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Effect of enzymatic treatment on rheological properties, glass temperature transition and microstructure of date syrup

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ABSTRACT

A knowledge of rheological properties is of importance in processing, handling, process design, product development and quality control. This study investigates the effect of commercial hydrolytic enzymes (pectinase and cellulase) on rheological properties and glass temperature transition of date syrup. Date syrups obtained by enzymatic extraction exhibited a quasi Newtonian behaviour.

The enzyme-treated date syrups showed the highest values of activation energy (E_a). Therefore, these syrups showed the most temperature dependency. Dynamic shear results revealed viscous behaviour for date syrups indicating that both syrups displayed liquid-like behaviour. Temperature significantly affected both (G') and (G''). The glass transition temperature (Tg) of date syrups varied between −39.56 and −45.74 °C depending on their composition. Glass transition temperature of date syrups decreased linearly with an increase in water content.

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1. Introduction

The world production of dates (*Phoenix dactylifera* L.) increased considerably during the last 30 years. Tunisia is considered to be one of the date producing countries, and the first exporter (in value) of dates (Besbes, Drira, Blecker, Deroanne, & Attia, 2009). The quantities of date produced annually in Tunisia reached 180,000 tonnes in 2011 (FAOSTAT, 2013).

This production progress is accompanied by a substantial increase of loss during picking, storage, commercialization and technologic transformation (Besbes et al., 2009; Masmoudi et al., 2008). These lost dates could amount to 30% of the total production (Besbes et al., 2006). This by-product is not consumed by humans because of its too hard texture or contamination with fungus and/or infestation by insect or simply due to their low quality. This category of dates is generally discarded or partially integrated in animal feed (Besbes et al., 2006). This by-product which is not accepted for the packaging and transformation industries is sold at low prices. Low quality dates are a rich source of carbohydrates (73.3–89.55 g/100 g dray matter) composed mainly of sugars, dietary fibres and other substances and are safe for human consumption (Abbès et al., 2011; Besbes et al., 2009). Industrially, they are used to produce several products such as jam, syrup, date-jelly, date-pastes, date butter, liquid sugar, vinegar, ethyl alcohol, single cell protein, …

Date syrup, a food commodity locally known as «Rub Al Tamr», is produced in Libya, Iraq and Tunisia from some local date varieties (Barreveld, 1993).

Date syrups are rich in some nutrients (sugar, phenolic compounds …) and provide a good source of rapid energy due to their high sugar content. Indeed, the high sugar content should justify their use as a source of liquid sugar suitable to many food products such as: base of beverage, bakery products, ice cream and in confectionery. Furthermore, date syrup can be used as a sweetening and flavouring agent for dairy products such as fermented milk products.

Some researches have been made to use date syrup in the preparation of cake, cookies and sweet breads and for direct use on pancakes. Furthermore, date syrup can be used to replace caramel in candy and fruit bars (Barreveld, 1993).

Food processing can affect physico-chemical properties and sensory characteristics of food products. In the literature, many studies reported that the enzymatic processed fruits and vegetables have different composition than unprocessed products (Buchert et al., 2005; Demir, Acar, Sarioğlu, & Mutlu, 2001; Rai, Majumdar, DasGupta, & De, 2004). In a previous work (Abbès et al., 2011),

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we found a significant increase in reducing sugars content when date syrup was prepared after extraction with pectinase and cellulase mixture. For industrial processes to be technically and economically feasible, it is important to have the knowledge of the physical and chemical properties. The rheological behaviour is one of the most important of these properties for food product development and because many problems are encountered during the manufacturing process including mixing, pumping, transport in tubes and evaporation.

The dynamic rheological properties, storage modulus (G') and loss modulus (G'') are frequently used to characterize the visco-elastic properties, especially during thermal treatment. There are many published works about the rheological properties of concentrated fruit juices (Ibraz, Gonzalez, & Esplugas, 1994; Ibraz, Gonzalez, Esplugas, & Vicente, 1992; Ibarz & Pagan, 1987; Ibarz, Pagan, Gutierrez, & Vicente, 1989), as well as syrups prepared from date palm sap (Ben Thabet et al., 2009). However, there is no information about the rheological properties of date fruit syrup. Studies on the rheological properties of date syrups, like other fluid foods, could be important for its production, processing, handling, quality control, sensory analysis and storage. The effect of temperature on rheological properties also needs to be documented because a wide range of temperatures encountered processing and storage problems of liquid foods (Rao, 1999).

The aim of this work is to study the effect of some hydrolytic enzymes on rheological properties of syrups obtained from three date varieties.

2. Materials and methods

2.1. Materials

Low quality dates (*P. dactylifera* L.) of the most abundant cultivars in Tunisia: Deglet Nour (DN), Allig (A) and Kentichi (K) were obtained from the National Institute of Arid Zone (Degach, Tunisia, 2006). Dates were collected at full maturity "Tamr stage", presented hard texture and were not infected by fungi. Ten kilograms from each variety were directly divided into bags of 500 g and stored at −20 °C until use.

Prior to syrup processing, date fruits were gently defrosted, cleaned, pitted and crushed in a meat grinder (Moulinex, type NE 401, France) to produce date paste.

Commercial pectinolytic enzymes, pectinase and cellulolytic enzymes from *Aspergillus niger* were obtained from Sigma Chemical Co., St Louis, MO, USA and stored at 4 °C.

2.2. Sample preparation

Three independent experiments were conducted in each date sample.

Date syrup was prepared according to the method of Abbès et al. (2011). In brief, the traditional extraction (TE) was carried out as follows: Date paste (400 g) obtained from each date variety was mixed with 1200 ml of water. Sample was placed in a thermostatically controlled water bath at 100 °C for 15 min. The produced juice was filtered by a filtering cloth and then centrifuged at 2907 × *g* for 15 min. To prepare date syrup, juice was concentrated at 100 °C using a hotplate (Ufesa, Turkey) to 80 °Brix.

To test the effect of pectinase and cellulase, a portion of 400 g of date paste was weighed and homogenized with 1200 ml of water. The pH was adjusted to 4, using citric acid, before the addition of enzyme preparation (833.5 nKat of pectinase/100 g and 1.38 nKat of cellulase/100 g). Samples were mixed thoroughly and placed in a thermostatically controlled water bath at 50 °C during 120 min. Then, the enzyme was inactivated by heating the suspension at 90 °C for 5 min. After completion of the enzymatic maceration, the produced juice was filtered by a filtering cloth and then centrifuged at 2907 *g* for 15 min. To prepare date syrup, juice was concentrated at 100 °C using a hotplate (Ufesa, Turkey) to 80 °Brix.

2.3. Moisture content

Moisture of samples was determined by Karl Fischer titration (Ronkart et al., 2006) The Karl Fischer titration was carried out with a Karl Fischer titrator DL31 from Mettler Toledo using Hydranal® methanol rapid and the one-component reagent for volumetric Karl Fisher titration Hydranal® composite (5 mg H_2O/ml), both purchased from Riedel-DeHaën (Seelze, Germany).

2.4. Rheological properties

The rheological properties of syrups were measured using a controlled-stress rheometer (Bohlin Model CVO 120 Rheometer, Bohlin Instruments, UK), equipped with a cone and plate geometry (40 mm diameter, 4° angle and 150 μm gap). The measurement was controlled by Bohlin Software. For each test, a sample was placed on the bottom plate of the rheometer. The sample temperature was ramped between 20 and 80 °C with incremental step of 10 °C. The shear rate range applied was 0–500 s^{-1}. The exposed sample perimeter was covered with plastic trop to prevent sample dehydratation at higher temperature.

Flow behaviour was described by the fitting of the experimental results of shear stress against the shear rate with the power law model.

$$\tau = K\dot{\gamma}^n$$

where τ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), K is the consistency coefficient (Pa s^n) and n is the flow behaviour index.

The temperature dependency of apparent viscosity of samples was evaluated using the Arrhenius relationship:

$$\eta = \eta_0 e^{(E_a/RT)}$$

where η is the viscosity at temperature T (Pa s), η_0 is a pre-exponential factor (Pa s), E_a is the activation energy (kJ mol^{-1}), R is the perfect gas constant (kJ mol^{-1} K^{-1}) and T is the absolute temperature (K).

Dynamic oscillatory tests were carried out at a frequency sweep of 5 Hz and a stress sweep of 10 Pa. The oscillation stress was selected based on linear part of the visco-elastic range. Measurements were made between 0 °C and 90 °C with incremental step of 10 °C. Each time, a new sample of syrup was used for rheological measurement.

All the rheological measurements were performed in triplicate. Storage modulus G' indicates the elastic property of the food, while the loss modulus G'' measures the viscous property. The loss tangent (tan δ) is a relative measurement of both contributions, viscous and elastic ones, and indicates which characteristic prevails.

2.5. Measurement of thermal transition

Differential Scanning calorimeter (DSC) (Q 100, TA Instruments, New Castle, USA) was used to determine the thermal profile of syrups. A mechanical refrigerated cooling system (TA Instruments, New Castle, USA) was used to cool the syrup to −90 °C. The calorimeter was calibred with indium and n-dodecane standards. Nitrogen was used as purge gas at a flow rate of 50 ml/min in order to

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