/L ninhydrin, and 3g/L fructose). The tubes were immersed in boiling water for 16 minutes. They were then chilled in a water bath to 20-25 °C. Each received 5ml of dilution solution (2g of KIO3, 1L of a mixture of 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 obtained results were compared to the control and standard results. To create the blank, 2ml of distilled water was used instead of the diluted extract. The standard was 2 ml of glycine (10.72 mg/L) instead of the diluted extract. The following relationship determined the proportion of free amino acids:

$$FAN\,(mg/L) = \frac{2 \times A_1}{A_2} \times d \qquad (10)$$

FAN: free amino nitrogen (mg/L); A_1: Absorbance of the test solution at 570 nm; A_2: Average absorbance of the standard solution; d: dilution factor

Table 1: Matrix of coded and real variables in the centered composite design

Trials	x1	x2	x3	x4	Temperature (°C)	Time (min)	Ratio (water/pulp)	Enzyme volume (ml)
1	0	0	0	0	60.0	65	3.5	0.25
2	-1	-1	1	1	38.2	31	4.4	0.41
3	1	1	1	1	81.8	99	4.4	0.41
4	1	1	-1	-1	81.8	99	2.6	0.09
5	-1	-1	-1	-1	38.2	31	2.6	0.09
6	0	0	1.607	0	60.0	65	5.0	0.25
7	0	0	-1.607	0	60.0	65	2.0	0.25
8	1	-1	-1	-1	81.8	31	2.6	0.09
9	-1	1	-1	1	38.2	99	2.6	0.41
10	0	-1.607	0	0	60.0	10	3.5	0.25
11	1.607	0	0	0	95.0	65	3.5	0.25
12	1	1	1	-1	81.8	99	4.4	0.09
13	0	1.607	0	0	60.0	120	3.5	0.25
14	1	1	-1	1	81.8	99	2.6	0.41
15	0	0	0	1.607	60.0	65	3.5	0.50
16	-1	1	1	-1	38.2	99	4.4	0.09
17	0	0	0	0	60.0	65	3.5	0.25
18	-1.607	0	0	0	25.0	65	3.5	0.25
19	1	-1	1	-1	81.8	31	4.4	0.09
20	-1	-1	-1	1	38.2	31	2.6	0.41
21	-1	1	-1	-1	38.2	99	2.6	0.09
22	0	0	0	0	60.0	65	3.5	0.25
23	0	0	0	-1.607	60.0	65	3.5	0.00
24	-1	1	1	1	38.2	99	4.4	0.41
25	0	0	0	0	60.0	65	3.5	0.25
26	1	-1	1	1	81.8	31	4.4	0.41
27	-1	-1	1	-1	38.2	31	4.4	0.09
28	1	-1	-1	1	81.8	31	2.6	0.41

2.7.6 pH measurement of date juice

The electrode of the pH meter (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.

2.7.7 Determination of the Vitamin C content of 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) (HPO_4-CH_3 COOH) and 10 mL of distilled water. A second empty Erlenmeyer flask was filled with a standard ascorbic acid solution (250 mg/L). Vitamin C (Vit C) was titrated with dichlorophenolindophenol (DCPIP) solution ($8.61x10^{-3}$ mol/L) for 30 seconds until a pink hue persists. The procedure was repeated thrice. The following formula was used to calculate the vitamin C content.

$$Vit\ C\ (mg/L) = \frac{[DCPIP] \times V \times M}{V_0} \qquad (11)$$

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

2.7.8 Determination of total phenolic content in date juice

The polyphenols in date juice were measured using the Folin-Ciocalteu reagent (Matloob & Balakita, 2016), which produces a blue phosphotungstic-phosphomolybdenum complex. 2 mL of distilled water and 1.0 mL of Folin-Ciocalteu reagent (diluted at 1/10) were added to 100 µL of sample extract. After allowing the mixture to stand for 5 min, 0.75 mL of Na_2CO_3 solution (60 g/L) was added. After 90 min, absorption was measured at 765 nm using an UV-visible spectrophotometer (Jenway Ltd Felstd, Dunmow, Essex CM6 3LB, UK) against water as a blank. For three replicates, the total phenol concentration was expressed as g of gallic acid equivalent (GAE) per 100 g of fresh sample.

3. Results and Discussion

3.1 Morphological characterization of date

The morphological characteristics of the *Bournow* date were measured as well as its water content. The results are presented in Table 2.

Bournow dates have a water content of 12% (Table 2). This characteristic leads to a relative stability of their quality over a relatively long period of time. This value is consistent with those discovered in the literature which ranged from 12% to 45% (Acourene *et al.,* 2001). Given the low moisture content of this date, it is classified as having a dry consistency according to Djoudi, (2013). In terms of date length, the cultivar *Bournow* produced shorter dates, which measured 1.78 cm (Table 2). This value was lesser than the maximum reported for other date cultivars, which ranged from 2.59 to 6 cm (Acourene *et al.,* 2001; Djoudi, 2013). In Table 2, it can be seen that the cultivar *Bournow* has a diameter of 0.72 cm. This value differed from those found in the 58 cultivars studied by Acourene *et al.* (2001) in the Ziban region which diameter was between 1.43 cm and 2.40 cm. This could be attributed to varietal differences. According to the literature classification (Mohammed *et al.,* 1983), the fruit and pulp masses of 5.56 g and 4.69 g are low because they are less than 6 g and 5 g, respectively (Table 2). Definitely, *Bournow* dates have a poor character globally for all measured characteristics. This would justify their use in Chad as animal feed. It could also be considered for valorization in beverages.

Table 2: Water content and morphological characterization of *Bournow* dates.

Characteristics	Values obtained	Limit	Caracter	decision	Reference
Water content (%)	12	12 % - 24 %	Medium	Reasonable	(Mohammed et al., 1983)
Diameter (cm)	0.72	< 1.5	Very little	Bad character	
Length (cm)	1.78	< 3.5	Short	Bad character	
Mass of the date (g)	5.56	< 6	Low	Bad character	
Pulp weight (g)	4.69	< 5	Low	Bad character	

3.2 Physicochemical characterization of the date juice

Although focused on the responses that are the subject of this work (Brix, color and sugar/acid ratio), other physicochemical characteristics were determined to have a global view on the properties of date juice. These data are exhibited in Table 3. From there it was seen that the pH ranged from 4.21 ± 0.02 to 5.62 ± 0.01, vitamin C from 45 ± 3 to 126 ± 6 mg/100 g, titratable acidity from 0.37 ± 0.04 to 2.13 ± 0.03 g/L, free amino nitrogen 430 ± 8 to 617 ± 11 mg/L and polyphenols from 270 ± 6 to 687 ± 8 mg GAE/100 g DM. When compared with literature, it was noticed that, the value obtained were for some characteristics (pH and titratable acidity) within the range obtained by Abbès *et al.* (2011) after applying pectinase/cellulase to obtain juice from date. Indeed, the values obtained by Abbès *et al.* (2011) were for pH and titratable acidity, respectively 3.12 to 4.87, 0.18 to 1.29 g/L. The result obtained demonstrates the impact of chosen process variables.

Table 3: Other physicochemical characteristics of *Bournow* date juice

	value	
	min	max
pH	4.21±0.02	5.62±0.01
Vit C (mg/100g)	45±3	126.±6
Titratable acidity (g/L)	0.37±0.04	2.13±0.03
Free amino nitrogen (mg/L)	430±8	617±11
Polyphenols (mg GAE/100g DM)	270±6	687±8

Table 4: Summary of physicochemical analysis of *Bournow* date (*Phoenix dactylifera* L.) juice

x1	x2	x3	x4	Brix (°B)	Color (ASBC)	Sugar/acid ratio
0	0	0	0	18	148	12.0624
-1	-1	1	1	15.5	172.5	10.9313
1	1	1	1	14.4	141.75	18.5757
1	1	-1	-1	21.2	180.5	46.9027
-1	-1	-1	-1	20.9	198	18.3041
0	0	1.607	0	13.2	103.25	12.5756
0	0	-1.607	0	25.9	168	34.078
1	-1	-1	-1	21.9	201	57.4171
-1	1	-1	1	21.5	153.5	21.278
0	-1.607	0	0	16.8	171.75	11.6193
1.607	0	0	0	17.3	145.25	35.2342
1	1	1	-1	13.4	148.25	36.2084
0	1.607	0	0	18	167.5	17.8241
1	1	-1	1	20.5	145.75	16.2319
0	0	0	1.607	17.1	179.25	11.0824
-1	1	1	-1	13.6	155	16.6667
0	0	0	0	17.1	143.75	9.6042
-1.607	0	0	0	17.3	166.5	13.9247
1	-1	1	-1	13.8	125.5	19.1332
-1	-1	-1	1	21.4	198	17.9681
-1	1	-1	-1	20.8	206	27.8075
0	0	0	0	16.7	134	11.016
0	0	0	-1.607	15.6	178.25	10.5774
-1	1	1	1	14.5	175.75	16.3848
0	0	0	0	16.3	150	8.8889
1	-1	1	1	13.9	167.5	16.1053
-1	-1	1	-1	13.3	158.75	6.2441
1	-1	-1	1	22.3	186.5	30.883

Table 5: Validation criteria of the different models from juice attributes

Settings	R^2	R^2_{adj}	AAD	Bf	Af
Y_{Bx}	98.34	96.55	0.061	0.994	1.064
Y_{Col}	93.37	86.24	0.088	0.934	1.096
$Y_{S/A}$	98.56	97.00	0.062	0.982	1.066

3.3 Modeling and optimization of physicochemical parameters of date juice

The influence of operating parameters (temperature, time, ratio, and enzyme volume) on the extraction of date juice was determined. The findings are provided in Table 4.

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

$$Y_{Bx} = 17.117 - 0.0014x_1 - 0.0286x_2 - 2.3075x_3 + 0.2208x_4 + 0.0055x_1^2 + 0.0180x_2^2 + 0.3454x_3^2 - 0.1294x_4^2 - 0.0411x_1x_2 - 0.0670x_1x_3 - 0.0895x_1x_4 + 0.0460x_2x_3 - 0.0282x_2x_4 + 0.0783x_3x_4 \quad (12)$$

$$Y_{S/A} = 10.11 + 0.186x_1 - 0.12x_2 - 1.46x_3 + 0.131x_4 + 0.042x_1^2 + 0.046x_2^2 + 0.248x_3^2 - 0.076x_4^2 - 0.006x_1x_2 - 0.086x_1x_3 - 0.053x_1x_4 + 0.056x_2x_3 - 0.018x_2x_4 + 0.047x_3x_4 \quad (13)$$

$$Y_{Col} = 143.66 - 4.55x_1 - 3.18x_2 - 9.65x_3 - 0.89x_4 + 1.889x_1^2 + 3.951x_2^2 - 1.148x_3^2 + 5.319x_4^2 - 0.659x_1x_2 - 0.902x_1x_3 + 0.103x_1x_4 + 2.281x_2x_3 - 2.765x_2x_4 + 4.157x_3x_4 \quad (14)$$

With, Y_{Bx}: Brix; $Y_{S/A}$: Sugar/acid ratio; Y_{col}: Color; x1: Temperature; x2: Time; x3: Ratio water/pulp; x4: Pectinase volume.

These interactive second-degree models are beneficial if a few input variables are precised. Table 5 shows that all models are valid and can thoroughly evaluate components. Table 5 ANOVA only considers variables with probability <0.05. Thus, they are the only relevant elements.

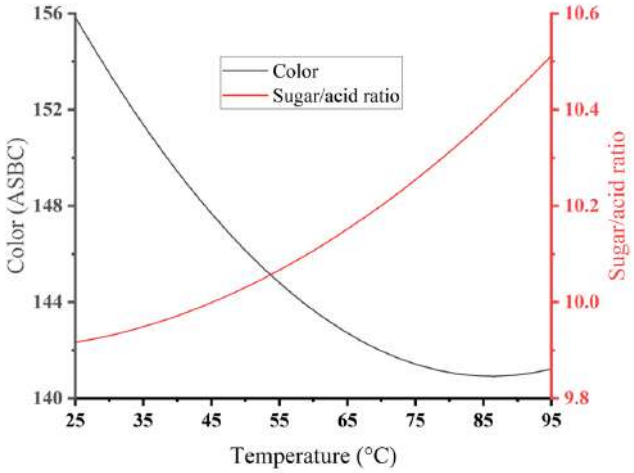

Figure 2: Evolution of color and sugar/acid ratio, as a function of temperature (time, water/pulp ratio, and pectinase volume fixed respectively at 65 min, 3.5, and 0.25 mL)

Table 6: ANOVA for the significance of the factors used during extraction of some constituents from *Bournow* date (*Phoenix dactylifera* L.) juice

Terms	Brix	Color	Sugar/acid ratio
	Probabilities (P)		
Constant	**0.000**	**0.000**	**0.000**
x_1-Temperature (°C)	0.988	**0.002**	**0.003**
x_2-Time (min)	0.744	**0.020**	**0.037**
x_3-Ratio (water/pulp)	**0.000**	**0.000**	**0.000**
x_4-Enzyme volume (mL)	**0.023**	0.475	**0.024**
x_1^2	0.935	0.067	0.319
x_2^2	0.792	**0.001**	0.277
x_3^2	0.100	0.246	**0.000**
x_4^2	0.076	**0.000**	0.081
x_1x_2	0.514	0.458	0.865
x_1x_3	0.295	0.314	**0.036**
x_1x_4	0.168	0.907	0.176
x_2x_3	0.467	**0.020**	0.152
x_2x_4	0.653	**0.007**	0.629
x_3x_4	0.224	**0.000**	0.224

3.3.1 Impact of singular factors on responses

3.3.1.1 Impact of temperature and time

The sugar/acid ratio increased significantly (P=0.000, Table 6) from 9.92 to 10.51 as the temperature increased from 25°C to 95°C (Figure 2). Although temperature has no effect on Brix in this scenario, it does cause cell weakening and thus more mineral extraction. Because the juice has alkaline ions in solution, they would engage in acid-base reactions, lowering the titratable acidity by that means. This resulted in an increase in the ratio. The sugar/acid ratio range (7.82-15.4)

indicated a healthy balance of sweetness and acidity, which was important for must quality and flavor. The findings were consistent with the literature, which placed this ratio at 10.78 for the production of good juice (Benidir *et al.,* 2020).

For temperatures ranging from 25 to 95 °C, the color of date juice decreased significantly (P=0.000, Table 6) from 155 to 141 ASBC. Date pigments include chlorophyll, carotene, and anthocyanins, which provide green, yellow, and red colors, respectively (Ashraf & Hamidi-Esfahani, 2011). Date syrups' color intensity may be affected by pigments such as carotenoids, flavonoids, tannin derivatives, and polyphenols (Benamara *et al.,* 1999). Lutein is the most abundant carotenoid pigment in dates, followed by β-carotene (Boudries *et al.,* 2007). Carotenoids were susceptible to destruction during processing and storage due to exposure to high temperatures, light, or pro-oxidant chemicals due to their highly unsaturated nature. As a result, carotenoid loss during food processing was documented and quantified in various articles (Aman *et al.,* 2005; Hiranvarachat *et al.,* 2008). The two major carotenoid changes that occurred during processing was isomerization and oxidation. Carotenoids occurred naturally in all-trans form. Heating, on the other hand, caused isomerization of all-trans-carotene to cis forms (Achir *et al.,* 2010).

For a duration of 10 to 120 min, the sugar/acid ratio decreased significantly (P=0.037, Table 6) from 10.42 to 10.03 (Figure 3). The consequence of the Maillard reactions was a reduction in the sugar/acid ratio, which is the opposite of what was shown for the temperature. In essence, the reducing sugar like glucose first condensed with a substance that had a free amino group (most commonly the E-amino group of lysine, but also the a-amino groups of terminal amino acids in proteins) to produce N-substituted glycosilamine, which then rearranged to produce the Amadori rearrangement product (Martins *et al.,* 2000). The residual amount lowers when the sugars were involved in that process, which caused the ratio to fall. This Maillard reactions induced also the reduction of amino nitrogen with time.

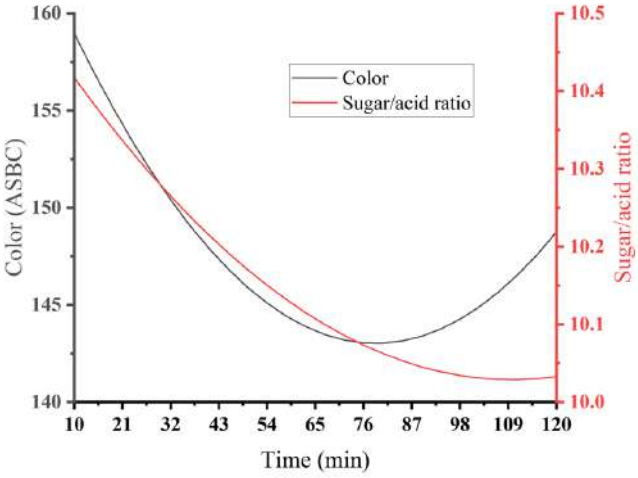

Figure 3: Evolution of color and sugar/acid ratio, as a function of time (temperature, water/pulp ratio, and pectinase volume fixed respectively at 65 min, 3.5, and 0.25 mL)

At 10 min, the color was 158.97 ASBC, and at 79 min, it was 143.02 ASBC, therefore it drops significantly (P=0.020, Table 6). Following that, there is a significant increase (P=0.001, Table 5) till 148.75 ASBC at 120 min (Figure 3). During the extraction, this phenomenon of date juice discolouration as a function of time and temperature was noticed (Benahmed Djilali & Adiba, 2012). Several pigments, including carotenoids, anthocyanins, flavones, flavonoles, lycopene, carotenes, flavoxanthin, and lutein, were found in dates (Echegaray *et al.,* 2021). The alteration of color and pigment during food products' heat processing is controlled by a variety

of factors. These include browning and process conditions that are enzymatic and non-enzymatic. Numerous studies on the qualitative characteristics of fruit-based pigments, particularly on carotenoid loss after heat processing, have been published in the literature (Ahmed & Ramaswamy, 2005). Indeed, compounds such as anthocyanins are sensitive to heat treatment (Weber & Larsen, 2017) and, the time of application of heat treatment would therefore contribute to a reduction of anthocyanins and therefore color. The color enhancement in the second stage could be due to Maillard reactions that increased with time. Thermally treated food can cause a chain reaction of reactions known as the Maillard reactions or nonenzymatic browning. These reactions are crucial in the development of flavor and color in heated food. Under certain conditions, the reaction happens between carbonyl groups of reducing sugars and free amino groups of amino acids, peptides, or proteins. The final stage of the Maillard reaction, where condensation of carbonyls and amines generates brown-colored high molecular weight molecules known as melanoidins, has been related to color development (Starowicz & Zieliński, 2019).

3.3.1.2 Impact of ratio water/pulp

Brix declined significantly (P=0.000, Table 6) from 21.72 °B to 14.30 °B as the water/pulp ratio increased from 2 to 5 (Figure 4). This could be explained by the diluting effect that an increase in water would have on sugar extraction. This was consistent with the literature, which reported the similar pattern for plantain juice extraction (Bentahar *et al.,* 2014; Makebe *et al.,* 2017). Like the brix, the decrease in all the other responses with the increase of water/pulp ratio (Figure 4) could be explained by the dilution brought with the increase of water.

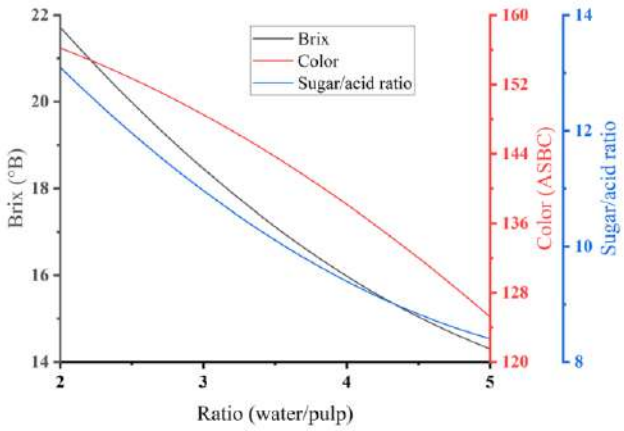

Figure 4: Evolution of brix, color and sugar/acid ratio, as a function of ratio water/pulp (temperature, time, and pectinase volume fixed respectively at 60°C, 65 min, and 0.25 mL)

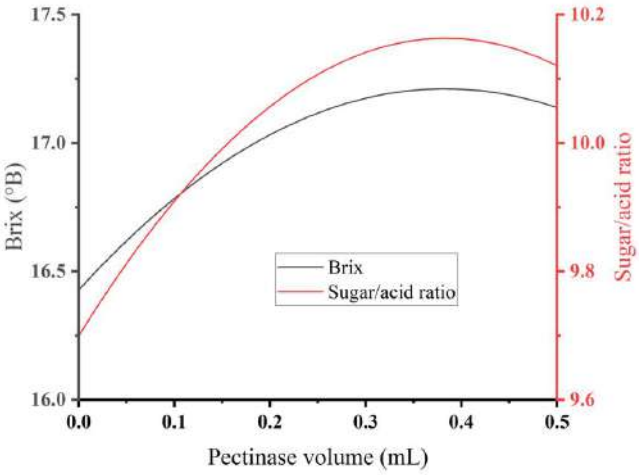

Figure 5: Evolution of brix and sugar/acid ratio, as a function of pectinase volume (temperature, time, and ratio water/pulp fixed respectively at 60°C, 65 min, and 3.5)

3.3.1.3 Impact of pectinase volume

Brix begun at 16.42 °B before increasing significantly (P=0.023, Table 6) to 17.14 °B, with the addition of 0.5 mL pectinase. Abbès *et al.* (2011) made the same observation for the production of date syrups. This was related to the

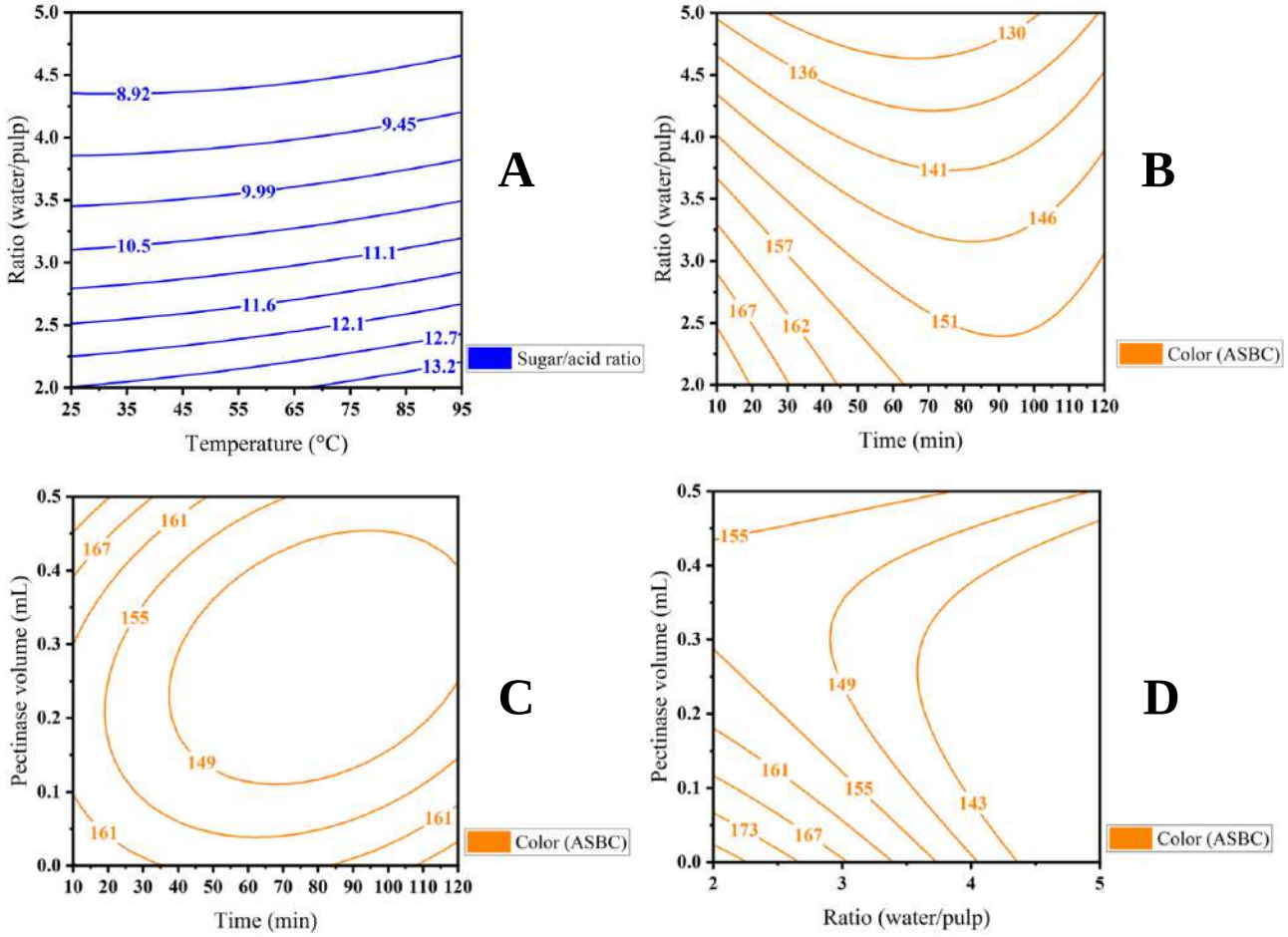

Figure 6: Evolution of sugar/acid ratio and color as a function of temperature/ratio (A), time/ratio (B), time/pectinase volume (C) and ratio/pectinase volume (D). All the other factors for each curve are fixed at the central value of the domain

breakdown of pectin into galacturonic acid units and sugars in the plant cell wall matrix and middle lamella (Abbès *et al.,* 2011; Demir *et al.,* 2001; Landbo *et al.,* 2007).

The volume of pectinase that was used, resulted in a significant increase (P=0.024, Table 6) in the sugar/acid ratio. It ranged from 9.7 when there was no pectinase present to 10.12 when there was 0.5 mL of pectinase present in volume (Figure 5). This rise can be explained by the fact that, as was observed earlier, the sugar release kinetics seemed faster than the titratable acid release kinetics, which resulted in an overall increase in the sugar/acid ratio.

3.2.2 Impact of interactions on responses

The interaction between x1 and x3 (temperature/water/pulp ratio) greatly reduced (P=0.036, Table 6) the sugar/acid ratio. In fact, this effect was accentuated simultaneously by a fall in temperature and a rise in the water/pulp ratio (Figure 6 A). This might be explained by the fact that the dilution effect dropped the brix quicker than the titratable acidity, which decreased the sugar/acid ratio. Paul *et al.* (2018), who evaluted

the effect of dilution on banana fermentation, made similar observation. The simultaneous drop in temperature and sugar/acid ratio demonstrates that the date's structure plays a crucial role in fixing sugar and restricting its release.

The x2x3 interaction (time, water/pulp ratio) significantly contributed to the color increase (P=0.020, Table 6). This was seen when the extraction time increased while the water/pulp ratio decreased (Figure 6 B). Indeed, a longer extraction time would help to extract the elements that contributed to the color, such as pigments and polyphenols, while a decrease in the water/pulp ratio would concentrate the medium, resulting in an increase of the date juice's color. Furthermore, a longer extraction time would permit an increase in color due to Maillard reactions since the temperature was fixed at 60 °C. Melanoidins are compounds formed during the late stages of the Maillard reaction from reducing sugars and proteins or amino acids. In food, they have been identified as anionic and colored compounds (Echavarría *et al.,* 2012).

The x2x4 interaction (time, pectinase volume) significantly contributed to color reduction (P=0.007, Table 6). This occurred when the extraction time increased while the pectinase volume reduced (Figure 6 C). With time, the pigments responsible for the color degrade (Benahmed Djilali & Adiba, 2012), and the decrease in the volume of pectinase contributes to the non-destruction of the pulp structure, reducing the extraction of the compounds responsible for the color.

The x3x4 interaction (time, pectinase volume) significantly contributed to the color increase (P=0.000, Table 6). This was seen when the volume of pectinase increased while the water/pulp ratio decreased (Figure 6 D). Indeed, lowering the ratio helps to reduce the dilution effect, while increasing the pectinase volume helps to weaken the pulp through pectin hydrolysis (Srivastava & Tyagi, 2013), resulting in the release of the compounds responsible for the color.

3.2.3 Optimization

To achieve the best physicochemical properties of the date juice, multi-response optimization was used. Brix, color, and sugar/acid ratio were all maximized for this purpose. At the end of this Minitab 21.3 optimization, the compromise was as follows: extraction temperature 95 °C, time 10 min, water/pulp ratio 2 and pectinase volume 0 mL. This combination produced a Brix of 21.89 °B, a sugar/acid ratio of 13.99, and a color of 197.49 ASBC. The individual desirability for the brix, color and ratio sugar/acid were respectively 0.683, 0.917 and 0.768. While the composite desirability was 0.784. The composite desirability (0.7840) in the study was close to 1, indicating that the parameters appeared to produce favorable results for all responses as a whole. Individual desirability, on the other hand, indicated that the parameters were more effective in maximizing color (0.91718) than for sugar/acid ratio (0.76893), and finally for brix (0.68336).

4. Conclusion

The goal of this work was to contribute to the valorization of the date *Bournow* variety in the field of drinks. Its poor physical character, which renders it unmarketable and consumable as is, confirmed the need for valorization. The response surface methodology was used to extract the juice, which allowed us to see that the selected factors, which are temperature, time, water/pulp ratio, and pectinase volume, have different effects on the responses Brix, color, and sugar/acid ratio, either increasing or decreasing them significantly. This extraction allowed us to highlight the nutrient richness of date juice, as observed by other

authors on other varieties of dates. The optimization, which consisted of maximizing all of the responses (Brix, color, and sugar/acid ratio), resulted in the quadruplet: temperature 95 °C, time 10 min, water/pulp ratio 2, and pectinase volume 0 mL. The resulting Brix was 21.89 °B, the sugar/acid ratio was 13.99, and the color was 197.49 ASBC. The nutrient richness of the juice produced under optimal conditions suggests that the juice's quality could be improved, and the Bournow date could be used in other industries such as fermented beverages.

Acknowledgments

The authors warmly thank the Department of Process Engineering of the National School of Agro-industrial Sciences (ENSAI), University of Ngaoundere (Cameroon), the LAGETA and LBP laboratories of ENSAI and the IUT respectively, as well as the University of Ngaoundere for having provided the necessary facilities for the success of this research work Furthermore, great gratitude goes also to the University of Ndjamena through its doctoral school.

Conflict of interest

The authors declare that there are not conflicts of interest.

Ethics

This Study does not involve Human or Animal Testing.

References

Abbès, F., Bouaziz, M. A., Blecker, C., Masmoudi, M., Attia, H., & Besbes, S. (2011). Date syrup: effect of hydrolytic enzymes (pectinase/cellulase) on physico-chemical characteristics, sensory and functional properties. *LWT-Food Science and Technology*, *44* (8), 1827–1834.

Achir, N., Randrianatoandro, V. A., Bohuon, P., Laffargue, A., & Avallone, S. (2010). Kinetic study of β-carotene and lutein degradation in oils during heat treatment. *European Journal of Lipid Science and Technology*, *112* (3), 349–361.

Acourene, S., Buelguedj, M., Tama, M., & Taleb, B. (2001). Caractérisation, évaluation de la qualité de la datte et identification des cultivars rares de palmier dattier de la région des Ziban. *Recherche Agronomique, Éd. INRAA*, *8*, 19–20.

Ahmed, J., & Ramaswamy, H. S. (2005). Effect of temperature on dynamic rheology and colour degradation kinetics of date paste. *Food and Bioproducts Processing*, *83* (3), 198–202.

Allarangaye, M. D., Sougnabe, P., & Ache, A. (2011). *Etude diagnostique des contraintes affectant la culture des palmiers dattiers dans les oasis de l'Ennedi au Tchad*.

Amadou, I., & Le, G. W. (2017). Nutritional and sensory attributes of desert date (Balanites aegyptiaca) juice. *Journal of Food Measurement and Characterization*, *11* (4), 1978–1986.

Aman, R., Biehl, J., Carle, R., Conrad, J., Beifuss, U., & Schieber, A. (2005). Application of HPLC coupled with DAD, APcI-MS and NMR to the analysis of lutein and zeaxanthin stereoisomers in thermally processed vegetables. *Food Chemistry*, *92* (4), 753–763.

ASBC. (2009). *Methods of Analysis* (2009th ed.). American Society of Brewing Chemists Inc.

Ashraf, Z., & Hamidi-Esfahani, Z. (2011). Date and date processing: A review. *Food Reviews International*, *27*(2), 101–133. https://doi.org/10.1080/87559129.2010.535231

Baş, D., & Boyaci, I. H. (2007). Modeling and optimization i: Usability of response surface methodology. *Journal of Food Engineering*, *78*(3), 836–845. https://doi.org/10.1016/j.jfoodeng.2005.11.024

Benahmed Djilali, A., & Adiba. (2012). *Analyse des aptitudes technologiques de poudres de dattes (Phoenix-dactylifera L.) améliorées par la spiruline : étude des propriétés rhéologiques, nutritionnelles et antibactériennes* [Thèse de Doctorat]. Faculté des

Sciences de l'Ingénieur de l'Université M'Hamed Bougara .

Benamara, S., Messoudi, Z., Bouanane, A., & Chibane, H. (1999). Formulation et analyse de la couleur d'une confiture d'abricot allégée. *Industries Alimentaires et Agricoles*, *116* (1–2), 27–33.

Benidir, M., el Massoudi, S., el Ghadraoui, L., Lazraq, A., Benjelloun, M., & Errachidi, F. (2020). Study of nutritional and organoleptic quality of formulated juices from jujube (*Ziziphus lotus* L.) and dates (*Phoenix dactylifera* L.) fruits. *The Scientific World Journal*, *2020*.

Bentahar, I., Hassouna, M., & Amrane, A. (2014). Optimisation de l'extraction des jus de sous-produits de dattes (Phoenix dactilyphera L.) et valorisation par production de bioéthanol. *Revue Des Energies Renouvelables*, *17* (4), 529–540.

Boudries, H., Kefalas, P., & Hornero-Méndez, D. (2007). Carotenoid composition of Algerian date varieties (*Phoenix dactylifera*) at different edible maturation stages. *Food Chemistry*, *101* (4), 1372–1377.

Burapalit, K. (2019). *Comparison of date palm juice and syrup made from premature fruit drop, fresh and dried date pulp Barhee cultivar (Phoenix dactylifera L.)*.

Chirinos, R., Pedreschi, R., Rogez, H., Larondelle, Y., & Campos, D. (2013). Phenolic compound contents and antioxidant activity in plants with nutritional and/or medicinal properties from the Peruvian Andean region. *Industrial Crops and Products*, *47*, 145–152. https://doi.org/10.1016/J.INDCROP.2013.02.025

Dalgaard, P., & Jørgensen, L. V. (1998). Predicted and observed growth of Listeria monocytogenes in seafood challenge tests and in naturally contaminated cold-smoked salmon. *International Journal of Food Microbiology*, *40*(1–2), 105–115. https://doi.org/10.1016/S0168-1605(98)00019-1

Demir, N., Acar, J., Sarioglu, K., & Mutlu, M. (2001). Use of commercial pectinase in fruit juice industry. Part 3: Immobilized pectinase for mash treatment. *Journal of Food Engineering*, *47*(4), 275–280. https://doi.org/10.1016/S0260-8774(00)00127-8

Desobgo, Z. S. C., Nso, E. J., Tenin, D., & Kayem, G. J. (2010). Modelling and optimizing of mashing enzymes - Effect on yield of filtrate of unmalted sorghum by use of response surface methodology. *Journal of the Institute of Brewing*, *116* (1), 62–69. https://doi.org/10.1002/j.2050-0416.2010.tb00399.x

Djoudi, I. (2013). *Contribution à l'identification et à la caractérisation de quelques accessions du palmier dattier (Phoenix Dactylifera.l) dans la région de Biskra* [Magister]. Faculté des Sciences Exactes et Sciences de la Nature et de la Vie, Université Mohamed Kheider Biskra.

EBC-Analysis-Committee. (1998). *Analytica-EBC, The European reference methods for breweries by EBC*. Fachverlag Hans Carl.

Echavarría, A. P., Pagán, J., & Ibarz, A. (2012). Melanoidins Formed by Maillard Reaction in Food and Their Biological Activity. *Food Engineering Reviews*, *4*(4), 203–223. https://doi.org/10.1007/S12393-012-9057-9/Metrics

Echegaray, N., Gullón, B., Pateiro, M., Amarowicz, R., Misihairabgwi, J. M., & Lorenzo, J. M. (2021). Date Fruit and Its By-products as Promising Source of Bioactive Components: A Review. https://doi.org/10.1080/87559129.2021.1934003

Estanove, P. (1990). *Note technique: Valorisation de la datte*. CIHEAM-IAMM.

Gervais, G. (2007). Modélisation et réalité : la rencontre des mathématiques et des sciences. *Spectre Thématique*, *37*, 19–24.

Hiranvarachat, B., Suvarnakuta, P., & Devahastin, S. (2008). Isomerisation kinetics and antioxidant activities of β-carotene in carrots undergoing different drying techniques and conditions. *Food Chemistry*, *107* (4), 1538–1546.

Landbo, A. K., Kaack, K., & Meyer, A. S. (2007). Statistically designed two step response surface optimization of enzymatic prepress treatment to increase juice yield and lower turbidity of elderberry juice. *Innovative Food Science and Emerging Technologies*, *8*(1), 135–142. https://doi.org/10.1016/j.ifset.2006.08.006

Mahmoud, O. H. A., Zango, O., Gapili, N., Ibrahim, M. L. A., Rafiou, A., Chabrillange, N., Aberlenc, F., Bakasso, Y., & Bell, J. M. (2022). Agrobiodiversity

and Sustainability of Oasis Agrosystems in Palm Groves of Sahara and Sahel in Chad. *Journal of Agricultural Science*, *14* (4), 99-113.

Makebe, C., Desobgo, Z., & Nso, E. (2017). Optimization of the Juice Extraction Process and Investigation on Must Fermentation of Overripe Giant Horn Plantains. *Beverages*, *3*(4), 19. https://doi.org/10.3390/beverages3020019

Martínez, J. J., Hernández, F., Abdelmajid, H., Legua, P., Martínez, R., el Amine, A., & Melgarejo, P. (2012). Physico-chemical characterization of six pomegranate cultivars from Morocco: processing and fresh market aptitudes. *Scientia Horticulturae*, *140*, 100–106.

Martins, S. I. F. S., Jongen, W. M. F., & van Boekel, M. A. J. S. (2000). A review of Maillard reaction in food and implications to kinetic modelling. In *Trends in Food Science and Technology* (Vol. 11, Issues 9–10, pp. 364–373). Elsevier. https://doi.org/10.1016/S0924-2244(01)00022-X

Mathieu, D., & Phan-tan-luu, R. (1997). Approach of response surface methodology. In J. Droesbeke, J. Fine, & G. Saporta (Eds.), *Surface of experiments* (pp. 211–277). Technip editions.

Matloob, M. H., & Balakita, A. A. A. H. (2016). Phenolic content of various date palms fruits and vinegars from Iraq. *International Journal of Chemical Sciences*, *14*, 1893–1906.

Mohammed, S., Shabana, H. R., & Mawlod, E. A. (1983). Evaluation and identification of Iraqi date cultivars: fruit characteristics of fifty cultivars. *Date Palm Journal*, *2* (1), 27–55.

Paul, G. P., Satav, P. D., Pethe, A. S., & More, S. M. (2018). Effect of must dilution on fermentation of banana fruit pulp into white wine. *International Journal of Food Science and Nutrition*, *3* (2), 207–208.

Ross, T. (1996). Indices for performance evaluation of predictive models in food microbiology. *Journal of Applied Bacteriology*, *81*(5), 501–508. https://doi.org/10.1111/j.1365-2672.1996.tb03539.x

Sawaya, W. N., Khalil, J. K., Safi, W. N., & Al-Shalhat, A. (1983). Physical and Chemical Characterization of Three Saudi Date Cultivars at Various Stages of Development. *Canadian Institute of Food Science and Technology Journal*, *16* (2), 87–92. https://doi.org/10.1016/S0315-5463(83)72065-1

Solomakhin, A., & Blanke, M. M. (2010). Can coloured hailnets improve taste (sugar, sugar: acid ratio), consumer appeal (colouration) and nutritional value (anthocyanin, vitamin C) of apple fruit? *LWT - Food Science and Technology*, *43*(8), 1277–1284. https://doi.org/10.1016/J.LWT.2010.02.020

Srivastava, S., & Tyagi, S. K. (2013). Effect of enzymatic hydrolysis on the juice yield from apple fruit (*Malus domestica*) pulp. *International Journal of Biotechnology and Bioengineering Research*, *4*(4), 299–306.

Starowicz, M., & Zieliński, H. (2019). How Maillard reaction influences sensorial properties (color, flavor and texture) of food products? *Food Reviews International*, *35*(8), 707–725.

Weber, F., & Larsen, L. R. (2017). Influence of fruit juice processing on anthocyanin stability. *Food Research International*, *100*, 354–365.

Cite this paper as: Kadlezir, F., Mohagir, A.M. & Desobgo, Z.S.C. (2023). Application of response surface methodology in date (*Phoenix dactylifera* L.) juice extraction: Effect of process parameters on Brix, color and sugar/acid ratio. *Journal of Food Stability*, 6 (2), 66-83. DOI: 10.36400/J.Food.Stab.6.2.2023-005