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Comparative study between carob and date syrups

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Abstract

Date and carob syrups are traditional foods produced and consumed in several countries, including Tunisia. Each syrup is characterized by its interesting nutritional and bioactive composition. Several studies have been carried out on the production and characterization of each syrup separately, but this study tends to compare the aromatic, physico-chemical, nutritional and functional properties of commercial carob and date syrups. The results showed a comparable aromatic composition whose major compounds are terpenes (carvone and menthol), a difference in color since carob syrup was characterized by a darker brown color, a nutritional composition rich in sugars and a difference in functional properties since carob syrup had greater emulsifying capacity and antioxidant activity. Thus, the characteristics of carob and date syrups encourage their use in the food industry according to the appropriate product.

1. INTRODUCTION

Syrup is a concentrated juice generally produced from various high-sugar fruits to preserve seasonal fruits. It is a well-known traditional food in many Mediterranean countries under the name "Rub" in Tunisia, "Pekmez" in Turkey and "Débès" in Lebanon (Dhaouadi et al. 2014; Tounsi and Kechaou 2017). The manufacture of syrup is done with different techniques taking into account the species of fruit used as raw materials, but it mainly goes through two stages: the extraction and the concentration of the juice (Sengül et al. 2007). Concentrating juice as a heat treatment could promote chemical reactions during syrup processing and storage, including the non-enzymatic browning reactions (caramelization and Maillard reaction). Caramelization occurs by the breakdown of sugar at high temperature, while Maillard reaction takes place between amino acids and reducing sugars. Browning reactions could improve the sensory characteristics (color, aroma and flavor) of certain food products such as coffee. They also lead to the loss of certain nutrients (e.g. amino acids) and the formation of other compounds such as brown pigment (melanoidins) and 5-hydroxymethylfurfural (HMF) (Vaikousi, Koutsoumanis, and Biliaderis 2008; Batu 2010)

Among the most consumed fruit syrups are carob syrup and date syrup.

Carob syrup is produced from the fruits of the carob tree (*Ceratonia siliqua* L.) which is a typical tree of the Mediterranean basin (Tounsi and Kechaou 2017). It is locally known as "Rub El Kharroub". The production and marketing of carob syrup is widespread in Turkey. It is an energy food rich in sugars (mainly sucrose, glucose and fructose) and minerals (in particular K, Ca, Mg, Na, P and Fe). In addition to its nutritional compounds, it also contains bioactive compounds (especially polyphenols and products of browning reactions), which justifies its use in folk medicine as a natural remedy to treat certain diseases such as diarrhea (Tounsi and Kechaou 2019). It could be also consumed

directly for breakfast, or mixed with other food products such as yogurt (Karaca, Saydam, and Guven 2012) and sesame paste (Tounsi et al. 2019).
Date syrup is produced from the fruits of the date palm (*Phoenix dactylifera* L.) which is a typical tree of desert regions (arid and semi-arid regions). It is known locally as "Rub Al Tamr". The production and marketing of date syrup is widespread in Libya, Iraq and Tunisia (Abbès et al. 2011). Date syrup is considered as a source of calories since it is rich in sugars, a source of minerals and a source of phenolic compounds endowed with several biological activities (Abbès et al. 2011; Abbès, Kchaou, et al. 2013; Al-Hooti et al 2002; Al-Farsi et al 2007). It could be directly consumed or used as an ingredient in food formulations such as dairy products (Jridi et al. 2015) and sesame paste (Razavi, Habibi Najafi, and Alaee 2007).
Several researchs have focused on the preparation and characterization of carob syrup (Sengül et al. 2007; Tounsi et al. 2017; Tetik et al. 2011; Toker et al. 2013; Dhaouadi et al. 2014; Tounsi, Ghazala, and Kechaou 2020) and date syrup (Batu 2010; Abbès, Kchaou, et al. 2013; Abbès et al. 2011; Abbès, Besbes, et al. 2013; Al-Hooti et al. 2002; Al-Farsi et al 2007), but to our knowledge, there are no scientific studies comparing these two syrups. Therefore, the objective of this work is to compare the aromatic, physico-chemical, nutritional and functional properties of commercial carob and date syrups.

2. MATERIAL AND METHODS

2.1. Samples

Carob and date syrups were purchased from the local market in Sfax. Table 1 presents the manufacturing process of each syrup according to its producer.

2.2. Aromatic analysis

Aromatic analysis was carried out in two successive steps: volatile compounds extraction and then chromatographic analysis.
The extraction was performed with hexane using the Soxhlet system according to Orphanides et al. (2011) with slight modifications.
The obtained extracts were then analyzed by a GC system (HP model, MSD6980, Agilent-Technologies, CA, USA), equipped with a HP-MS capillary column (30 m length; 0.25 mm width; 0.25 μm film thickness) and coupled to a mass selective detector (ionization energy of 70 eV; mass range at 50–550 m/z).

Table 1. General process for the manufacture of commercial syrups

Carob syrup	Date syrup
1- washing	1- washing and pitting
2- fragmentation	2- grinding
3- water immersion and cooking	3- maceration in water (solid/liquid ratio equal to 1/3) for 1 hour
4- sieve filtration	4- manual filtration
5- concentration by cooking until thickening	5- concentration by cooking up to 70°Brix
6- cooling and conditioning	6- filtration and hot conditioning

2.3. Physico-chemical analysis

Soluble solids content (°Brix) was measured using a refractometer (OpTech, Germany) previously calibrated with distilled water.
Water activity (aw) was determined by a aw-meter apparatus (Hygropalm, Rotronic, France).
Viscosity was measured at 25 °C with a viscosimeter (HA, Brookfield, USA).
pH was measured using a pH meter (METTLER TOLEDO MP 220, Switzerland).
Color was measured by a colorimeter (Konica Minolta Chroma Meter CR 400-410, Japan) and expressed by L* a* b*coordinates. Results were expressed by brown color intensity, indicating the degree of brown color as a result of concentration process, and calculated by equations according to Maskan (2001).
Acidity was determined according to the titration method described by AFNOR (1974).
Browning index was determined by measuring the absorbance at 420 nm of diluted syrup up to 4 °Brix with distilled water (Abbès et al. 2013).
For HMF determination, molasses samples were firstly clarified following the method described by Toker et al. (2013) and then assayed to quantify the HMF concentration according to Cohen et al. (1998).

2.4. Nutritional analysis

The determination of soluble sugars was carried out by the sulfuric phenol method at 490 nm (Dubois et al. 1956).
The determination of total fibers was performed by the enzymatic method described by Prosky et al. (1988) with slight modifications.
The determination of phenolic compounds was carried out according to the Folin-Ciocalteu method (Singleton and Rossi 1965).

The protein, lipid and mineral contents were determined according to the methods described by AOAC (2000). The dosage of minerals was determined by incineration in a muffle furnace at 550°C until complete combustion of the organic matter. The protein assay was carried out by the standard Kjeldahl method. The dosage of lipids was performed by the Soxhlet method.

The determination of the mineral composition was made following an acid attack of the ash with hydrochloric acid (2N) according to Chew et al. (2011). Calcium, potassium, sodium, magnesium, iron, copper, zinc and manganese were analyzed separately using an atomic absorption spectrophotometer, while phosphorus was analyzed by colorimetric assay.

2.5. Functional analysis

The antioxidant activity of the syrups was evaluated by the phosphomolydenum method described by Prieto et al. (1999).

The emulsifying capacity of syrups was determined according to the method reported by Ogunwolu et al. (2009) with slight modifications.

2.6. Statistical analysis

Statistical analysis was performed using SPSS software (Version 20.0) to determine significant differences between the samples at a 95% confidence interval ($P < 0.05$). Student test (T test) was used to compare the two samples.

3. RESULTS AND DISCUSSION

3.1. Aromatic characterization

Table 2 presents the aromatic profile of the hexanoic extracts of the two syrups analyzed by GC-MS.

Chromatographic analysis of the studied syrups revealed the presence of several volatile compounds belonging to different chemical families (terpenes, siloxanes, heterocycles and fatty acids) with different percentages that vary from one sample to another depending on the raw material and/or the manufacturing process.

Indeed, terpenes, especially carvone and menthol, are the major compounds responsible for the aromatic characteristics of the studied syrups (carob syrup and date syrup). The presence of siloxanes in the commercial syrups is also noted. Siloxanes are synthetic chemicals that could be food additives, namely polydimethylsiloxane (E900), also known by silicone oil as an anti-foaming agent (Codex Alimentarius 2013).

Tounsi et al. (2020) also reported the aromatic composition of traditional carob syrup which is characterized by the dominance of fatty acids, especially palmitic acid, and terpenes, especially carvone.

According to the literature, some identified aromatic compounds, in particular, fatty acids (palmitic acid), terpenes (menthol and carvone)

Table 2. Aromatic profile of carob and date syrups

Compounds[a] (chemical formula)	**RT[b] (min)**	**Content[c] (%)**	
		Carob	**Date**
Terpenes		**28.36**	**31.35**
Carvone ($C_{10}H_{14}O$)	11.45	14.22	14.31
Menthol ($C_{10}H_{20}O$)	9.75	14.14	14.9
Limonene ($C_{10}H_{16}$)	6.51		2.14
Siloxanes		**22.37**	**13.36**
Tetradecamethyl-cycloheptasiloxane ($C_{14}H_{42}O_7Si_7$)	16.74	4.61	6
Eicosamethyl-cyclodecasiloxane ($C_{20}H_{60}O_{10}Si_{10}$)	25.38	7.64	6.59
Hexadecamethyl-cyclooctasiloxane ($C_{16}H_{48}O_8Si_8$)	20.00	9.22	
Hexadecamethyl-heptasiloxane ($C_{16}H_{48}O_6Si_7$)	18.36	0.9	0.77
Heterocycles		**8.30**	**15.90**
4- (3,4-dimethoxybenzylidene)-1-(4-nitrophenyl)-3-phenyl-2-	22.84	5.86	15.23
Fer, monocarbonyl- (acide 1,3-butadiène-1,4 dicarbonique, ester	37.04	2.44	0.67
Fatty acids		**1.48**	**2.71**
Palmitic acid ($C_{16}H_{32}O_2$)	24.47	1.48	
Caproic acid ($C_6H_{12}O_2$)	7.48		1.43
Oleic acid ($C_{18}H_{34}O_2$)	28.95		1.28

a : Identification of components based on GC–MS Wiely 7.1 version library ;
b : Retention time ;
c : Percentages were obtained from electronic integration measurements using a selective mass detector.

and heterocycles ($C_{24}H_{19}N_3O_5$), also possess interesting medicinal properties such as antibacterial, antifungal, antioxidant, anti-inflammatory, antiviral, antitumor, activities (Harada et al. 2002; Djilani and Dicko 2012; Raj et al. 2013).

3.2. Physico-chemical characterization

The physico-chemical properties of carob and date syrups are presented in Table 3.

By comparing the studied syrups, the statistical analysis shows significant differences ($P < 0.05$) in terms of °Brix, viscosity and color. Indeed, date syrup was characterized by a lower soluble matter content (°Brix) and therefore a lower viscosity. This result could be attributed either to the raw material or to the manufacturing process. Carob syrup was characterized by a darker brown color mainly due to the dark brown pigments characteristic of carob pods.

Browning index and HMF content are often determined to assess the quality of heat-treated foods, including syrups or concentrated fruit juices. The browning index (absorbance at 420 nm) is generally used as a quick and easy method for detecting brown pigments, while the concentration of HMF is widely used as an indicator of excessive heating of foods containing sugars (Vaikousi, Koutsoumanis, and Biliaderis 2008). As shown in Table 3, the two commercial syrups have statistically the same values for the browning index (1.9) and the HMF content (33 mg/100 g). A previous study by Abbès, Besbes, et al. (2013) also determined these two parameters in date syrup and reported different values. Another study by Tounsi, Ghazala, and Kechaou (2020) found similar results for traditional carob syrup. These two studies indicated that the HMF compound, a product of non-enzymatic browning reactions, possesses on the one hand beneficial biological effects for health (such as antioxidant, antimicrobial and anticancer effects) and on the other hand cytotoxic effects harmful to health (such as eye and respiratory tract irritation) at high concentrations. Kus, Gogus, and Eren (2005) determined the concentration of HMF in several concentrated food products and reported that fruit syrups had the highest values ranging from 1.3 to 350 mg/100 g. This variability could be attributed to several factors. Firstly, the composition of the fruits, in particular the proteins and sugars which are substrates for non-enzymatic browning reactions, as well as the phenolic compounds which might sometimes inhibit the Maillard reaction. In addition, the type effect of sugars, for example fructose, is about 40 times more reactive than glucose as a precursor to the formation of HMF. Furthermore, the formation of HMF in heat-treated foods is also associated with the temperature and duration of the treatment, the water activity and the pH of the products (Bozkurt et al. 1999 ; Kus et al. 2005 ; Toker et al. 2013).

Table 3. Physico-chemical properties of carob and date syrups

Samples	Carob syrup	Date syrup
°Brix	75.00 ± 0.02 b	70.80 ± 0.01 a
a_w	0.77 ± 0.01 a	0.77 ± 0.00 a
Viscosity (1)	1284.00 ± 62.23 b	428.00 ± 5.66 a
BCI	2.31 ± 0.04 b	0.91 ± 0.00 a
pH	4.44 ± 0.00 a	4.25 ± 0.00 a
Acidity (2)	0.75 ± 0.12 a	0.71 ± 0.17 a
Browning index	1.90 ± 0.00 a	1.93 ± 0.04 a
HMF (3)	32.98 ± 1.67 a	33.17 ± 1.08 a

BCI : Brown color intensity (1) mPa.s (2) g citric acid/100 g syrup (3) mg/100 g syrup

3.3. Nutritional characterization

The nutritional composition of carob and date suryps is illutrated in Table 4.

Both syrups are characterized by the dominance of soluble sugars (51%) and the absence of lipids (0%). These results are confirmed by Abbès et al. (2011) for date syrup and by Tounsi et al. (2020) for carob syrup.

By comparing the two syrups, the statistical analysis shows significant differences ($P < 0.05$) in terms of fiber, protein, mineral and phenolic contents.

Carob syrup is characterized by a higher dry matter content (fibers, proteins and minerals), and therefore a higher viscosity. This material either comes from the raw material or escapes during filtration.

The commercial carob syrup contains a higher content of phenolic compounds (1674.27 ± 23.84 mg gallic acid/100 g), comparable to that found by Tounsi et al. (2020) for traditional carob syrup (1597.75 ± 46.06 mg gallic acid/ 100 g). A previous study carried out by Abbès et al. (2011) also reported comparable values for date syrup.

Regarding the mineral composition, both carob and date syrups contain the same mineral elements with different contents, of which

Table 4. Nutritional composition of carob and date syrups

Samples	Carob syrup	Date syrup
Soluble sugars (%)	50.81 ± 2.20 a	50.62 ± 1.73 a
Total fibers (%)	30.09 ± 0.39 b	15.57 ± 2.06 a
Proteins (%)	3.60 ± 0.00 b	1.36 ± 0.02 a
Lipids (%)	0	0
Polyphenols (mg gallic acid/100g)	1674.27 ± 23.84 b	478.24 ± 18.40 a
Minerals (%)	3.06 ± 0.05 b	1.88 ± 0.09 a
K (mg/100g)	893.27 ± 88.09 b	483.51 ± 83.08 a
Mg (mg/100g)	59.57 ± 1.23 b	40.06 ± 2.99 a
Ca (mg/100g)	63.88 ± 8.85 b	40.92 ± 1.24 a
Na (mg/100g)	268.87 ± 50.88 b	115.99 ± 10.15 a
P (mg/100g)	62.86 ± 1.10 b	43.51 ± 0.68 a
Fe (mg/100g)	3.32 ± 0.10 b	2.24 ± 0.51 a
Zn (mg/100g)	2.85 ± 0.07 b	1.24 ± 0.28 a
Mn (mg/100g)	0.26 ± 0.03 b	0.07 ± 0.01 a
Cu (mg/100g)	0.04±0.01	0

Values followed by different letters within the same row are statistically different ($P < 0.05$)

potassium is the major mineral. Such composition is also reported by Abbès et al. (2011) for date syrup and by Tounsi et al. (2020) for carob syrup.

3.4. Functional characterization

Fig. 1 illustrates the functional properties of the studied syrups, in particular the emulsifying capacity (A) and the antioxidant activity (B).
Carob syrup was characterized by a greater emulsifying capacity probably linked to the high content of siloxanes (Table 2) also used as emulsifiers according to Codex Alimentarius (2013).
In addition, carob syrup was characterized by a greater antioxidant activity generally attributed to the high content of phenolic compounds. Abbès et al. (2013) and Tounsi et al. (2020) also determined the antioxidant activity of date syrup and carob syrup, respectively, which is mainly attributed to polyphenols. They also suggested other antioxidant compounds such as peptides,

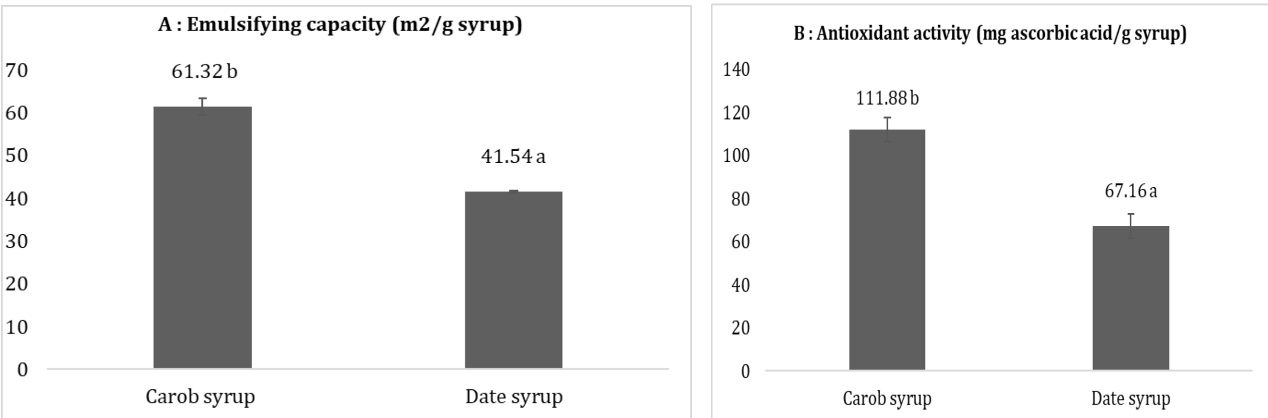

Fig 1. Functional properties of carob and date syrup
Values followed by different letters are statistically different ($P < 0.05$)

organic acids and products of non-enzymatic browning reactions, in particular melanoidins and HMF, which are naturally produced in foods during heat treatment and storage.

4. CONCLUSION

Both carob and date syrups had common characteristics, especially the richness in sugars and bioactive compounds. However, there are differences between them in terms of color, functional properties, aromatic and nutritional composition. Therefore, this study encourages the cultivation of the carob tree and the industrial production of carob syrup as is the case for the date syrup widely produced in Tunisia.

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