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Mixture design for the optimization of Tunisian Deglet Nour Date Jam formulation

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Abstract
The valorization activities of second grade Deglet Nour date fruits into innovative products may help to overcome the new challenges of meeting foods growing needs. Date jam production is among these activities that is increasingly developed during last years. The objective of this work was to model and optimize the formulation of date jam using three-factor mixture design. Results showed that reduced quadratic models predict well the studied responses of TSS (%), aw, pH, color and overall appreciation. Optimal formulation based on healthy constraints, namely sugar reduction as well as best sensory overall appreciation is 150 g of date paste, 68.12 g of sugar and 381.89 g of water. The jam produced following the optimal formulation is characterized by a high amount of date paste which promotes its nutritional value. Physicochemical analysis showed that it has a TSS (Brix) of69.5%, a_w of 0.785 and pH of 4.94. The findings of sensory analysis revealed that date jam is well acceptable given that overall appreciation and color attribute are about 3.5 and 3.83, respectively.

Keywords: Deglet Nour date paste, formulation, date jam, D-optimal mixture design, optimization

Introduction

Consumption of fruits has always been associated with better health as well as stronger immunity and is highly recommended by the World Health Organization (Aggarwal *et al*., 2020). Date, fruit of palm tree (*Phoenix dactylifera* L.), is known to be a good source of minerals, dietary fiber with an average content of 13.1-15.8% of dry matter and bioactive compounds [1] whose health benefits have been increasingly studied over the past two decades and are well demonstrated [2].

Date palm (*Phoenix dactylifera* L.) is abundantly cultivated in arid and semi-arid regions of the world, such as North Africa, the Arabian Peninsula and Iran [3]. In Tunisia, the growing of palm trees and the production of dates are major agricultural activities that play a significant role in agro-ecosystem. With an annual production of 241,000 metric tons of dates and a total area of 42,000 hectares, Tunisia is one of the most important date producers in the world [4] and is currently the first exporter of dates in value. As a result, a significant industrial activity has been developed. However, this latter activity faces a major sustainability challenge as over 10% (w/w) of production is disposed of as waste or by-products [5]. Overcoming this challenge and valorizing second-grade dates have gained great importance given theirunique properties, high nutritional value and considerable health benefits [6]. Several processed productshave been developed such as jam, juice, syrup,paste and fermented products [7, 8].

Jams are popular not only fortheir essential nutrients but also for their versatile uses and good shelf life with organoleptic characteristic maintained for over one year [9]. Date jamis commonly prepared by cooking a mixture of fruit pulp, sugar and water until adesired consistency, characteristic of semi-solid food product, is obtained. It has a delicious taste [6] mainly due to sugars and floral composition of dates. Despite, the advantages of date jam production, there is limited scientific data on optimizing jam formulation from the Tunisian Deglet Nour variety.

In the present study, we used the Response Surface Methodology (RSM) to formulate jam based on paste produced from second-grade Deglet Nour dates. A three-factor (date paste, sugar and water) D-optimal mixture design was performed in order to optimize jam formulation considering its physico-chemical and sensory characteristics.

Materials and Methods

Materials

The ingredientswhich were used in the date jam formulations are date paste, commercial crystalized sucrose and distilled water. Date paste used in this study for preparing jam formulations was produced in Vacpa Boudjebel Company from second-grade Deglet Nour dates under optimized conditions as described by Ben Amara *et al*. [5].

Date Jam preparation

The weighted three ingredients (water, date paste, sugar) were firstly blended using an electric stirrer (Moulinex HV8, SEB, France). The obtained mixture was then cooked in a laboratory heater (Afrimesure, Tunisia)at about 90°C for three hours. The prepared jams were then cooled until reaching 4°C.

Physicochemical and sensory analysis

Total Soluble Solids content (TSS)

TSS (Brix) content (%) was measured according to ISO 2173 standard using arefractometer (ATAGO, HUMO, France).

Water activity

The measurement of water activity (a_w) was performedaccording to ISO 18787 standard using water activity meter (Lab Swif, Novasina, Switzerland).

pH

pH was measured using pH meter (HI5221, HANNA, USA) according to ISO 1842 standard.

Overall appreciation and color

Color and overall appreciation are among the main parameters that characterize the quality of date jam. Sensory evaluation of these parameters were undertaken by a group of 15 panelists using 5-point scale. Very appreciated and light color as well as disliked and very dark color correspond to 5 and 1, respectively.

Experimental Mixture Design

A D-optimal mixture design was used in this study to realize formulations based on the three factors corresponding to the ingredients used for date jam preparation. The levels of the three factors were chosen based on industrial previous experiments and are summarized in table 1.

Table 1: Levels of D-optimal mixture design factors

Variable	Variable Symbols		Level	
	Coded	Uncoded	Coded	Uncoded
Date paste (g)	x_1	X_1	0	50
			0.5	150
Sugar (g)	X_2	X_2	0	50
			0.5	150
Water (g)	X_3	X_3	0	300
			0.5	400

The D-optimal design used in this work consists of ten randomized experimental runs which are shown in Table 2.

Table 2: D-optimal mixture design runs

Run	Factors		
	Date paste (g)	Sugar (g)	Water (g)
1	116.67	116.67	366.67
2	100	150	350
3	116.67	116.67	366.67
4	150	100	350
5	50	150	400
6	116.67	116.67	366.67
7	150	150	300
8	100	100	400
9	150	50	400
10	100	100	400

Each determined response (Y_i) of TSS, aw, pH and color as well as overall appreciation was modeled based on the reduced quadratic equation[9] described as follows:

$$Y_i = \sum_{i=1}^{3} \beta_i X_i + \sum_{\substack{i,j=1 \\ i<j}}^{3} \beta_{i,j} X_i X_j$$

Where β_i and $\beta_{i,i}$ are the coefficents of the model.

Formulation optimization and validation

Optimization of the formulations was performed based on reducing the amount of added sucrose in order to produce a morehealthy jam along with maximizing the sensory overall appreciation. As it is recommended by industrial actors' requirements, TSS (Brix) was also set to a target of 69% since it promotes, in particular, the shelf life stability of date jam. All the responses of the optimal found formulation were measured experimentally in order to validate the optimization procedure.

Statistical analysis

The significance level for testing models and their coefficients is 5%. Statistical validation has been undertaken based on the predicted interval of the responses values of the optimal formulation replicated three times.

Results and discussion

The date paste material used in this study has been produced by Ben Amara *et al.* [5] under optimal process production conditions. It is characterized by a medium moisture content 17.00%±0.5 (w.b), and it presents a high nutritional valuewith high amounts of carbohydrates (68.50± 3.18 g/100 g), total dietary fibers (9.80 ± 2.10 g/100 g)and total phenols (261.00 ± 6.20 mg GAE/100 g). Furthermore, Date paste has a considerable antioxidant potential with a value of 2.94 ± 0.05 mmolTroloxEq/g Extract [5].

Experimental mixture Design and modeling

All experimental determined values of the responses of the performed three factors D-optimal mixture design are shown in table 3.

Table 3: Responses values of D-optimal mixture design runs

Run	Factors			Responses				
	Date paste (g)	Sugar (g)	Water (g)	TSS (%)	a_w	pH	Overall appreciation	Color
1	116.67	116.67	366.67	69.4	0.8	5.05	3.2	3.8
2	100	150	350	67.6	0.79	5.05	1.87	3.53
3	116.67	116.67	366.67	67.9	0.8	4.82	3.13	4.27
4	150	100	350	72.1	0.77	5.02	3.6	3.27
5	50	150	400	71.3	0.76	5.54	1	1
6	116.67	116.67	366.67	67.9	0.8	4.82	3.07	4.27
7	150	150	300	69.7	0.78	5.04	3.73	3.6
8	100	100	400	65.7	0.82	4.96	3.47	4.4
9	150	50	400	65.6	0.8	4.99	3.33	3.8
10	100	100	400	65.7	0.82	4.96	3.47	4.4

The following equations are the predictive models for each response in terms of actual factors:

$$Y_{TSS} = 0.739\,X_1 - 0.38\,X_2 + 0.036\,X_3 - 0.001X_1X_2 - 0.0$$

$$Y_{a_w} = -3.653 \cdot 10^{-3}\,X_1 + 2.347 \cdot 10^{-3}X_2 + 1.855 \cdot 10^{-3}$$
$$- 7.429 \cdot 10^{-6}\,X_2X_3$$

$$Y_{pH} = 5.228 \cdot 10^{-2}\,X_1 + 1.358 \cdot 10^{-2}X_2 + 1.022 \cdot 10^{-2}\,X_3$$

$$Y_{OA} = 2.58 \cdot 10^{-2}\,X_1 - 8.82 \cdot 10^{-2}X_2 + 2.536 \cdot 10^{-2}\,X_3 +$$

$$Y_{Color} = -2.339 \cdot 10^{-1}\,X_1 + 004.733 \cdot 10^{-3}X_2 + 1.266 \cdot 1$$

Where Y_{TSS}, Ya_w, Y_{pH}, Y_{Color} and Y_{OA} are the Brix (%), water activity, pH, color and overall appreciation responses, respectively.

Table 4: Statistical parameters of models

	TSS (%)	a_w	pH	Overall appreciation	Color
p (model)	0.0041	<0.0001	0.0127	<0.0001	0.0012
R^2	96.88%	99.83%	89.09%	99.81%	98.35%
Adjusted R^2	92.98%	99.63%	80.37%	99.57%	96.29%

The statistical parameters (Table 4) show the significance and the adequacy of all reduced quadratic responses' models.

Formulation optimization and validation

As it is stated previously, optimization of the formulation has been performed on the basis of ensuring the stability of the jam during storage, healthy considerations (sugar reduction) and enhancing appreciation.

Figures 1a, 1b, 1c, 1d and 1e show the contour plots of the responses studied in this work. In order to enhance TSS content to reach the target value while reducing sugar, Figure 1a shows that date paste should be in the right upper region between contours of 68 and 70%. From figure 1d, it can be seen that the same right upper region corresponds to the highest values of overall appreciation meaning that enhancing date paste and decreasing sucrose contribute to a better taste. In this region, the values of a_w, pH and color are between contours of 0.78 and 0.8, close to 5 and greater than 3, respectively (Figure 1b, 1c and 1e).

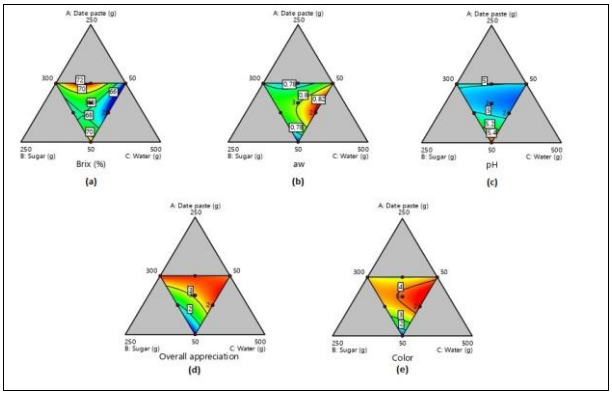

Fig 1: Contour plotsof Brix **(a)**, a_w **(b)**, pH **(c)**, overall appreciation **(d)** and color **(e)**

Optimal formulation of date Jam (figure 2) is composed of 150 g of date paste, 68.12 g of sugar and 381.89 g of water.

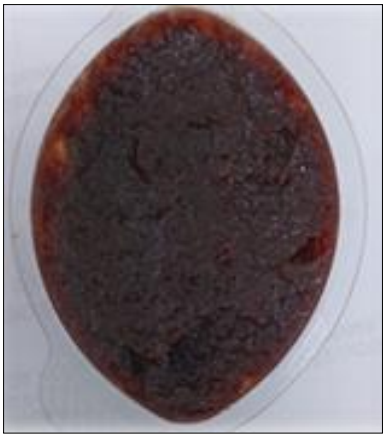

Fig 2: Date Jam prepared following optimal formulation

The predicted responses and experimental average values of TSS, aw, pH, overall appreciation and color are69%, 0.785, 4.995, 3.46 and 3.524, respectively. The 95% predicted intervals were [67.45%, 70.55%], [0.782, 0.788], [4.78, 5.21], [3.33, 3.59] and [3.08, 3.96], respectively for TSS, aw, pH, overall appreciation and color. Optimal formulation experimental average measured values of TSS, aw, pH, overall appreciation and color are 69.52%, 0.785, 4.94, 3.5 and 3.83, respectively. Consequently, the validity of the optimal formulation is demonstrated given that experimental average values are within predicted intervals.

The TSS value of optimal formulation is in agreement with the finding of Besbes *et al.* [3] and shows that date jam is very rich in sugars. It's worth mentioning that the significant amount of sugarsplays an important role for its microbiological stability [3]. The obtained value of water activity (0.785) confirms its classification as an intermediate moisture food product and its safety for development of the majority of bacteria given that its water activity is lower than 0.86 [10].

The obtained pH of the optimum product was 4.94 which is close to the values reported by Allouache *et al.* [11] for some persimmon jelly preparations (about 4.60). Without any addition of citric acid like it was performed in [3], the date jam obtained in this study is naturally acid. This acidity enhances its shelf life stability as it has an inhibitory effect on microorganisms' growth [12]. According to the literature, the color produced by jam depends on the level of maturity of fruit used [13] or by the cooking effect. Alqahtani *et al.* [6] reported that jam products showed a slightly dark color which could be due to the non-enzymatic browning through sugar caramelization and Maillard reactions. Nevertheless, the brown color of our final date jam is mainly reminiscent of the whole date color. The positive sensory scores of color (3.83) and overall appreciation (3.5) of our jam produced under optimal formulation confirm its good acceptability.

Conclusion

In this study, a jam preparation process from a highly nutritious fruit (*Phoenix dactylifera* L.) usingdate paste was developed. Date jam formulation was realized using an experimental mixture design with three independent factors (date paste, sugar and water). The physicochemical and sensory parameters were studied and modeled using the reduced quadratic equation. Optimization based on sugar reduction, enhancing appreciation and enhancing the jam stability during storage was performed. The findings of the present work suggest that the date jam was successfully developed with good overall appreciation and desirable characteristics.

The knowledge of the physico-chemical and sensory properties of this product will encourage the production of date jam on an industrial scale. The use of dates may also be attractive to consumers as a positive alternative to commonly used fruits in jam production.

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