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Preparation and characterization of syrup from native dried date (*Phoenix dactylifera* L., variety *Mech-Degla*) fruits

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Presently, three different evaporation procedures (oven heating, rotary evaporator, and lyophilization) were investigated at laboratory scale, for obtaining date (*Phoenix dactylifera* L.) syrup (DS) from date juice (DJ). The DJ was prepared by immersion of pitted date fruit (DF) pieces in pure water, according to the local traditional procedure. Compared to oven heating and lyophilization, the use of the rotavapor at 70°C seems to be more rational, since three hours of treatment are needed to reach a total of soluble solids (TSS) of about 61 Bx, against 6 and 18 h in the case of the two other cases, respectively. In addition, the DS thus obtained shows a lowest content in hydroxymethylfurfural (HMF). From the point of view of rheological behavior, the final DS is of pseudo-plastic (Bingham) type, comparable to white syrup taken here as reference. Considering the growing interest for natural products, the DS can advantageously replace commercial white syrups.

Keywords: Chemical properties, Concentration, Date juice, Date syrup, Rheological properties

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The date palm (*Phoenix dactylifera* L.) is mainly popular for its sweet fruit. Dates are in fact, a high-energy food because of their sugars. They also provide dietary fibers, vitamins, minerals, polyphenolics and antioxidants with medicinal properties[1,2].

In Algeria, the date palm cultivation is mainly located in the Saharan region with the presence of a significant varietal diversity[3]. The average production of date fruits (DF) in Algeria is about 500,000 tons per year[4]. A non-negligible proportion of this production is represented by the common varieties which are less attractive to consumers, on one hand and they are subject of extensive campaigns of replacement in favor of more profitable varieties, on the other hand. The research of new formulations from DF may be a solution that may favor the emergence of new technological process of arboriculture activity and thus contribute to safeguarding the phoenicicolous tradition.

Presently, three different evaporation procedures (oven heating, rotary evaporator, and lyophilization) were investigated at laboratory scale, for obtaining date (*Phoenix dactylifera* L.) syrup (DS) from date juice (DJ). The DJ was prepared by immersion of pitted DF pieces in pure water, according to the local traditional procedure. In this context, several works are devoted to the preparation of date syrup (DS), using other varieties and other procedures for the extraction and concentration of DJ[5-11]. Syrups are also prepared from other fruits. Thus, Sobhani *et al.*[12] have formulated and characterized a syrup from jujube (*Ziziphus jujuba* Mill.) using a rotary evaporator.

Traditionally, a date drink (nabidh) was prepared by soaking mashed up DFs in water for one night[13]. In addition to its organoleptic properties and natural character highly sought by consumers[14], the DS is of a good digestibility thanks to its fluid texture, which may be appropriate for certain people (patients, infants). The resulting syrup may be also used as carrier for herbal extracts. Alanazi has already experienced the use of DS as a binder in pharmaceutical tablets[15].

Methodology

The native dried DF variety *Mech-Degla* (Fig. 1) used in this study is widespread in the palm groves of South-eastern Algeria. The fruits were from the region of Biskra and commercialized in Algiers. Biskra is a commune in the northeast of the Algerian Sahara, capital of the wilaya (region) of Biskra, located about 400 km south-east of Algiers (Fig. 2)[16].

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Materials and Methods

Date fruit juice

The preparation procedure of DJ was adapted from that traditionally applied in Algerian Sahara as rapported by Barreveld[13]. DF were first washed, removing excess of water with absorbing paper and each fruit was divided into small pieces of approximately equal sizes (5×4×2 mm). It is important to note that the too small suspended particles, including colloids, are difficult to separate from the liquid phase and are often undesirable[17].

The DJ was prepared by immersion of date pieces in hot water at 50°C, with dates / water (weight/weight) ratio = 1/6, applying regular agitation. The extraction process was stopped when the level of total soluble solids (TSS) of the liquid phase was stabilized, whereupon the extract is recovered by filtration. Immersion was repeated three times, and all extracts were combined in a sealed glass jar and stored in the freezer until the evaporation-concentration. The extraction rate (ER) of soluble solids was calculated (but not presented here) according to the following equation[9]:

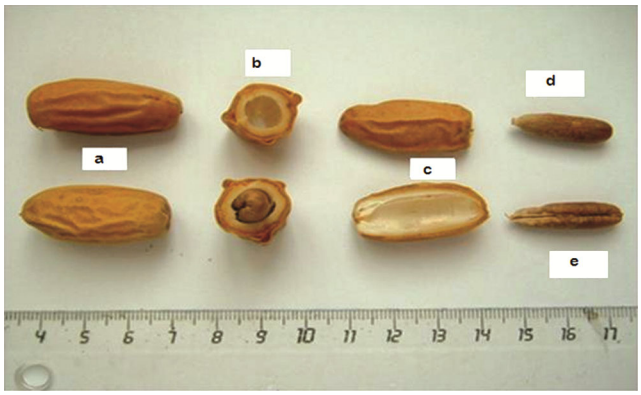

Fig. 1 — Photographs of *Mech-Degla* native dried date fruit (DF): a-Whole fruit, b-Transversal cut of the fruit, c-Longitudinal cut, d-Date seed (dorsal view), and e-Date seed (ventral view)

Fig. 2 — Map showing the Biskra region in Algeria[16]

$$ER\ (\%) = m_{ext} \times TSS_{ext}/m_0$$

Where, m_{ext} and m_0 = weight of obtained extract and initial date flesh, respectively.

The final DJ was also characterized by two other indices: 1) Brown index (BI) expressed through the optical density (OD) at 420 nm, and 2) pH.

Preparation of syrup

The objective of the concentration by evaporation is to remove 50-65% of the free water contained in the DJ. Three types of syrups (S) are obtained according to the method applied: 1) OS: by concentration in an oven at 70°C (Type Memmer) at atmospheric pressure, 2) RS: by concentration in a rotary vacuum evaporator(Type IKA RV 10 digital) at 70°C, and 3) LS: by lyophilization at - 45°C (apparatus of type TELSTAR cryodos).

Physicochemical characterization of date fruit flesh and syrups

Some indices of DF flesh and obtained syrup were determined using the same procedures: TSS by refractometry (see above), water content (by drying at 70°C to constant weight of homogenized sample), pH (pH-meter), titratable acidity (dosage with a solution of 0.1 N NaOH), ash (calcination in a muffle furnace at 550°C), minerals (atomic absorption), total sugars[18], reducing sugars[19], pectin and cellulose[20], protein (Kjeldahl method), polyphenols[21], hydroxyl-methylfurfural (HMF)[22], reducing power[23] as indicator of the antioxidant activity (AA).

The color of DS was measured in CIELab system according to three parameters: L^* (0 = black and 100 = white), a^* (-60 = green and + 60 = red) and b^* (-60 = blue and +60 = yellow) using chroma (reflectance) meter (CM- 2500d Minolta Japan).

Rheological behavior of syrups

Rheological tests on different DS are performed using a rotational viscometer VT550 (THERMO-HAAKE) with concentric cylinder geometry MV-DIN of 42 mm diameter, at shear rates ranging between 0 and 200 s^{-1}. Flow curves were registered by means of micro-software Rheowin.

Statistics

Except for extraction rate, ash and minerals, the other obtained results are expressed as the mean of three measurements ± standard deviation and the multiple comparison test of Duncan for the significance level of 5% was applied through analysis of variance (ANOVA) using XLSTAT 2007 software.

Results and Discussion

DJ and DS characterization

The DJ obtained at 50°C presented a lower BI value (Fig. 3a) and a pH value close to that of the DF (Fig. 3b and Table 1) and it was consequently chosen to undergo the concentration process.

The value of TSS depends on the concentration procedure (Table 1). Globally, the three tested laboratory-scale evaporators allowed to reach a TSS value comparable to that (63 °Bx) reported by Al-Farsi *et al*.[5] about the *Shahal* date variety of Oman. However, in addition to water removal, the evaporation, as a thermal operation unit may imply some other changes in chemical composition of processed juice.

The pectin content of the three final products are low in comparison with that of the initial fruit flesh. It is well known that a high content of pectin promotes a cloudy appearance of juices which can affect the sensory quality of the processed syrups, making the depectinisation / clarification, unavoidable[24].

Apart from other macro and micronutrients, selenium deserves special attention because of its antioxidant properties widely documented[25-27]. Results indicated that over 90% of the DF selenium passes into syrup, making the final product interesting source of antioxidants (Table 1). The presence of HMF illustrates both the production of Maillard compounds and dehydration of sugars during the heat treatment[28].

Among the three final products, RS presents the lowest HMF content but it is not statistically different with LS ($p>0.05$). These results can be explained by the favorable combined effect of low temperature and vacuum, two technological parameters that determine

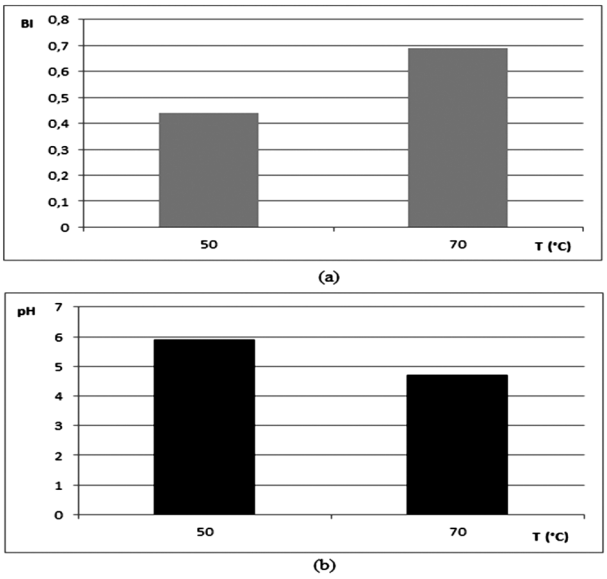

Fig. 3 — Effect of the temperature on the brown index (BI) (a) and pH (b) of date juice (DJ) used for syrup preparation

Table 1 — Physicochemical characterization of date flesh and its syrups (g/100 g of wet weight (ww), unless otherwise stated)

Parameter	DF flesh	OS	RS	LS
Water content	14.75 ± 0.20^{d}	35.3 ± 0.10^{c}	36.75 ± 0.08^{b}	37.07 ± 0.09^{a}
pH	5.49 ± 0.14^{a}	4.80 ± 0.02^{b}	5.14 ± 0.15^{a,b}	4.93 ± 0.37
Titratable acidity	0.19 ± 0,03^{b}	0.30 ± 0,01^{a}	0.20 ± 0.01^{b}	0.22 ± 0.01^{b}
TSS (°Bx)	72.11 ± 1.08^{a}	62,5 ± 0.00^{b}	61.0 ± 0.00^{c}	60.5 ± 0.00^{d}
Total sugars	74.89 ± 2,36^{a}	58.57 ± 0,70^{b}	57.97 ± 0,15 ^{b}	57.72 ± 0.14 ^{b}
Reducing sugars	25.36 ± 0.80^{d}	47.43 ± 0.67^{a}	42.72 ± 1.45^{b}	40.29 ± 0.66^{c}
Pectins	0.23 ± 0.12^{a}	0.19 ± 0.03 ^{a}	0.16 ± 0.05^{a}	0.17 ± 0.01^{a}
Proteins	2.33 ± 0.04^{a}	1.97 ± 0.04^{b}	1.59 ± 0.01^{c}	2.03 ± 0.08^{b}
Ash	1.42	1.07	1.02	1.21
Potassium*	528.24	430.20	422.54	445.11
Calcium*	59.98	43.10	52.88	46.17
Magnesium*	38.82	22.40	24.48	24.25
Sodium*	45.33	34.15	37.87	39.47
Zinc*	0.41	0.30	0.29	0.23
Copper*	0.24	traces	traces	traces
Selenium*	2.52	2.32	2.48	2.44
H.M.F*	-	0.77 ± 0.28^{a}	0.15 ± 0.08^{b}	0.35 ± 0.22 $^{a, b}$
Antioxidant activity**	1350 ± 0,04^{a}	151.10 ± 0,52^{c}	154.38 ± 1.64^{b}	153.10 ± 0.48^{b}
Total polyphenols ***	318.74 ± 0.83^{a}	66.56 ± 1.50^{c}	70.04 ± 0.47^{b}	67.13 ± 1.50^{c}

DF = date fruit; OS, RS and LS = syrups obtained by oven heating, rotary evaporation and lyophilization, respectively.
The values with same letters on the same line are not statistically different ($p\leq0.05$); ***:** mg / 100 g (WM); **: mg EAA (equivalent ascorbic acid) /100 g (WM); ***: mg EAG (equivalent gallic acid) /100 g (WM).

the duration of concentration process (Fig. 4). The values found are also lower than those (30-120 mg / 100 g w.w) revealed by El-Nagga and Abd-El Taweb for syrups prepared by three extraction techniques (water bath, rotary evaporator and micro -wave) and concentrated to 72 °Bx with two methods (rotary evaporator and microwave at 70°C)[9]. This difference can be attributed to the method of concentration and duration of treatment.

The polyphenol content of the RS is higher than those of OS and LS. But the three concentrates are less rich in polyphenols, compared to the initial date fruits. The oxidation process during the extraction may explain the loss of phenolic substances. According to Mehinagic *et al.*[29], the steps that deconstruct a product may imply the change of the phenolic composition. In addition, a long time of extraction can involve oxidation of these compounds[30]. It is easy to observe that RS and LS are similar from viewpoint of antioxidant activity, displaying a great value compared with OS. For the three concentrates, the antioxidant potential is lower than that of the date flesh, but higher than that (15.1-29.5 mg EAA / 100 g) reported for various Indian honeys[31]. This comparison is not fortuitous since the date syrup has enough similarities with honey, especially on the appearance, texture and sugar content level. Thus, characterizing blue agave syrup (*Agave tequilana* Weber var. azul), Mellado-Mogica and Lopez-Perez[32] refer to honey for comparing their results. On the other hand, the industrial production of date honey was already mentioned in the literature[6,33].

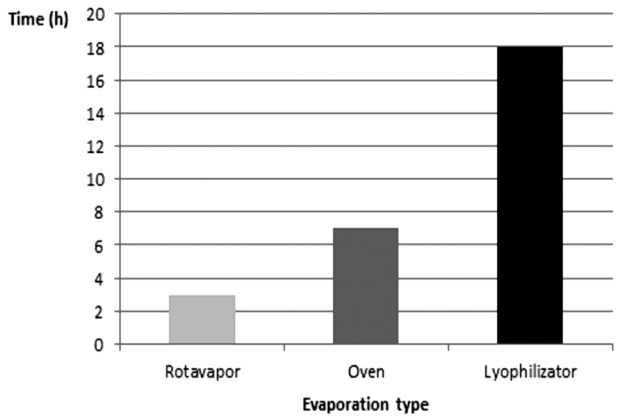

Fig. 4 — Evaporation time needed to reach a final TSS value of about 60 °Bx, according to evaporation procedure. The different lowercase letters upper columns indicate the presence of significant difference (p≤0.05) between values.

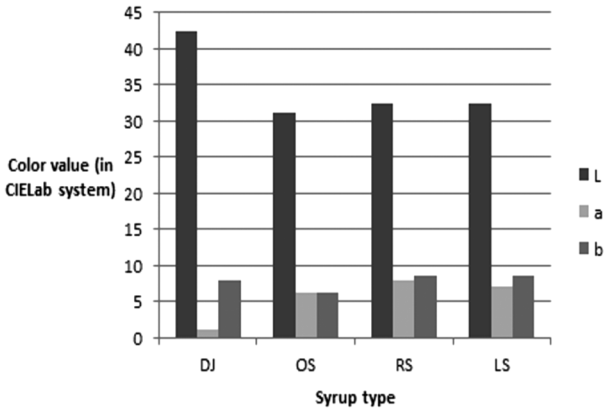

Fig. 5 — Values of color parameters (L*, a*, b*) versus date syrup (DS) type, obtained from date juice (DJ) by oven heating (OS), rotary evaporation (RS) and lyophilization (LS)

It should be recalled here that the color is a very important attribute that governs the acceptability of food by the consumer. According to the histogram of Figure 5, the values of chromatic coordinates (a^*) and (b^*) are found positive, indicating that the color of DS is in yellow-red region of the CIELab space. These observations are confirmed by the luminance (L^*) of the three syrup types and which is lower than that of the original juice. The values of these three parameters are in the ranges (L^* = 40-60, a^* = 0-10 and b^* = 10-30) highlighted by Kadar *et al.*[34] about acacia honey, sunflower and lime. Also, the whiteness values (L*) of DS are in the range (25-41) found by Batu and Arslan[35] for Turkish delight (lokum) enriched with black grape and sour cherry syrups, which suggests the possibility to use such combinations with DS.

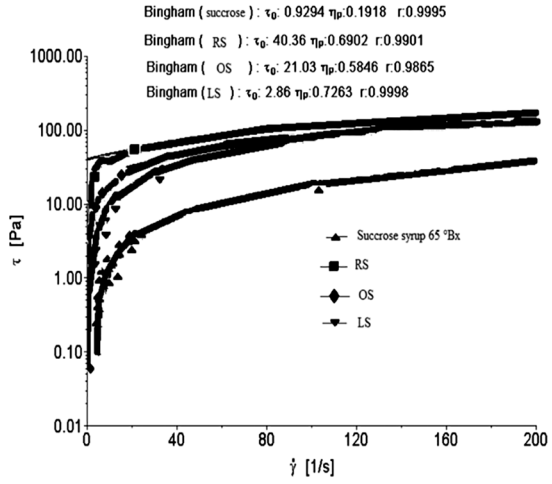

Fig. 6 — Flow curves corresponding to sucrose syrup and date syrups (DS) obtained by means of rotary evaporation (RS), oven heating (OS) and lyophilization (LS)

Rheological analysis of syrups

In the food industry, several types of sugars are used as thickening agents for modifying the rheological properties of foods[36]. The flow curves of prepared DS and that of white syrup (reference) (Fig. 6) allowed to say that the finished products display a rheological behavior of an ideal fluid plastic which follows the Bingham model described by the following well-known equation:

$$\tau = \tau_0 + \eta_P \dot{\gamma}$$

where, τ_0: yield point ; η_P: plastic viscosity and $\dot{\gamma}$: shear rate.

The ideal plastic flow (or Bingham flow) is associated with the presence of flocculated particles in the concentrated suspensions. This behavior has already been revealed by Scher[37] and Feys *et al*.[38] concerning the melted chocolate and honey, respectively. For their part, Pongsawatmanit *et al*.[39] have revealed that, in comparison with the Herschel-Bulkley models and Bingham, the power law is most appropriate on the rheological behavior of blueberry syrup obtained by adding sugar syrups and other purified ingredients. Applying three models (Power Law, Casson and Herschel-Bulkley models) on chocolate syrups, Ramli and Ying[40] have found that the Herschel-Bulkley model is most appropriate. But these authors have chosen the power law for further investigations, based on the fact that the yield stress is incompatible with chocolate syrup. All these differences in flow properties indicate the complexity of food matrices which may be the result of the composition of the viscous mixture.

Conclusions

The concentration of DJ under partial vacuum in a rotary evaporator at 70°C is more suitable, compared to both other methods applied (oven heating and lyophilization). The obtained DS recalls the honey by the appearance (color) and melted chocolate by the flow properties (ideal fluid plastic).

The DS could be of various uses. In particular, it can play advantageously the role of white sugar substitute in several food preparations. In this context, it would be desirable to give further consideration to the issue, especially, the following aspects: 1) the use of the syrup as carrier of plant extracts, and 2) recovery of extraction residue. Such valorization way is susceptible to add a value to native dried DF, which might contribute to safeguarding the oasis ecosystem, and beyond, the local traditional knowledge.

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Conflict of Interest

Authors declare no conflict of interest.

Authors' Contributions

H M, A B and Kh B designed and performed the experiments. They interpreted the results S B supervised the work and wrote the manuscript

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