Feasibility study of date (*Phoenix dactylifera* L.) fruit syrup-based natural jelly using central composite design

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Abstract A feasibility study of natural fruit jelly from three Algerian raw materials, namely date (*Phoenix dactylifera* L.) fruit syrup and suspension of orange albedo powder (OAP) in lemon juice (LJ) was performed by response surface methodology (RSM) based on central composite design (CCD). The textural properties of the final jelly were investigated through two dependent variables: hardness and stickiness. The cooking temperature (X_1), corresponding to that of thermo stated oil bath, and cooking time (X_2), taken for heating the initial fruit mixture in the oil bath (from ambient temperature without fixing however, the final temperature), were found to be the most influent factors, compared to °Brix of date syrup (X_3) and temperature (X_4) of the cooling stage following the cooking process. Results have also shown that the second-degree polynomial models correctly fit experimental data (R^2 , adjusted R^2 (R^2_{adj}) and cross-validation (Q^2) ≈ 1). Considering textural properties of commercial jellies as a reference, it was found that the cooking temperature of 155 °C for 10 min gave a jelly with suitable textural properties. On the other hand, FT-IR spectra revealed that the structure of such jelly was partially close to that of pectin molecules. Finally, the color analysis in the CIELab system of the fruit mixture over the cooking

process showed that both lightness (L^*) and a^*/b^* ratio were not affected by the experienced temperature range (80–155 °C).

Keywords Jelly · Textural properties · Central composite design · Date syrup · Suspension of orange albedo powder in lemon juice

Introduction

Several research studies highlight the determining role of food in health preservation. In general, the resulting recommendations converge to emphasize fruits and vegetables, considering the synergistic effects of their phytochemicals (Liu 2003) and their natural character, which are associated with reduction of several diseases like cancer (Verhoeven et al. 1996).

Beside nutritional and therapeutic values, the rheological and textural properties also determine the general quality of fruit jams and jellies. Some works were devoted to mechanical properties of banana peel powder-based jelly (Lee et al. 2010), strawberry jams (Kopjar et al. 2009), sapota (*Quararibea cordata* Vischer) pulp jelly (Carvalho et al. 2012). However, to our knowledge, no work has dealt with the gelling process of complex suspension from natural raw components without any chemically processed ingredients. In addition, among all reports found in the literature, none has discussed the simultaneous effect of temperature and time of both cooking and cooling stages, on the quality of jellies, texture in particular.

The present work consists of the feasibility study of a natural food jelly from three natural ingredients, namely date fruit syrup and suspension of type orange albedo powder in lemon juice (OAP)/(LJ). A central composite design (CCD), most popular of experimental designs (Akram et al. 2003), was used to report the influence of temperature and time of cooking and cooling steps on textural properties of the final

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jelly. In this context, many authors have used different factorial designs to optimize jelly processing, taking into account other independent variables: low calorie grape jelly (Sousa et al. 1997); grape juice jellies (Gaspar et al. 1998); low-calorie Blackberry (*Rubus irasuensis* Liebm.) jelly (Acosta et al. 2008); apple pomace and quince jelly (Royer et al. 2006).

The raw materials are well known for their health benefits. De Moraes Crizel et al. (2013) have recently recommended the use of fibers from byproducts of the orange juice as ingredients for their nutritional and functional characteristics. In this context, Youssef and Mousa (2012) have already fortified wheat biscuits with 10 % of orange and lemon peel powders, whereas other authors have evaluated the lemon fruit as a fundamental part for a healthy diet (González-Molina et al. 2010). Al-Shahib and Marshall (2003) advocated date fruit as an ideal food for its numerous phytochemicals, whereas Wan Ismail and Mohd Radzi (2013) have evocated its health potentials, including hepatoprotective and neuroprotective properties.

Material and methods

Date fruit syrup

The native dried date (*Phoenix dactylifera* L.) (var. *Mech-Degla*) fruits cultivated in the Algerian Sahara (Tolga region), were bought in October–November 2012 from a merchant of Tolga city and kept at 4 °C until use.

At first, according to the procedure adapted from Chaira et al. (2007), the juice was obtained by simple diffusion at 80 °C for 90 min with date/water ratio of 1/3 (w/v). Then, the mixture was pressed through a synthetic canvas and finally centrifuged at 8,000 rpm at 20 °C for 20 min. In a second time, the clarified juice was concentrated by following the protocol described by Al-Farsi (2003). The obtained syrup was the unique source of sugars used presently for the jelly preparation.

OAP/LJ suspension

To begin, the OAP from Algerian orange (var. *Thomson Navel*) was obtained by hot drying at 50 °C, followed by grinding and sieving with mesh size of 125 µm to obtain small particles according to the classification of Genovese et al. (2010). The obtained powder was used as a gelling agent because of its high pectin content. In fact, Thakur et al. (1997) showed that the orange peel is richer in pectin than the other citrus fruits and whole apple, whereas Liu et al. (2006) found 73 % of pectin in albedo (dry basis, db).

The preparation of the suspension consisted of mixing OAP (5 %) and fresh LJ (95 %) at 50 °C for 30 min using a

heating water bath with magnetic stirrer. So, the lemon juice can be considered here as the extraction solvent of pectins, a regulator of pH and source of vitamin C. It must be mentioned that Masmoudi et al. (2008) have extracted pectin from lemon using date juice acidified by citric acid as solvent, whereas Yapó (2009) revealed that natural lemon juice as extraction solvent allowed to obtain pectins with good gelling power. Furthermore, Borges et al. (2011) have investigated the quality of various jellies from extracts of banana peel, known to be rich in pectin substances.

Concerning the functional properties, Osfor et al. (2013) have recently demonstrated the hypocholesterolemic and hypoglycemic power of the orange albedo on laboratory animals and consequently recommended its use as an ingredient in the formulation of new functional foods. Previously, Lario et al. (2004) developed from lemon peel a dietary fiber-rich powder. Finally, Kang et al. (2006) have already underlined the opportunity to use citrus peel powder as a functional component in the food processing.

Preparation of the jelly

As far as we know, few authors have investigated date fruit-based jellies. Yousif et al. (1990) and Masmoudi et al. (2010) have analyzed the process of a jelly elaboration using date juice. However, these authors added white sugar and purified pectin as sweetening and gelling agent respectively.

In addition to the specification of factor levels, the composition of the initial mixture intended for gelation was predetermined according to preliminary tests.

Date fruit syrup-based natural jelly was prepared on laboratory scale by cooking the mixture of 80 % (w/w) date syrup and 20 % (w/w) OAP/LJ suspension. The cooking process aimed to heat the initial fruit mixture in thermostated oil bath at different temperatures and for different times, without pre-setting the final temperature. The optimization of jelly making was performed by applying RSM.

Quality criteria analysis

The textural properties of the jelly samples were determined by penetration of a 25 mm-diameter cylindrical probe using a texture analyzer (TA PLUS model, single column / LLOYD INSTRUMENTS, copyright AMETEK inc.2007) with the assistance of the NEXYGEN software. Prior to measurement, following parameters were set in advance: penetration depth= 5 mm, the compression speed 1=40 mm / min, speed back 1= 20 mm / min, retention time=1 s, speed compression 2= 40 mm / min and speed back 2=20 mm / min.

The vitamin C content was determined on 2 g of final jelly according to the HPLC procedure recommended by Chaira et al. (2007). The operatory conditions consisted of a stationary phase, column of type LiChrosorb Diol (5 µm), column

prefilled RT 250–4. (E. Merck Darmstadt. F. R. Germany) in isocratic mode and the mobile phase was the metaphosphoric acid (pH 2.2) at 0.5 ml min^{-1} flow-rate. The detection was performed by a UV detector at a wavelength of 254 nm at room temperature.

The sugar content (sucrose, glucose, and fructose) was also given by the HPLC, according to Coutte et al. (2009). The Aminex[®]HPX-87p column, $300 \times 7.8 \text{ mm}$ (Bio-Rad) (for separation) and a refractometer (for the detection) were used. The elution was carried out with degassed ultra-pure water as mobile phase, at a flow rate of 0.6 ml min^{-1} in isocratic mode at a column temperature of 70°C .

The FT-IR spectra of different samples (OAP, date syrup and three jelly types) were obtained using a spectrophotometer IR Shimadzu (VECTOR 22 BRUKER) in the 400 and $4,000 \text{ cm}^{-1}$ gap and potassium bromide disks. The three jellies presented the same qualitative and quantitative composition as previously indicated but different textural properties (liquid, hard, and conform to standards) obtained at different cooking conditions. This section allows, before all, to see whether the native pectin molecule, presumed to be the principal gelling substance, undergoes modifications during the cooking process.

The color parameters in the CIELab system (colorimeter Minolta Co. Ltd., Osaka/ Japan) of the fruit mass during the cooking process at different temperatures ($80, 105, 130$ and 155°C) were monitored through the direct measurement of the lightness (L^*), varying from ~ 0 (black) to 100 (white), and a^*/b^* ratio calculation from a^* (redness or greenness) and b^* (yellowness or blueness) parameters. Both L^* and a^*/b^* criteria were used here as indicator of browning index as already suggested by Koca et al. (2003) and Helyes and Pék (2006), respectively.

Experimental design

Bearing in mind that the textural properties and sugar content being the principal sensory features of jellies (Javanmard and Endan 2010), four dependent variables were considered: hardness, stickiness, sugars (saccharose, glucose and fructose) and vitamin C content. The independent variables were : temperature of cooking (X_1), corresponding to that of the thermostated oil bath; cooking time (X_2) applied for heating the initial fruit mixture in the oil bath, from ambient temperature without fixing however the final temperature; °Brix (X_3) of date syrup; and temperature (X_4) of the cooling step following the cooking process. Also, for both responses hardness and stickiness, results were carried out after three different times ($2, 4$ and 24 h) of cooling step, whereas the other responses were measured after 24 h . Although many works were devoted to the jelly preparation, the simultaneous influence of temperature and

time of both cooking and cooling steps on the jellification is, to our knowledge, still non-existent. Most of authors heated the initial fruit mixture to the boiling state, with or without specifying the applied temperature: 108°C /date fruit (Yousif et al. 1990); $100\text{--}112^\circ\text{C}$ /orange (Zakari et al. 2009); cooking until 65% -°Brix/quince (*Cydonia oblonga* Mill.) (Sharma et al. 2011). Parsayee et al. (2013) have prepared a low calorie sour cherry jam by heating the ingredients at 70°C . In all cases, the authors failed to specify the time spent for heating the mixture from room temperature to the temperature of properly so called cooking.

The total number (N) of experiments is a combination of the number of experiments related to the full factorial design (n_f), stars design (n_α) and points of center of the domain (n_0). It was computed by taking into account the characteristics of a CCD:

$N = n_f + n_\alpha + n_0$ with $n_f = 2^k = 2^4 = 16 = \text{factorial point}$, $n_\alpha = 2$, $k = 2 \times 4 = 8 = \text{axial points}$, $n_0 = 2 = \text{center points}$ and $k = \text{number of independent variables} = 4$. The levels of factors are given in Table 1, whereas the CCD model can be represented in the following general form:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j}^k \beta_{ij} X_i X_j$$

where $Y = \text{response observed}$, $x = (x_1, x_2, \dots, x_k) = \text{factors}$, $\beta_0 = \text{intercept}$, $\beta_i = \text{linear effect of the } i\text{-factor}$, $\beta_{ii} = \text{quadratic effect of the } i\text{-factor}$, and $\beta_{ij} = \text{interaction effect between } i\text{- and } j\text{- factors}$.

Statistical analysis

Data related to CCD (second-order form) were treated using MODDE software/ Umetrics, whereas color parameters are presented in histogram form as mean values $\pm$ standard deviation ($n=3$), with ANOVA at $p \leq 0.05$.

Table 1 Levels of factors of experimental design

Variables	Levels				
	$(-\alpha) -2$	-1	0	$+1$	$(+\alpha) +2$
X_1 ($^\circ\text{C}$)	50	85	120	155	190
X_2 (min)	5	10	15	20	25
X_3 (°Bx)	67.5	70	72.5	75	77.5
X_4 ($^\circ\text{C}$)	5	10	15	20	25

$\alpha = \text{distance of star points at the center of the study domain}$

X_1, X_2, X_3 and $X_4 = \text{independent variables (factors) already defined in the text and in figure captions}$

Results and discussion

Textural criteria: Hardness and stickiness (adhesiveness)

Preliminary tests allowed to highlight that the cooking temperature, corresponding here to that of thermo stated oil bath, was susceptible to implicate or not the burning, according to: i) the cooking time needed for heating initial fruit mixture from ambient temperature without presetting the final one, and ii) the temperature and time of the cooling stage following the cooking process. The CCD and RSM are useful tool to surpass this qualitative evaluation and to model the effects of these factors on jelly quality.

For all foods, texture is known as a very important multi-dimensional group of attributes (Kopjar et al. 2009), the hardness and stickiness being moreover decisive for food quality analysis (Zhou et al. 2001).

Experimental data and ANOVA results are given in Tables 2 and 3, respectively. The values of the relative

standard deviation (RSD) (<3 %) and the three criteria R^2 , R^2_{adj} and Q^2 (~1) indicated an excellent adequacy of the obtained models, except the measured stickiness after 2 h of cooling ($Q^2 < 1$). Apart from this last case, our statistical criteria values largely exceeded those advanced by several authors about different factorial designs applied for modeling various food preparations, including fruit jellies : $R^2=0.940$ and $R^2_{adj}=0.832$: effects of sweetener, low methoxyl pectin and calcium content on the overall acceptability of a tropical mixed fruit (pineapple, banana and passion fruit) jelly (Acosta et al. 2008); $R^2=0.660$ – 0.863 : textural characteristics and acceptability of apple pomace jelly (Royer et al. 2006); $R^2=0.748$, $R^2_{adj}=0.911$ and $Q^2=0.779$: hardness of traditional Chinese peanut recipe (Tan et al. 2013). All these authors have concluded on the good fit of the tested models.

From the multiple linear regression analysis, considering the most influential factors at a 95 % confidence level, the

Table 2 Experimental data

Run	X_1 (°C)	X_2 (min)	X_3 (°Bx)	X_4 (°C)	H_{2h} (N)	H_{4h} (N)	H_{24h}	St_{2h} (N)	(N) St_{4h}	St_{24h} (N)	Vit C (%) (N)	Suc (%)	Fru (%)	Glu (%)
1	85	10	70	10	0.14	0.094	0.122	0.04	−0.079	0.019	0.004019	32	14.92	15.43
2	155	10	70	10	0.143	0.31	0.652	−0.019	0.186	0.14	0.004639	29.34	17.67	18.77
3	85	20	70	10	0.065	0.112	0.293	−0.118	0.019	−0.033	0.008832	34.44	15.57	16.47
4	155	20	70	10	46.833	63	59.64	10.581	121.668	17.584	0.006923	34.33	22.9	26.83
5	85	10	75	10	0.0771	0.077	0.226	−0.096	0.012	0.198	0.004717	36.17	15.87	16.45
6	155	10	75	10	0.448	2.38	3.975	0.117	0.645	0.885	0.002785	37.97	18.93	19.50
7	85	20	75	10	0.111	0.313	1.837	−0.15	0.139	0.734	0.004108	39.83	18.12	18.39
8	155	20	75	10	74.315	78.186	150	34.854	37.078	31.996	0.00591	30.58	24.19	30.93
9	85	10	70	20	0.058	0.0844	0.122	−0.158	−0.053	−0.033	0.003692	34.17	15.65	16.49
10	155	10	70	20	0.693	3.444	3.874	0.971	0.498	1.068	0.10876	39.48	16.87	18.17
11	85	20	70	20	0.067	0.199	0.269	−0.179	0.25	0.029	0.002222	34.79	15.5	16.16
12	155	20	70	20	150	150	150	10.182	121.262	36.334	0.00484	33.12	23.74	29.09
13	85	10	75	20	0.165	0.127	0.374	0.051	1.819	0.134	0.003793	36.6	15.2	15.57
14	155	10	75	20	2.765	6.4	6.405	0.102	0.182	0.243	0.083149	37.48	18.46	19.5
15	85	20	75	20	0.17	0.408	1.808	0.49	0.265	0.452	0.004511	37.85	16.35	16.79
16	155	20	75	20	80	80	80	2.39	121.13	31.719	0.046567	31.23	24.5	30.5
17	50	15	73	15	0.092	0.096	0.107	−0.397	−0.052	0.002	0.002993	34.16	13.96	14.81
18	190	15	73	15	150	150	150	70.953	122.226	−9.748	0.033558	34.74	23.62	29.35
19	120	5	73	15	0.124	0.204	0.16	−0.013	−0.08	−0.005	0.003666	35.16	14.44	15.32
20	120	25	73	15	35.58	50.681	26.856	7.418	12.477	6.362	0.009349	43.39	18.83	20.55
21	120	15	68	15	0.088	0.829	2.28	0.087	0.222	0.527	0.003031	37.99	16.82	17.61
22	120	15	78	15	5.128	5.605	10.172	1.69	1.568	2.69	0.004157	41.49	18.75	19.56
23	120	15	73	5	6.017	0.805	3.556	1.431	1.167	1.021	0.003517	37.41	17.55	18.66
24	120	15	73	25	0.166	0.169	5.837	0.133	0.052	2.308	0.003566	35.08	16.76	17.8
25	120	15	73	15	3.482	0.316	5.026	0.683	0.518	1.452	0.004264	38.43	16.67	17.69
26	120	15	73	15	5.225	5.705	11.537	1.199	1.282	2.665	0.004844	41.52	18.94	20.27

X_1, X_2, X_3 and X_4 =independent variables (factors) already defined in the text and in figure captions; H=hardness, St=stickiness, Vit C=vitamin C, Suc=sucrose, Fru=fructose, and Glu=glucose

following mathematical models, evaluated in terms of uncoded (dimensional) variables were deduced:

$$Y_{hardness/2h} = 4.353 + 19.143X_1 + 19.096X_2 \\ + 19.099X_1X_2 + 8.275X_1^2 + 8.491X_2^2$$

$$Y_{hardness/4h} = 3.01 + 19.516X_1 + 18.296X_2 \\ + 18.215X_1X_2 + 9.462X_1^2 + 8.447X_2^2$$

$$Y_{hardness/24h} = 8.281 + 19.371X_1 + 18.188X_2 \\ + 17.768X_1X_2 + 6.209X_1^2 + 7.064X_2^2$$

$$Y_{stickiness/4h} = 0.900 + 30.411X_1 + 30.242X_2 \\ + 30.370X_1X_2 + 15.126X_1^2 + 14.876X_2^2$$

$$Y_{stickiness/24h} = 2.058 + 7.881X_1 + 7.720X_2 \\ + 7.612X_1X_2 + 2.712X_1^2 + 3.344X_2^2$$

As it could be seen, only the factors X_1 (temperature of the heat source) and X_2 (heating time) and their interactions (X_1X_2) presented considerable positive effects on both hardness and stickiness. Furthermore, the positive signs of the effects imply that any increase of one of the factors comes

along with an increase in the textural criteria of the final jelly. It is also worth mentioning the synergistic effect between heating temperature and time. These observations were consistent with the study of El-Nawawi and Heikel (1997) that proved the influence of both temperature and time of cooking on the process of pectin gelation. On the other hand, Javanmard and Endan (2010) have evocated time and temperature of the cooking process as two parameters which can affect the rheology of jam, another jelly-like food. In opposite, Royer et al. (2006) have not found any effect of cooking time on textural characteristics of jelly from apple pomace and quince. Based on previous study of Laos et al. (2006) about crystallization of sugar solutions, we think that the simultaneous increase of both hardness and stickiness during jelling process may be linked with the evolution of the saturation level of the fruit liquid mixture during heating, i.e. the water loss.

It could also be observed that the effects of factors X_1 and X_2 and their interaction (X_1X_2) were identical for a given response and duration of the cooling step. However, the impact of the latter was clear on the model constant coefficient as it doubled when cooling moved from 2 or 4 h to 24 h whatever the textural criterion.

The response surfaces illustrated in Fig. 1 (for hardness) and Fig. 2 (for stickiness) presented the same appearance whatever the cooling time: The higher the temperature or the longer the cooking time, the higher the hardness and stickiness. Such surfaces with different colored zones allow to select the cooking conditions according to the suitable texture of the final jelly.

Presently, to choose the ideal hardness, several commercial jellies (raspberry, blackberry and quince) have been analyzed as references and a value of 10 N seemed to be the most suitable. According to Royer et al. (2006), this value ranged in the interval (6–16 N) for some of jellies from apple pomace

Table 3 ANOVA results related to the second-order polynomial model

	Hardness			stickiness			Vit C	Suc	Glu	Fru
	after staying/cooling time of (h)									
	2	4	24	2	4	24	at different conditions			
Q ²	0.991	0.988	0.971	−2.443	0.996	0.963	0.721	0.849	0.852	0.632
R ²	0.999	0.997	0.996	0.864	1	0.998	0.99	0.967	0.981	0.963
R _{adj} ²	0.997	0.986	0.977	0.23	0.999	0.989	0.942	0.81	0.893	0.79
Cond. no.	8.543	8.543	8.543	8.543	8.543	8.543	8.543	8.543	8.543	8.543
Y-miss	0	0	0	0	0	0	0	0	0	0
RSD	1.017	2.236	2.823	0.635	0.701	0.756	0.007	1.269	1.121	1.067
Conf. lev.	0.95	0.95	0.95	0.95	0.95	0.95	0.95	0.95	0.95	0.95
N	18	18	18	18	18	18	18	18	18	18
DF	3	3	3	3	3	3	3	3	3	3

Vit C, Suc, Glu and Fru=dependent variables (responses) already defined (see Table 2)

Fig. 1 Response surfaces corresponding to the hardness of the jelly, after cooling time following the cooking process of 2, 4 and 24 h. The factors X_3 (°Bx of date syrup) and X_4 (temperature of the cooling stage following the cooking process) are fixed at 75 % and 20 °C, respectively

and quince. Regarding stickiness of references, the values ranged from 0.53 (quince) to 4.64 (blackberries). Therefore, to achieve this consistence in the case of our jelly, it was imperative to fix $X_1=155$ °C and $X_2=10$ min. Two hypotheses could explain these heating temperature and time values: i) the thermal inertia of the initial fruit mixture, requiring a higher temperature of the heat source, ii) the kinetic characteristics of water evaporation and jelling process, making favorable the treatment of type high temperature- low time. Since the pectin, the main component of the orange albedo, can be assumed as being the gelling agent in the used suspension (mixture of date syrup+lemon juice+

Fig. 2 Response surfaces corresponding to the stickiness of the jelly, after cooling time following the cooking process of 2, 4 and 24 h. For the levels of factors X_3 and X_4 see Fig. 1

powder of orange albedo), we believe that any change in the cooking temperature and time can imply modification on the pectin behavior through either its extraction from albedo particles, or its structural characteristics.

Since the °Brix (X_3) and the cooling temperature (X_4) were the least influential variables regarding textural properties, it was economically judicious to fix them at the most profitable levels, i.e. ~75 °Bx and 20 °C, respectively. Moreover, as it has been reported by This (1993), a jelly prepared by cooling at free area becomes firmer than jelly quickly cooled in the refrigerator since, according to the same authors, at too low temperatures, molecular movements allowing the association of gelling compound helices are slowed down.

Nutritional criteria: Vitamin C and sugars

It must be noticed that these criteria were measured after cooling time of 24 h.

The models obtained for different sugars are given below:

$$Y_{\text{Sucrose}} = 39.975 - 0.933X_2 + 0.852X_3 - 1.929X_1X_2 - 1.041X_1X_3 - 1.670X_2^2 - 0.059X_3^2$$

$$Y_{\text{Glucose}} = 18.98 + 3.908X_1 + 3.069X_2 + 2.585X_1X_2 + 1.103X_1^2 + 0.619X_2^2$$

$$Y_{\text{Fructose}} = 17.805 + 2.546X_1 + 1.837X_2 + 1.349X_1X_2 + 0.678X_1^2 + 0.077X_2^2$$

The effects of factors can also be seen in the 3D surface plots (Fig. 3)

From ANOVA analysis (Table 3), the models seemed to be satisfactory predictive, considering coefficient values of R^2 (0.967, 0.981 and 0.963), Q^2 (0.849, 0.852 and 0.632) and R^2_{adj} (0.81, 0.893 and 0.79) obtained for the concentrations of sucrose, glucose and fructose in the final product, respectively.

The concentration of sucrose was unsurprisingly favored by the °Brix (X_3) whose positive effect was opposite to that of the cooking time (X_2). At the same time, the mean effect of factors on sucrose concentration was twice higher than that exerted by cooking temperature (X_1) and cooking time on glucose and fructose. These findings highlighted the inversion of sucrose during the cooking process. The obtained inverted sugar, besides its contribution for increasing a dry matter, was also more soluble (5 g/g at 20 °C) (David 2011) than sucrose (2 g/g at 20 °C) (Grabkowski 2006) which was an advantage in terms of prevention of the undesirable phenomenon of

Fig. 3 Response surfaces related to the different sugars: Saccharose (sucrose), at cooking temperature (X_1)=155 °C, and cooling temperature (X_4)=20 °C; glucose and fructose, at °Bx of date syrup (X_3)=75 %, and cooling temperature (X_4)=20 °C

crystallization (Potter and Hotchkiss 1998) during the storage of the final jelly. Regarding the textural properties of the jelly, the heating of the fruit mixture as sugar-rich solution led to the increase of both hardness and stickiness of the final product. In fact, studying the crystallization of sucrose, fructose and glucose solutions, Laos et al. (2006) have already demonstrated that the hardness and stickiness dramatically increased with decrease of water content.

At the nutritional and physiological level, the fructose induces a less important glycemic response than sucrose (Food Safety Authority 2011).

Fig. 4 Response surface corresponding to the vitamin C content, at cooking time (X_2)=10 min, and °Brix of date syrup (X_3)=75 °Bx

As previously mentioned, the following second-order polynomial equation was found to be appropriate to predict the concentration of vitamin C in the processed jelly:

$$Y_{\text{vitamin C}} = 0.004 + 0.013X_1 - 0.01X_2 + 0.021X_4 - 0.011X_1X_2 + 0.022X_1X_4 + 0.03X_1^2 - 0.005X_2^2 - 0.011X_4^2$$

Fig. 5 FT-IR spectra of orange albedo powder **a**, date syrup **b** and jellies with different textural properties **c**

First, the vitamin C low content in the jelly must be linked with the low proportion of lemon juice in the initial fruit mixture. Moreover, several experts have considered that taking as little as 1.000 mg of liposomal vitamin C everyday can stop cataracts (Document Natural health 365). Hence, one should not neglect its behavior during gelation process.

The positive coefficient of cross term (X_1X_4) indicated a synergistic effect between cooking temperature (X_1) and cooling temperature (X_4), while factor X_1 at second order denoted quadratic relationships suggesting its crucial role in the evolution of the vitamin C content during jelly processing. At the same time, a negative sign observed for the regression coefficient (X_2) showed that an increase in the cooking time resulted in a decrease in vitamin C. The response surface plot of Fig. 4, drawn as a function of X_1 (cooking temperature) and X_4 (cooling temperature), confirmed that the highest content of vitamin C (red zone) was obtained for the highest cooking temperature and with cooling temperature between 20 and 25 °C. However, the effect of the cooking time was clearly visible too (see the negative effect of X_2 in the model), and a fast cooking process favored a better safeguard of vitamin C. This result proved the useful application of well known high temperature- shorten time treatment, generally applied in thermal processing of foods.

FT-IR analysis

We recall that this section allows, before all, to see whether the native pectin molecule, presumed to be the principal gelling substance, undergoes modifications during the cooking process. The IR spectrum of the OAP (Fig. 5a) shows important similarities with that of the native pectin communicated by Monfregola et al. (2011): band OH ($3500\text{--}3,200\text{ cm}^{-1}$), CH ($\sim 2,945\text{ cm}^{-1}$), double band C=O ($1,700\text{ cm}^{-1}$), CH₃-ester ($1,452\text{ cm}^{-1}$) and a series of vibrational bands C-O/C-C ($1,150$ and 950 cm^{-1}), contained in the glycosidic patterns and pyranone rings. For their part, Cardenas et al. (2008) and Souza et al. (2012) have revealed the C=O group, known as vibration on the purified pectin, on the nopal cactus (*Opuntia ficus indica*) (at $1,749\text{ cm}^{-1}$) and pumpkin (at $1,743\text{ cm}^{-1}$), respectively and that their authors attribute it to the non-ionized carboxylic groups. These analogies confirmed the presence of pectin in the orange albedo. On top of that, the $1,338\text{ cm}^{-1}$ band represented a typical band of the methyl group, whereas the series of bands between 1100 and 900 cm^{-1} corresponded to the C-O and C-C groups.

From IR spectrum of the date syrup (Fig. 5b), a stretching band at $1,658\text{ cm}^{-1}$ corresponding to the antisymmetric COO[−] group should be particularly noted. But more importantly,

Fig. 6 Color parameters of the elaborated jelly versus cooking time, at 80 **a**, 105 **b**, 150 **c** and 155 °C **d**

what attracted our attention was the lack of the methyl carboxylic group, thus highlighting the non-methyl characteristic of the low quantities of pectins present in the date syrup.

Globally, the typical bands of the two basic ingredients (OAP and date syrup), discussed earlier, were visible on jellies with different textures (Fig. 5c). These spectra proved that the gelation process depended not only on the initial pectin content in the raw material but also on the cooking temperature and cooking time. Moreover, it should be noted that the structure of jelly with suitable texture was partially close to that of pectin because of the two clear bands situated at 1,750 and 1,668 cm^{-1} attributed respectively to the esterified and non-esterified carboxyl groups. Besides, the weak intensity of the band at about 3,500–3,000 cm^{-1} could be considered as a successful gelling process in the case of the convenient jelly, generating hydrogen links between the pectin molecules.

Color change of the fruit mixture during the jelly processing

It is well known that the color of food is a determinant quality criterion for consumer acceptability. The variation of L^* and a^*/b^* parameter values versus cooking time at different temperatures was considered. From Fig. 6a,b,c,d, it could be easily observed that both parameters were not affected by the cooking process whatever the temperature applied. Despite the extremely low values of the a^*/b^* ratio, they are worth mentioning because they indicate the absence of the non-enzymatic browning reactions, including the formation of the hydroxymethylfurfural known to be an intermediate product of Maillard reactions. This result was confirmed by the values of the L^* parameter which can be considered as another browning indicator. In fact, the data related to the lightness (L^*) remained globally stable for the considered time intervals ($p \leq 0.05$), whatever the applied cooking temperature.

Conclusion

Based on RSM combined with CCD, the obtained results have demonstrated the feasibility of natural jelly from three Algerian raw materials, namely date syrup, lemon juice and orange albedo powder.

Among the four technological parameters considered, the cooking temperature and cooking time were found to be the most influent factors on textural properties of the final jelly. Moreover, to reach textural properties of commercial jellies, the temperature of cooking process, corresponding to that of thermostated oil bath and the heating time of the initial fruit mixture had to be fixed at 155 °C and 10 min respectively. Besides, the jelly obtained at these conditions presented FT-IR spectra partially similar to that of natural pectin.

Based on the lightness and red-to-yellow pigment ratio, the color of the processed fruit mixture seemed to be stable during the cooking process whatever the temperature applied.

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