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Characterisation of Date Juices Extracted from the Rest of Sorting of Deglet Nour Variety

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Abstract: The composition of three types of date juices, that differs by their couple of extraction and obtained from the rest of the sorting of cultivars Deglet Nour, were studied. The fruits were grown in Djerid region (Tozeur, Tunisia). Juices were characterised by yield, pH, soluble solids, organic acid, minerals content, individual carbohydrates, vitamin C, yeasts and moulds, coliforms and flora total aerobe contents. For the physicochemical parameters, only the J3 presents the best yields with content in citric acid of 2.13 g L^{-1}, in phosphor of 0.083% (dry mater), in glucose 26.529 g L^{-1}, in fructose 39.59 g L^{-1} and in sucrose 185.883 g L^{-1}. For the bacteriological parameters, the results show that all prepared juices answer the microbiological requirements of hygiene well.

Key words: Oasis, date palm, rest of sorting, juice

INTRODUCTION

The palm date (*Phoenix dactylifera* L.) plays a very important economic, social and ecological role for the people of the arid and semi arid regions. Indeed, the world production tripled while passing from 2,289,511 tons in 1974 to 6,772,068 tons in 2004 (FAOSTAT, 2005). In Tunisia, the path of the dates knew a remarkable development during the last years and the production reached an average of 110,000 tons per year and about 60% of which is of Deglet Nour which has a very substantial organoleptic quality and therefore a high sales value (FAOSTAT, 2005). This progress of production, on a world and national scale, is accompanied unfortunately by a substantial increase of the dates lost during the picking, the storage or conditioning of the dates. These losses can present some proportions superior to 30% of the production, what constitutes an enormous tonnage adjoining 30,000 tons $year^{-1}$ for Tunisia and neighbouring 2,000,000 tons $year^{-1}$ for all over the world.

Indeed, several works on the valorisation of these secondary dates have been done. In the middle east, for instance, pits date have been included in the diet of chickens, sheep and fish (Kamel *et al*., 1981; Elgassim *et al*., 1995; Yousif *et al*., 1996) and the effect of inclusion of date flesh and seeds in the diet of farm animals has been attempted by several works (Hamada *et al*., 2002; Hussein *et al*., 1998). Lately, few pharmacological studied have conducted on date palm and its derivates. For example, it has been shown that date extracts can be used to decrease or increase gastrointestinal transit in mice (Al Qarawi *et al*., 2003) and that date fruit extracts provided to the women after child birth stimulate their immune system (Puri *et al*., 2000). Natural fats from date flesh have been reported to prevent irritant contact dermatitis (SchliemannWillers *et al*., 2002) and that a polysaccharide isolated from Libyan dates presented an antitumor activity (Ishurd and Kennedy, 2005).

In addition to the above usage of date flesh and seeds, several date processing industries have developed and these process industries produce many products like date paste, date syrup, date dip and date vinegar from various date varieties. Recently, date pits were used as a new renewable energy source (Al-Omari, 2006).

This study addresses for the first time the valorisation of the rest of sorting from the Tunisia variety of Deglet Nour by preparation a juice for agro alimentary use. In fact, juice fruits contain many nutrients and an abundance of organic acids, vitamins and mineral elements. These juices have a potential in various aspects of the human biological function such controlling diseases and enhancing metabolism of the human body. There are extensive prospects to develop and utilise the fruit juice. For example, the suitability of citrus peels, generated by-product of the juice industry, was used as a source of antioxidant (Kang *et al*., 2006) and this same virtue was attributed to pomegranate juice (Rosenblat *et al*., 2006). On the other hand, fruit and vegetable juices can play an important role in delaying the onset of Alzheimer's disease (Dai *et al*., 2006).

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The objectives of this research were to perform chemical and bacteriologic analysis of three types of juices; these juices differ by their couple of extraction (T, t). The survey of a physicochemical and microbiologic characteristic permits to identify the optimal juice for an agroalimentary use. This juice is going to serve like raw material for the production of other products as the syrups and vinegar date.

MATERIALS AND METHODS

Material: The present work has been achieved from specific varieties from the region of Tozeur (Tunisia). It is about the rest of sorting of the Deglet-Nour variety.

Acetonitrile HPLC Reagent was LC grade and was obtained from Lab-Scan (Dublin, Ireland). Sucrose standard was purchased from Fluka (Buchs, Switzerland). Glucose and fructose were purchased from Carlo Erba (Val de Reuil, French). L-Ascorbic acid standard was Reagent grade and was obtained from MP Biomedicals (Eschwege, Germany). Phenolphthalein Ph Helv was from Fluka (Buchs, Germany). Orthophosphoric acid 85% was from Merck (French). The water used in HPLC and sampling was prepared with Millipore Simplicity (Millipore S.A.S, Molsheim, French).

Extraction of juice: The dates were first all washed, drained and pitted. A sample of 33 g of date cut into nubs was placed in a spade where 100 mL of ultra pure water was added and then the mixture was distributed in a bath. To study the effect of the parameters temperature and time on the composition of juice we repeated these stages for three different conditions:

- The first 75°C, during 90 min,
- The second 75°C, during 60 min,
- The third 80°C, during 90 min.

Once the mixture was prepared two operations will occur:

- Filtration was done by a cloth of textile (compress).
- Centrifugation: the goal was to have a clearer juice (20 min, 20°C, 8000 rpm).

Finally, the juice was poured in conical tubes that will be cooled before taking place in the refrigerator and to be used for the analysis.

The transformation of the dates in juice follows the following diagram (Fig. 1).

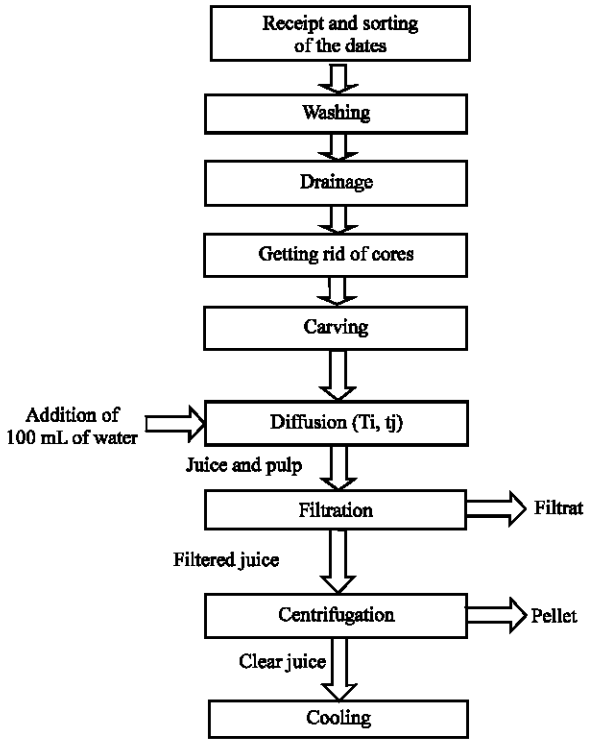

Fig. 1: Diagram of the transformation of the dates into juice

Analytical methods

Determination of the yield juice: It is the mass of juice gotten after filtration on the total juice mass:

$$R = Mj/Mt$$

Mj: mass of juice after filtration.
Mt: mass of total juice.

Soluble solids: The levels of soluble solids, expressed as degree Brix, were determined using a Reichert Refractometer (Models 10430, 0-30°Brix, Cambridge Instruments Inc, USA).

pH: The pH values were measured with a ph-meter (InoLab, Germany) after calibration with pH 4.0 and standard buffers.

Titratable acidity: It is about measuring out the citric acid that constitutes the major acid of date juice. This measure was achieved by neutralisation of the total free acidity with a solution decinormal of KOH. A sample of juice having a known volume of 10 mL has been titled by a solution of KOH1N, in presence of some drops of phenolphthalein indicator. To the zone of turn of the

colourful indicator the volume of basis versed Vb has been noted and the results were expressed in mill equivalent for 100 g product.

$$N_a = N_b \times V_b / V_a$$

With

N_a : Normality of acid.
N_b : Normality of the basis.
V_a : Volume of acid.
V_b : Volume of the basis.

Since a solution of 0.1 N of citric acid contains 64 g L^{-1} of this acid therefore a solution of Na normality contains 64 * Na g L^{-1}.

The quantity of citric acid (citric acid) contained in one litter of juice is expressed therefore by:

$$\text{Quantity of citric acid} = Na \times 64$$

Minerals contents: The sodium and potassium were determined by flame photometer (Sherwood 410, Sherwood Scientific Ltd, Cambridge, UK). Total phosphorus was determined by a Spectrophotometer (Secomam 1000, French).

Total sugars: The reducing and non-reducing sugars were determined by HPLC method. Prior to injection in the HPLC system, juice was filtered over a 0.45 μm membrane filter. Liquid Chromatography separation was carried out at room temperature on Eurospher NH2 column, 100 A pore size, 7μmm particle size, 250 m 4.6 mm I.D from Knauer (Germany). Prior to use, solvents were filtered over a 0.45 μm membrane filter and sonicated for 15 min in a ultrasonic bath (Ultrasonic Cleaner Model SM 25E-MT, Branson Ultrasonics Corporation, Dambury, USA). The mobile phase used was acetonitrile-water ultra-pure (80%, 20%, v/v). The LC was connected to RI Detector K-2301 from Knauer (Germany). The flow-rate and the injection volume during the experiment were 1.0 mL min^{-1} and 20 μL, respectively. The integrator was calibrated with external standards consisting of solutions of glucose (2%), fructose (2%) and sucrose (1%). Each sample was analysed in triplicate and quantification was carried out from integrated pick areas of the sample against the corresponding standard graph.

Analysis of ascorbic acid: To 1 mL of juice, 1 mL of 2% orthophosphoric acid was added, vortexed and the volume was adjusted to 5 mL by adding water ultra-pure. The mixture was centrifuged at 5000 rpm for 8 min at 4°C. The supernatant was filtered and vitamin C level was determined by HPLC, utilizing a column (250 mm*4.6 mm i.d.) packed with Eurospher C18 reversed-phase materiel (18.5 μm particle size) with mobile phase (water, pH 2.2) at 1 mL min^{-1} flow-rate.

Each sample was analysed in triplicate and quantification was carried out from integrated pick areas of the sample against the vitamin C standard graph.

Microbiological analysis of juice: A dilution of 10^{-1} of every juice has been done with the sterile water (with peptone).

Numbering of the yeasts and moulds: The numeration was achieved on petrifilm of yeast and moulds. It was a ready medium to use that contains the nutritious elements, of the antibiotics, a gelling soluble agent in cold water.

Numbering of the coliforms: The test CC petrifilm for the numeration of the coliforms contains a selective environment of VRBL type (composed of biliary salts, of purple crystal and neutral red), a gelling soluble agent in cold water and an indicator to the tetrazolium facilitating reading.

Numbering of flora total aerobe: The petrifilm test for the numeration of flora total aerobe, we chose a ready medium that contains the nutritious elements PCA: a gelling soluble agent in cold water and an indicator to the tetrabazolium facilitating reading. We used a standard colony meter.

Statistical analysis: Each parameter was analysed in triplicate. The collected data were subjected to a One-way ANOVA analysis. The test of least significant difference was applied to separate means when a significant value of F was obtained in the analysis of variance.

RESULTS AND DISCUSSION

Physicochemical characteristics of date juices: The composition of date juices showed significant differences among couple of extraction in terms of yields, glucose, fructose, sucrose, ascorbic acid, phosphorus, yeasts and moulds, coliforms and flora total aerobe content (Table 1). The J3 had significantly high fructose, glucose, sucrose, phosphorus and yeasts and moulds contents. Instead, the J2 showed high yield, ascorbic acid, coliformes and total aerobe contents.

The yield of juice according to couple (T, t) of the Deglet Nour variety studied was given by Table 1. Indeed, the best yield was recorded at the J2 (75°C, 60 min) by contribution to the other juices. The comparison between the J1 and J2 shows that at a same temperature, an increase of extraction time provokes a reduction of yield.

Table 1: Physicochemical and bacteriological characteristics of date juices extracted from different studied couples

Parameters	J1 (75°C, 90 min)	J2 (75°C, 60 min)	J2 (80°C, 90 min)
Yields (%)	83.390[b]	89.170[a]	80.300[a]
°Brix	17.200[a]	17.400[a]	17.567[a]
pH	5.46[a]	5.46[a]	5.46[a]
Citric acid (g L^{-1})	1.92[a]	1.92[a]	2.13[a]
Fructose (g L^{-1})	26.480[b]	36.177[a]	39.590[a]
Glucose (g L^{-1})	19.733[c]	22.938[b]	26.592[a]
Sucrose (g L^{-1})	128.038[c]	139.326[b]	185.863[a]
Vitamin C (mg L^{-1})	6.064[b]	7.327[a]	5.965[c]
Phosphorus (%)	0.056[b]	0.064[b]	0.083[a]
Sodium (%)	0.00131[a]	0.00120[a]	0.00151[a]
Potassium (%)	0.607[a]	0.556[a]	0.632[a]
Yeasts	150.67[b]	87.33[c]	236.33[a]
Coliforms	424.33[b]	899.67[a]	192.63[c]
Flora total aerobe	686.00[b]	931.00[a]	314.33[c]

Means values with different letter(s) in the same row are significantly different ($p<0.05$)

Whereas the comparison between the J1 and J3 shows that at a same time of extraction, an increase of temperature provokes a reduction of yield. We can conclude therefore that an increase of couple (T, t) provokes a reduction of production rate.

On other hand, no significant differences among juices were found for the °Brix and pH. Concerning the °Brix, the values of date juices ranged from 17.2 to 17.56% . In fact, this parameter could not exceed 10% in lime juice (Simmonds, 1984) or 11.1% in orange juices (Vieira *et al*., 2007).

For the total acidity, according to Table 1 we note that the J3 (80°C, 90 min) presents the most elevated value of citric acid (2.13 g L^{-1}). Indeed, this quantity remains insufficient because it doesn't permit a long conservation of this product. It is for this reason that one we resort to the addition of a citric acid solution that assures the correction of pH and the acidity at the same time. While leaning on the variation of the acidity and the variation of the pH according to couple (T, t) we note that a quantity of citric acid 0.21 g L^{-1} was necessary for a reduction of pH of 0.09.

Concerning the minerals content, the results show that the best yield in minerals was recorded at the J3 with content in phosphorus was of 0.083%, whereas the contents in sodium and in phosphorus were comparable at the three juices. In fact, many researchers have shown that high-potassium and low-sodium nutritional foods are beneficial to maintain the acid-balance within organisms and also helpful to prevent and cure hypertension (He, 1998). In comparison with other juice like lime juice, the tenor of potassium could not exceed 0.0082% (Ziena, 2000).

The works led by Reynes *et al*. (1994) on the minerals composition of the variety Deglet Nour showed that the content in phosphorus was 0.055%, in potassium is 0.639% and in sodium was 17.5 mg kg^{-1}. These results were near to those found at the J3 and the registered

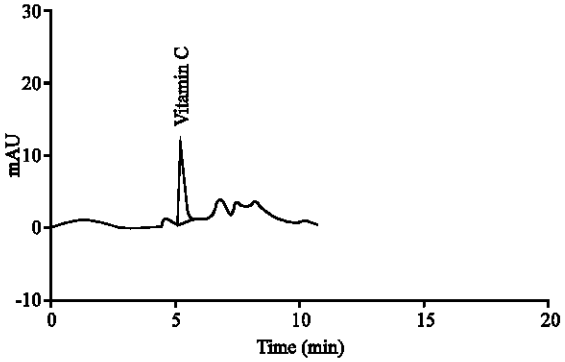

Fig. 2a: Chromatograms of C vitamin from juice 1

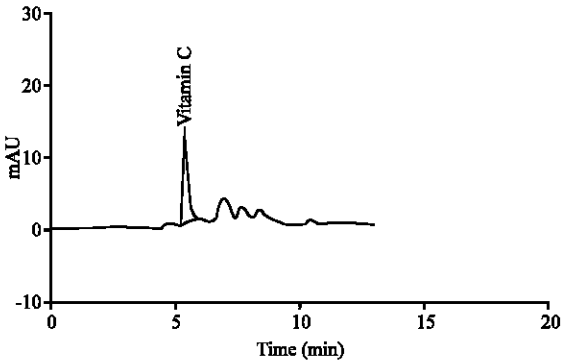

Fig. 2b: Chromatograms of C vitamin from juice 2

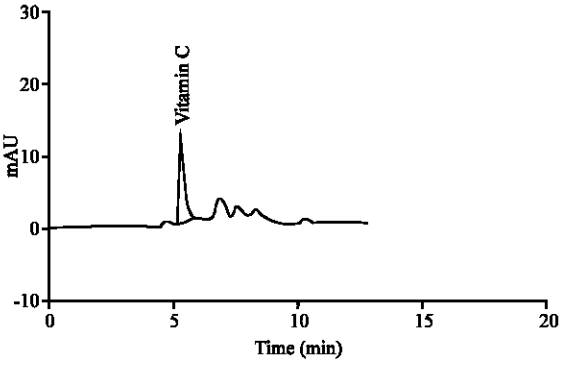

Fig. 2c: Chromatograms of C vitamin from juice 3

losses can be explained by their accumulation in the filtrates at the time of filtration. Indeed, an increase of couple (T, t) provokes a reduction of the filtrate mass gotten at the time of filtration and thereafter a reduction of the loss in minerals.

For the ascorbic acid content, the exam of data shows that the C vitamin was present in acceptable quantity (Fig. 2a-c). In fact, the values of this parameter ranged from 59.6 to 73.2 mg L^{-1} and this content reaches its maximum at the J2 (75°C, 60 min). We can conclude that the increase of the couple (T, t) provokes the destruction of this vitamin. However, these values were lower than

those showed in lime juice and in orange juice, respectively 390-620 and 196-634 mg L^{-1} (Simmonds, 1984; Meléndez-Martínez *et al.*, 2007).

Concerning the content sugars, the three majors sugars in date juices were sucrose, fructose and glucose (Fig. 3a-c). Each juice showed typical sugars contents, this being lowest in J1. The J3 had significantly high glucose, fructose and sucrose. Total sugars of date juices were found increase on time and temperature of extraction.

Microbiological characteristics of date juices: The results of the microbiologic analysis were distributed in Table 1. For the tenor of yeasts and moulds, the results show that the three juices were deprived of moulds, whereas the numbering of the yeasts indicates their presence to satisfactory proportions to NF standards ISO 7954/1988 (INDICATION OF ORDERING: V 08-022) that require that the number of germs L^{-1} for these bacteria must be lower than 5.10^2.

Concerning the number of flora total aerobe and coliforms, the obtained results were in the norms, they were satisfactory because the number of the coliforms was lower than 500 germs L^{-1} and the number of floras was lower than 103. These results show that the prepared juices answer the microbiological requirements of hygiene well.

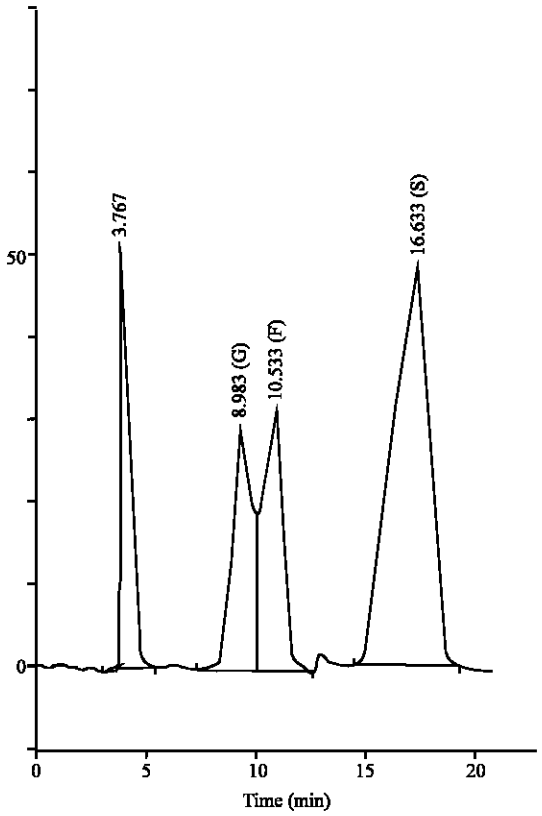

Fig. 3b: Chromatograms of sugars from juice 2

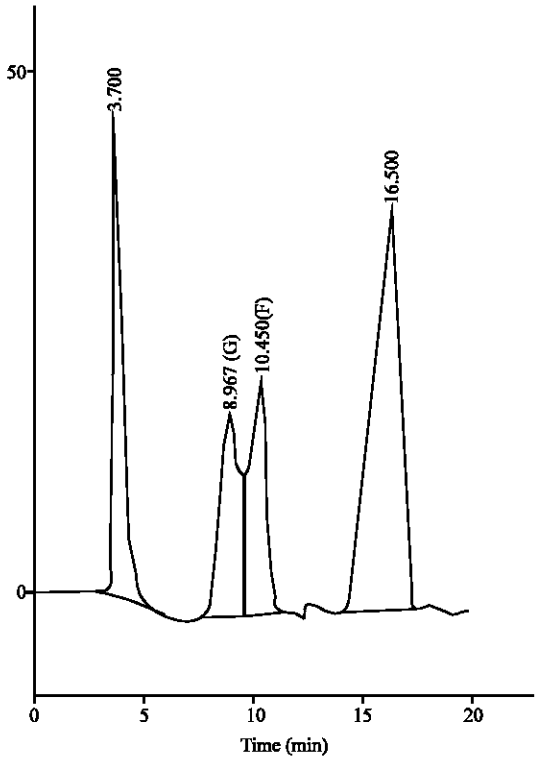

Fig. 3a: Chromatograms of sugars from juice 1

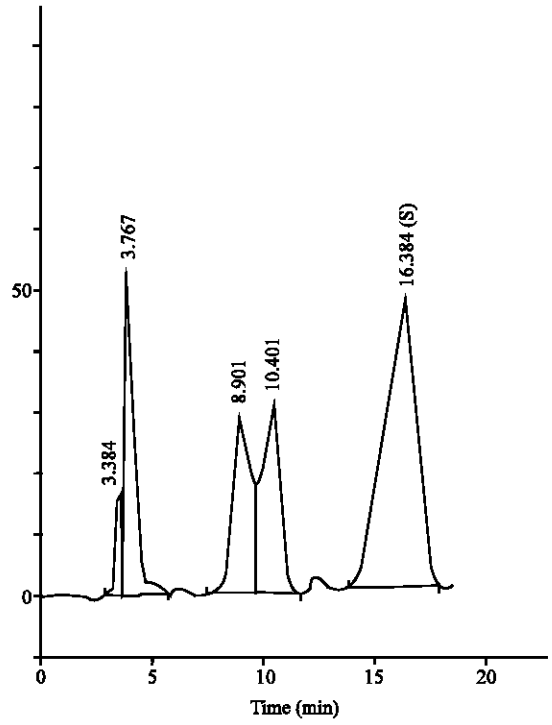

Fig. 3c: Chromatograms of sugars from juice 3

CONCLUSIONS

The objective of this research was the valorisation of the products of the date palms by producing juice from Deglet Nour variety. The physicochemical and microbiological analysis of the three juices prepared in conditions temperature-times different showed that J3 of which the couple (85°C, 90 min) can be considered as the best raw material for an agro alimentary use.

Indeed, in spite of the weakest yield in relation to the other juices, the J3 presents an important content, in reducing sugars (glucose, fructose), non reducing sugars (sucrose), in soluble solids, in minerals and middle in C vitamin. In the same way, the results show that this juice presents the most elevated value of citric acid. However, this quantity remains insufficient because it doesn't permit a long conservation of this product. In this context the addition of citric acid is necessary for a correction of pH and the acidity, thus permitting a better conservation. In addition, the microbiological analyses show that J3 presents the most satisfactory results.

This juice permits a diversification of products on the local market or even outside and constitutes a commercial outlet especially interesting for the high level of quantity of Delet Nour variety judged secondary. This juice can be considered like a raw material for the production of vinegar, alcohol, syrup and yeasts. It would be therefore useful to improve this new product by the addition of several additives and the clarification in order to increase its sales value and its appreciation by the consumers.

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