



## Effect of ultrafiltration process on physico-chemical, rheological, microstructure and thermal properties of syrups from male and female date palm saps



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### ABSTRACT

This study investigates the effect of the ultrafiltration process on physicochemical, rheological, microstructure and thermal properties of syrups from male and female date palm sap. All the studied syrups switched from pseudoplastic rheological behaviour ( $n = 0.783$ ) to Newtonian behaviour ( $n \sim 1$ ) from 10 to 50 °C respectively and present similar thermal profiles. Results revealed that the ultrafiltration process significantly affects the rheological behaviour of the male and female syrups. These differences on rheological properties are attributed to the variation of chemical composition between sap and sap permeate syrups. Furthermore, the effect of temperature on viscosity of the syrups was investigated during heating and cooling processes at the same shear rate ( $50 \text{ s}^{-1}$ ). This study provides idea of the stability of the syrup by evaluating the area between heating and cooling curves. Actually, the syrup prepared from male sap permeate is the most stable between the four studied syrups.

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## 1. Introduction

Date palm (*Phoenix dactylifera* L.) is a woody, perennial and dioecious plant species, with separate male and female trees, belonging to the *Arecaceae* family. In Tunisia, the total number of date palms is around 5.4 million of the 120 million world's date palms (Makhoulouf et al., 2015). In Tunisia, date palm tapping is a traditional practice. It's well known that sap extraction for a long tapping period (higher than three or four months) has always been marked by a potential risk for date palm tree. In fact, in this case

three to four years are needed for the tree to bear a full crop of date again and five years before tapping it again (Barreveld, 1993). However, date palm groves are preserved when limiting the tapping period to only two or three months or tapping mostly male palms and aged females respecting the optimal collection period. Conventionally, the sap collection period lasts in general 3–4 months (March–June) in which the total yield of sap per palm can easily reach 500 L (Barreveld, 1993). The date palm sap can be directly consumed as a fresh drink, called Lagmi or used as an alcoholic beverage after natural fermentation (Makhoulouf et al., 2015). Because of the demand of date palm sap and the difficulty to preserve it from natural fermentation, stabilizing it by producing syrup through thermal process is a solution to generate added value to this product. Indeed, a similar process has been applied by Li and Seeram (2011) to produce syrup by thermal evaporation

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of the colourless watery sap collected from maple (*Acer*) species. The maple sap has an important socioeconomic impact in the north-eastern region of North America as evoked (Li & Seeram, 2011), in addition, it is especially considered as a safe alternative to refined sugar as a sweetener (St-Pierre et al., 2014).

Date palm sap syrups, until now, have not been produced in an industrial scale. To our knowledge, this is the first study to valorize this natural drink by producing syrups which are marked by a good nutritional value. In fact, they are characterized by high amounts of sugars (58–75 g/100 g fresh matter basis), minerals (2.1–2.6 g/100 g fresh matter basis) and phenolics (147.61–224.55 mg of ferulic acid equivalents/kg fresh weight) (Ben Thabet et al., 2009). These new syrups can be used as an ingredient in to many food products such as dessert and cake topping, as is the case with maple syrup.

Processes leading to obtain stabilized products are, in general, delicate and depend on various parameters; one of the deterministic factors of the success of such a process in food industry is the viscosity of the treated fluid. Magerramov, Abdulagatov, Azizov, and Abdulagatov (2007) showed the importance of the accurate viscosity data of a fluid used in food technology, such as developing food processes and processing equipment, control of products, filters and mixers, quality control and an understanding of the structure of food and raw agricultural materials.

In addition, viscosity presents the fundamental parameter of the rheological study of liquids conducted by simple analyses and is more and more applied to determine the behaviour of treated solutions, suspensions or mixtures and to characterize the fluid texture (Rao, 1999).

Liquid viscosity depends essentially on temperature and composition. Rao (1999) showed the necessity to document the temperature effect on rheological properties, since a wide range of temperatures encountered processing and storage problems of liquid foods.

Moreover, the physicochemical properties of the fluid food could be altered during processing, such as ultrafiltration, which consist of separate micro scaled particles from the other elements of the treated matter affecting physicochemical properties and sensory characteristics of food products.

The ultrafiltration process becomes an essential part in food technology as a tool for separation and concentration (De Carvalho, de Castro, & da Silva, 2008). Its importance lies in the preservation of the natural juice constituents such as sugars, vitamins, as well as the volatile aroma profile, improving the microbiological quality of the permeates (Mohammad, Ng, Lim, & Ng, 2012).

Ben Thabet et al. (2009) studied the rheological behaviour of the syrups prepared from date palm sap. However, neither the effect of date palm sex nor the effect of the ultrafiltration process on rheological properties of syrups has been reported. The present work constitutes, to the best of our knowledge, the first study of the effect of the ultrafiltration process on physicochemical, rheological, microstructure and thermal properties of syrups prepared from male and female date palm saps, pursued by an investigation of the effect of temperature on viscosity of these syrups during heating and cooling processes.

## 2. Materials and methods

### 2.1. Syrup preparation

Exuding saps were collected by a traditional tapping method from a palm grove in Tunisia from male and female date palms (*P. dactylifera* L.). The trees were subjected to the same sunlight conditions, mineral nutrition and watering. After filtration through

a fine cloth, male sap was divided into two quantities; the first one was concentrated with a rotary vacuum evaporator, at 60 °C, to 74 °Brix. The second sap quantity was clarified by the ultrafiltration process (as described below in Section 2.2). The obtained permeate was after that concentrated with a rotary vacuum evaporator, at 60 °C, to 74 °Brix. The same procedure was applied for female sap. Final concentration value of the sap and permeate (74 °Brix) was chosen according to a previous study (Ben Thabet et al., 2009). Indeed, these authors found that sap syrups concentrated to 74 °Brix presents better sugar and phenolic compounds composition, better antioxidant activity and a better acceptability by consumers than syrups concentrated at lower values (64 and 68 °Brix).

Finally, 4 syrup samples, elaborated from male sap (MSS), male permeate (MPS), female sap (FSS) and female permeate (FPS), were obtained. The concentration of the juices was then carried out in the dark using aluminium foil, allowed to cool and stored at 4 °C before further uses.

### 2.2. Ultrafiltration process

Male and female date palm saps were clarified using a pilot-scale UF unit (Rhodi a Orelis, France). The UF pilot consisted of a screw pump, and two pressure transducers (0–6 bars) located at the inlet (Pi) and outlet (Po) of the membrane module. Transmembrane pressure TMP was calculated as  $TMP = (Po + Pi)/2$ . The feed temperature was adjusted by passing the date palm saps through a thermo stated bath fixed at  $25 \text{ °C} \pm 2$ . The feed flow rate was measured using a magnetic flow meter. Experiments were performed in a batch mode. Permeate was continuously recollected, and the flow rate was measured. CARBOSEP membrane with nominal molecular weight cut-off (MWC O) of 15 kDa (M2) made of zirconium oxide with carbon support, with inner and outer diameters of 6 and 10 mm, respectively, and 40 cm in length, with a surface area of  $0.0075 \text{ m}^2$  was used. The membrane was cleaned after each run according to the manufacturer's recommendations with NaOH ( $5\text{--}10 \text{ g L}^{-1}$ ,  $T = 80\text{--}85 \text{ °C}$ ; operating time = 30 min;  $TMP = 2\text{--}3 \text{ bars}$ ) and followed by an acidic treatment with  $\text{HNO}_3$  ( $3\text{--}5 \text{ mL L}^{-1}$  at  $55\text{--}60 \text{ °C}$  for 30 min at a  $TMP$  of 2–3 bars), until the original water flux was restored.

### 2.3. Physico-chemical analyses

Water activity was measured by a water activity meter (AW SPRINT TH-500 Novasina, Swiss). The pH was measured using a pH-meter (METTLER TOLEDO. MP220). The Brix degree was measured with an Abbe refractometer (Optech, Germany) at 20 °C.

Pectin content was determined as described by Englyst, Quigley, and Hudson (1994), using galacturonic acid as a standard. Ash content was estimated by combustion of 2 g of syrup sample in a Muffle furnace (Nabertherm, Germany) at 550 °C for 6 h (AFNOR, 1999). The residue was dissolved in  $\text{HNO}_3$  (14.44 mol/L) and mineral constituents (Ca, K, Mg, Na and Zn) were analysed separately, using an atomic absorption spectrophotometer (analytik jean, ZEE-nit, Germany) (Larrauri, Rupérez, Borroto, & Saura-Calixto, 1996).

Total sugars content was determined using the phenol sulphuric method (Dubois, Gilles, Hamilton, Rebers, & Smith, 1956). Reducing sugars were estimated according to Müller (1959).

### 2.4. Rheological properties

Rheological properties of syrups were measured using a modular compact rheometer (MCR 302, Anton Paar). Cone/plate geometry was used with a plate radius of 40 mm and a cone angle of 4°. The gap between the cone and plate was set at 150 µm. For each test, the sample was placed on the bottom plate of the rheometer.

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