1 **Title: Dietary Fibre from Tunisian Common Date Cultivars (*Phoenix dactylifera***
2 **L.): Chemical Composition, Functional Properties and Antioxidant Capacity.**

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14 Running title: Antioxidant dietary fibre from Tunisian common date cultivars

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ABSTRACT

The dietary fibres (DF) of ten date varieties from Tunisian oasis have been investigated. Gain further knowledge on the content, composition and technological applications of those fibres could support their genetic variability and promote the socio-economical development of growing areas. The composition, water- and oil-holding capacities, solubility, and antiradical activity have been determined. The DF content ranged from 4.7% (Matteta, Rochdi) to higher than 7% (Deglé Nour, Garen Gaze, Smeti). Composition varied significantly among cultivars and the results evidenced that uronic acids and lignin determine to great extension the organoleptic quality of dates. Many of the varieties that have been studied (Garen Gaze, Matteta, Kenta, Rochdi, Mermella, Korkobbi, Eguwa) resulted of great interest from a technological and functional point of view. Among their physico-chemical characteristics, these samples presented water- and oil-holding capacities higher than 17 and 4 mL/g fibre, respectively, which make them suitable for being used as additives in fibre-enriched food. Also, DF of Garen Gaze, Smeti, Mermella, and Eguwa had a high antiradical capacity (more than 230 Trolox equivalent/Kg fibre). We can conclude that some of these varieties could be grown as potential sources of DF, which could be included in the formulation of fibre- and antioxidant-enriched foods.

KEYWORDS: dietary fibre, date palm fruits, chemical composition, functional characteristics, antioxidant capacity

INTRODUCTION

The date (*Phoenix dactylifera* L.) is an important crop in arid and semiarid regions of the world. Nearly 2,000 cultivars of date palm are known in the world, but only some of them are evaluated for their performance and their fruit quality. The date production has tripled from 1,915,615 tons in 1975 to 6,002,040 tons in 2005¹. In Tunisia, the mean annual production of date fruits has remarkably improved and reached an average of 120,000 tons/year, dominated by the Deglé Nour variety (60% of total production), which has a highly appreciated sensory quality leading to a high marketing value. This progress in production, at the national and international scales, is unfortunately accompanied by a considerable increase loss in secondary or common-variety dates (approximately 30,000 tons for Tunisia and 2,000,000 tons for the world). These dates are generally rejected or in some limited cases used for animal feed. It is by this selective orientation that we are currently witnessing a progressive disappearance of secondary cultivars and therefore a reduction in genetic variability. Among the threatened cultivars are those of the coastline oasis of Gabès. To fight against this ecological and economic problem, several studies on the valorisation of common dates have been conducted. The literature mentions certain technological transformations, e.g., the production of jams, frosts, juice and syrup of dates^{2, 3}. The chemical composition of the date shows that the flesh is an important source of sugar (81–88%, mainly fructose, glucose and sucrose), DF (5–8.5%) and small amounts of protein, fat, ash and polyphenol^{4, 5}. Thus, dates provide a good source of rapid energy (sugars) and good nutritional value, based on their DF contents.

The demand of by-products from fruits and vegetables as sources of DF has been increasing because these sources offer higher nutritional quality, higher amounts of total and soluble fibre, lower caloric content, stronger antioxidant capacity, water- and oil-

retention capacities and colonic fermentability, as well as a lower phytic acid and caloric value content^{6, 7} than cereal by-products. DF plays an important role in human health and has shown beneficial effects in the prevention of several diseases, such as cardiovascular diseases, diverticulosis, coronary heart disease, constipation, irritable colon, colon cancer, atherosclerosis, obesity and diabetes^{5, 7, 8}.

To date, limited data are available regarding the compositional and functional characteristics of common dates grown in Tunisia⁹. The common date may possess high-value components that may be used in value-added applications, including their use as functional foods and ingredients in nutraceuticals^{5, 10}.

The objective of this study was to isolate common date dietary fibres and evaluate their chemical composition and functional properties. This is the first time that a complete study on chemical composition of date dietary fibre has been done. Nowadays, the antioxidant capacity is one of the most valuable among functional properties. We have studied this capacity in dietary fibre of common dates, especially the one linked to fibre. This is the characteristic which could ultimately promote the use of dates and their corresponding fibres as “antioxidant fibres” in the market of healthy ingredients for functional food formulation.

MATERIALS AND METHODS

Chemicals. 4-Morpholineethanesulfonic acid (MES), protease from *Bacillus licheniformis*, amyloglucosidase solution from *Aspergillus niger*, tris(hydroxymethyl)aminomethane (TRIS), trifluoroacetic acid, 3-phenylphenol, anthrone, Folin-Ciocalteu phenol reagent, 2,2-diphenyl-1-picrylhydrazyl (DPPH• free radical) were purchased from Sigma-Aldrich Química (Madrid, Spain). Amylase thermostable Thermamyl 120L was from Novo Nordisk Pharma (Madrid, Spain).

Na₂CO₃, sodium hydroxide and acetic acid were from Panreac Química S.A. (Barcelona, Spain). Standards of gallic acid (GA), myo-inositol, and *t*-cinnamic acid were purchased from Sigma-Aldrich Química (Madrid, Spain). Ethyl acetate and acetonitrile were of HPLC grade purity (Romyl, Teknokroma, Barcelona, Spain). Sulphuric acid and acetone was from Sharlau (Barcelona, Spain). Ethanol was purchased from Alcoholes del Sur (Córdoba, Spain)

Samples. Ten varieties of dates (Bouhattam, Matteta, Kenta, Eguwa, Garen Gaze, Limsi, Rochdi, Smeti, Mermella and Korkobbi) from the Gabès littoral oasis (south of Tunisia) were picked at commercial maturity (Tarm stage) during the 2010 harvest season (September-October). Identification of each cultivar was visually verified by the experienced farmers. The study included a total of 200 fresh samples consisting of 20 palm trees per variety, nearly 600 fruits per variety. Sample from the Deglé Nour cultivar was collected from the continental oasis of Tozeur. It was also picked at Tarm stage and it was used as a reference sample. All dates were stored in a refrigerator at approximately 4 °C (1-2 months) until all samples were collected for analysis and extraction.

Dietary fibre extraction. The amount of DF was determined using the protocol described by Lee, Prosky, and De Vries (1992)¹¹, with slight modifications. As samples contained a high level of sugar, they were previously extracted with 80% ethanol to remove most of the sugars. Triplicate dry and de-sugared samples (1 g each) were suspended in 40 mL MES-TRIS buffer and treated with 50 µL Thermamyl (heat stable alpha-amylase) at 100 °C for 15 min and then digested with 100 µL of a 50 mg/mL protease solution (60 °C, 30 min), followed by incubation with 100 µL amyloglucosidase (60 °C, 1 h) to remove protein and starch. Four volumes of 96% hot ethanol were then added to precipitate soluble DF. Total DF was recovered by filtration

on a sintered glass crucible (no. 2) using the Fibertec E system consisting of the 1023 Filtration Module. The residue was then washed with 80% ethanol and 96% ethanol and dried overnight at 105 °C in an air oven and then weighed. The dried fibres were ground in a hammer mill to a particle size smaller than 1 mm and stored at 4 °C until analysis.

Composition of dietary fibre. Neutral sugars, uronic acids, proteins, and Klason lignin were determined as described previously ¹². For neutral sugars, fibres were hydrolyzed with trifluoroacetic acid at 121 °C for 1 h, and the released sugars were quantified as alditol acetates by gas chromatography. A HP 6890 Plus+ gas chromatograph (Hewlett-Packard, Palo Alto, CA) fitted with a 30 m x 250 µm x 0.20 mm capillary column (SP-2330, Supelco, Bellefonte, PA) was used. The carrier gas was helium with a constant flow equal to 2.2 mL/min, pressure 21.5 psi (148.24 kPa). Injection was performed in splitless mode. The oven temperature was held at 50 °C for 2 min after injection, then programmed to 180 °C at 35 °C/min, held at 180 °C for 5 min, and then immediately increased to 220 °C at 5 °C/min, and held at 220 °C for 22 min. Total run was 40.7 min. The injector temperature was 250 °C, flame ionization detector (FID), 300 °C. Myo-inositol was used as internal standard.

Uronic acids were quantified using the phenyl-phenol method after sulphuric acid hydrolysis ¹³; proteins (for DF corrections) were analyzed by the Kjeldahl method using a Büchi Digestion Unit, K-424, and a Büchi Distillation Unit, K-314, and applying a factor of 6.25 to convert the total nitrogen into protein content; Klason lignin levels were determined gravimetrically as the amount of acid-insoluble material remaining after a two-stage sulphuric acid hydrolysis. Cellulose was quantified from the trifluoroacetic acid-insoluble residue after 72% sulphuric acid hydrolysis by the anthrone method.

Water-holding capacity. The water-holding capacity was determined using the method described by Jiménez et al. (2000)¹⁴. Samples (250 mg x3) were suspended in 15 ml of water. After 24 h of stirring at room temperature, the suspension was centrifuged at 14,000 g for 1 h. Supernatants were carefully eliminated, and the hydrated fibres were weighed. WHC was expressed as mL of water/g fibre. Hydrated pellets were freeze-dried, and their solubility in water was determined by the difference in weight between before and after the WHC assay, which was expressed as a percent.

Oil-holding capacity. The oil-holding capacity was determined using the method described by Jimenez et al. (2000)¹⁴. Oil-holding capacity was measured by adding 15 ml of sunflower oil (1.0054 g/mL density) to a concentrate of date fibres (250 mg x3) in a 50-mL centrifuge tube. The content was stirred for 24 h at room temperature; then, the tubes were centrifuged at 14,000 g for 1 h. Supernatants were carefully eliminated, and the oil-embedded fibres were weighed. Oil-holding capacity was expressed as mL oil/g of fibre.

Extraction of soluble phenols. Ten grams (by duplicate) of fresh pulp was homogenized in an Ultraturax at top speed for 1 minute with 50 mL of a 70:30 mixture of acetone:water. The suspension was extracted over 30 min by sonication in an ice bath. After filtration, the slurry was extracted again under the same conditions. Both filtrates were collected. The solvent was evaporated under vacuum and the extracts redissolved in 10 mL of the same solvent. These concentrated extracts were used to determine soluble phenols and soluble antiradical activity. The dry pellet was used to determine the antioxidant activity of the fibre portion. The yield of this extraction step was used to generate results for antioxidant activity in fresh weight (FW) basis.

Determination of soluble phenols. The total polyphenol content was quantified for each date extract according to the Folin-Ciocalteu spectrophotometric method, using

gallic acid as a reference standard. Aliquots of 0.2 mL of each sample were dosified in triplicate, and 0.5 mL of Folin-Ciocalteu phenol reagent (0.2 M) was added to each tube and mixed. Afterward, 0.4 mL of Na₂CO₃ (75 g/L) was added and mixed well. A microplate reader was set at 630 nm, and the absorbance was measured after 60 min. Results are expressed as gallic acid (GA) equivalents (mg/100 g fresh weight).

Determination of the antiradical activity. Soluble antioxidant activity was determined from the soluble phenol extracts by the DPPH· method ¹⁵. Fibre antioxidant activity was evaluated as described by Fuentes-Alventosa et al. (2009b) ¹⁶. Between 3 and 20 mg of soluble phenol-free fibres was transferred to an eppendorf tube (for weights lower than 3 mg, fibres had to be diluted with cellulose as an inert material), and the reaction was started by adding 1 mL of the DPPH· reagent (3.8 mg/50 mL methanol). After 30 min of continuous stirring, samples were centrifuged, and the absorbance of the cleared supernatants was measured (in triplicate) at 480 nm. Both antioxidant activities were expressed as millimoles of Trolox equivalent antioxidant capacity per kilogram of sample by means of a dose-response curve for Trolox.

Extraction and quantification of ester-linked phenolics. Ester-linked phenolics present in fibre samples were extracted and quantified as previously described ¹⁷. Briefly, soluble phenol-free samples (in duplicate) were treated with 2 N NaOH for 24 h at room temperature under nitrogen and in the dark. After filtration, *t*-cinnamic acid was added as an internal standard. Solutions were acidified and extracted three times with ethyl acetate. Ethyl acetate extracts were evaporated under nitrogen, re-dissolved in 50% methanol, and analyzed by HPLC. Phenolic compounds were quantified using a Synergy 4µ Hydro-RP80A reverse-phase column (25 cm x 4.6 mm i.d., 4µ; Phenomenex, Macclesfield, Cheshire, U.K.). The gradient profile was formed using solvent A (10% aqueous acetonitrile plus 2 mL/L acetic acid) and solvent B (40%

methanol, 40% acetonitrile, and 20% water plus 2 mL/L acetic acid) according to the following program: the proportion of B increased from 10 to 42.5% for the first 17 min, was held isocratically at 42.5% for a further 6 min, increased to 100% over the next 17 min, and finally returned to the initial conditions. The flow rate was 1 mL/min. Phenols were detected using a Jasco-LC-Net II ADC liquid chromatograph system equipped with DAD and a Rheodyne injection valve (20- μ L loop). Quantification was performed by integrating the peak areas at 280 nm with reference to calibrations performed while using known amounts of pure compounds.

Statistical analysis. The results are expressed as mean value $\pm$ standard deviation. To assess the differences in composition, functional characteristics and antiradical activity between the different date varieties, a multiple-sample comparison was performed using the Statgraphics Plus program Version 2.1. Multivariate analysis of variance (ANOVA), followed by Duncan's multiple comparison test, was performed to differentiate the groups. The level of significance was $P < 0.05$. Correlation coefficients (R) were determined using regression analysis at the same confidence level.

RESULTS AND DISCUSSION

Chemical composition of date dietary fibre.

Ten different varieties of dates from the coastline oases of Gabès were analyzed to characterise their DF content. The Deglé Nour variety was also studied as a standard for comparison. The percentage of DF in each variety is presented in **Table 1**. Deglé Nour, Garen Gaze and Smeti had the highest DF content: more than 7 % in fresh weight. Matteta and Rochdi had the lowest. These percentages were consistent with those found in the bibliography for these fruits: 5.9-8.7% for Omani dates^{5, 18}. In other studies, the percentage of fibre was much higher: 6.39-11.35%⁴, 10.88-13.45%⁹ or

6.99-17.67%¹⁹. Besides being attributed to variety, these differences could be also related to the stage of maturation, as during the ripening process, enzymes gradually break down polysaccharides to more soluble compounds¹⁸, decreasing the fibre content. The analytical method used to quantify the fibre content is also a factor because some authors do not de-sugar the samples before analysis, so the total DF content would have been overestimated⁵. The DF contents of a number of fresh fruits, such as apples, banana, cherry, mango, muskmelon, and peach, were reported by Punna and Paruchuri (2003)²⁰. The values obtained ranged from 0.8 g/100 g for muskmelon to 2 g/ 100 g for mango. In addition, the DF contents of other dried fruits, such as raisins (3.7%), plums (7.1%) or figs (9.8%)²¹, are similar to those observed for dates. Thus, date flesh could be considered a good source of DF compared with most fresh and dried fruits.

After DF hydrolysis, the content in neutral sugars, uronic acids, cellulose and Klason lignin was quantified (**Table 1**). These values ranged from 15.56 to 25.71 g/100 g DF for neutral sugars, 10.74 to 16.71 g/100 g DF for uronic acids, 17.01 to 24.82 g/100 g DF for cellulose, and 33.35 to 50.37 g/100 g DF for Klason lignin. The last one was the major component in all of the samples, as was the case for other date varieties⁹.²². Deglé Nour and Eguwa had a very similar composition. Both varieties had the highest amount of Klason lignin and the lowest of neutral sugars. The DF composition of some varieties was different with respect to those previously described. Elleuch et al. (2008)⁹ analyzed Deglé Nour and Allig varieties from the Déguech region (Tunisia). They found that Deglé Nour had a lower amount of lignin (26% for lignin, cellulose and uronic acids, and 21% for neutral sugars), though Allig was similar to Matteta, Kenta, Rochdi in our study. The compositional differences of the Deglé Nour variety could be due to the growth zone, as the Déguech zone is a mountain oasis. The Iranian variety Dalaki has been studied as a source of fibre and juice for fermentation²². This variety is

a waste date due to its harder texture and higher fibre density compared to commercial edible-grade fruit. The composition of Dalaki date flesh fibre is approximately 70% lignin, 15% cellulose, 15% neutral sugars and a negligible amount of uronic acids. It seems that lignin and uronic acids could be the key compounds in determining the quality of dates: high lignin and low pectins content could indicate inedibility²². On the contrary, low lignin and high pectins content are indicators of good quality. The varieties researched in this study exhibited intermediate contents, with Matteta, Kenta and Rochdi possessing a valuable composition (the highest in uronic acids and the lowest in Klason lignin).

Compared to other fruits, date fibres featured a neutral sugars content that is slightly higher than that found in other fruits or vegetables. In guava pulp²³, neutral sugars accounted for approximately 16%, but lower levels (5–7%) were found in cocoa bean husks²⁴ and some citrus by-products²⁵. Uronic acids contents also showed high variability, depending on the product studied. The lowest level was found in guava pulp²³, which was 2%. Lime peels had the highest level, around 25%²⁶. Uronic acid levels similar to those in date were found in peach pulp²⁷. The cellulose content of date fruit DF was also in the range of that of other fruits and vegetables, with the lowest level (around 10%) found in cocoa bean husks²⁴ and the highest (36–40%) in citrus²⁵. The cellulose contents in date fibres was similar to those reported by Jiménez-Escrig et al. (2001)²³ for guava pulp and Fuentes-Alventosa et al. (2009a)¹² for asparagus. The range of lignin content from other products was wide, varying from 6% for peach pulp²⁷ to 32% for cocoa bean husks²⁴.

The neutral sugar composition of DF was also studied (**Table 2**). Xylose was the main sugar, accounting for nearly 50% of the molar percentage. Other sugars of interest were arabinose (between 17-22%) and galactose (8-16%). Mannose and glucose were

near 5%, and rhamnose and fucose were found in lower amounts. Elleuch et al. (2008)⁹ reported higher percentages of rhamnose and galactose than those found in our varieties. These differences could be related to higher amounts of uronic acids also quantified in those samples. Moreover, pectins, xylans and arabinoxylans are the main non-cellulosic polysaccharides present in date DF, representing approximately 75% of the total neutral sugars. This composition is very similar to that of other lignocellulosic agricultural by-products. Some authors have compared it to hardwoods and straws rather than softwoods, which contain large amounts of mannose²².

Water- and oil-holding capacities and solubility of date fibre.

The results obtained for water-holding capacity, solubility and oil-holding capacity are presented in **Table 3**. Water-holding capacity is an important property of DF from both a physiological and technological point of view. The water-holding capacity of date DF ranged from 12.65 to 17.22 mL water/g fibre. The Kenta variety had the highest value, and Deglé Nour, Mermella, Rochdi and Matteta exhibited non-statistical differences with respect to water-holding capacity. Other authors⁹ reported a capacity of 15.5 mL water/g fibre for other varieties, value that is in the range above cited. We can argue that these other common varieties could have the same physiologically beneficial effects than Deglé Nour. However, if we consider those varieties as not suitable for human consumption, they could be a good source of fibre for food formulations. The results found in this work are similar or even higher than those reported for most described by-product fibres, e.g., 15.8 mL water/g for fibre from asparagus by-product¹² and 12.6 mL water/g for peach pulp fibre²⁷. Other agricultural by-products have lower values than those mentioned above, e.g., cocoa husks²⁴, which have a water-holding capacity value of 5 mL water/g fibre. Dates fibres have very low solubility, from 0.11 to 0.23%. These results are related to the high rate of insoluble to

soluble fibres in date flesh. In almost all of the reported data ^{9, 18}, the percentage of insoluble fibre was higher than 95%. The soluble and insoluble nature of DF involves differences in their technological functionality and physiological effects. Insoluble fibres are characterized by their porosity, their low density and by their ability to increase faecal bulk and decrease intestinal transit. Based on these values, date fibre could be used as a modifier of viscosity and texture of formulated products in addition to promoting the decrease in calories that this addition could imply.

The oil-holding capacity results are presented in **Table 3**. Eguwa, Garen Gaze and Korkobbi had the highest capacity (higher than 4 mL/g fibre), which was much lower than those found in a previously cited work: approximately 9.7 g/g fibre ⁹. These authors applied a force of 1,500 g to determinate the oil-holding capacity instead of the 14,000 g we applied to our samples. The values found in the literature for other by-products, e.g., 0.6-1.8 g/g for apple pomace and citrus peel ⁶ were much lower than those that we reported for date fibre. Thus, the use of this fibre may be appropriate in products that require emulsifying properties.

Polyphenol content and antioxidant activity of date fruits.

The polyphenol content and antioxidant activity of both fractions, soluble and linked to fibre, are presented in **Table 4**. Deglé Nour was the variety that had the highest soluble polyphenol content (221 mg GA/100 g FW). From the coastal growth zone, Bouhattam, Korkobbi, and Eguwa were richer in these compounds than the other varieties, followed by Kenta, Limsi, Matteta, Garen Gaze, Smeti, Mermella, and Rochdi. Besides variety and growth zone, the soluble polyphenol content also depends on date humidity. There are important differences between soft, semi-dry and dry dates. Biglari, AlKarki and Easa (2008, 2009) ^{28, 29}, who worked with Iranian dates, found that the polyphenol content in soft dates varies from 2 to 4 mg GA/100 g FW, in semi-dry

from 4 to 6 mg GA/100 g FW, and in dry 141 mg GA/100 g FW. Similar results were published for Algerian soft dates³⁰, between 2-8 mg GA/100 g FW, and for Omani dry dates⁵, from 172 to 246 mg GA/100 g FW. Chaira et al. (2009)³¹, who worked with Tunisian dates from some of the varieties presented in this study, found a smaller soluble polyphenol content (22-110 mg/100g FW), most likely due to differences in the extraction and quantification methods. In agreement with our work, they found that Korkobbi, Bouhattam and Kenta had the highest polyphenol content (they did not analyze Eguwa cultivar).

The antiradical activity of the soluble fraction was studied, the results of which are presented in **Table 4**. As was the case with the polyphenol content, Deglé Nour had the highest activity (50 mmol Trolox/Kg FW). Korkobbi and Bouhattam cultivars had nearly half of the Deglé Nour activity (28.68 and 24.60 mmol Trolox/Kg FW, respectively). The lowest values were those found for Rochdi and Mermella (3.07 and 3.18 mmol Trolox/Kg FW, respectively). These results are also in agreement with those published by Chaira et al. (2009)³¹. Although little interferences that sugars and other compounds could have on Folin-Ciocalteu quantification method, there was good correlation between the soluble polyphenol content and antiradical activity ($R = 0.8451$), as has been reported by other authors^{28, 30, 31}.

In this work, we also studied the phenol composition and antiradical activity of the insoluble fraction. This fraction could be very important regarding the total antioxidant activity of fruits and vegetables because, as Saura-Calixto (2011)³² suggested, the transportation of antioxidants through the gastrointestinal tract is an essential physiological function of DF. This aspect has received very little attention so far. Bound phenolics were extracted from all samples and identified and quantified by HPLC. The results are presented in **Table 4**. Ferulic acid was the most abundant simple

phenol in all of the samples, except for Korkobbi and Eguwa in which vanillin was the major phenol. Ferulic acid ranged from 14.35 to 56.35 mg/100g fibre in the Matteta and Deglé Nour varieties, respectively. The other phenolic acid identified, *p*-coumaric acid, was the minor component in almost all of the samples. The total amount and composition varied significantly among the samples, as reported by Al-Farsi et al. (2005)¹⁸, who worked with Omani dates.

Because we found that date pulp contained an interesting amount of phenols linked to fibre, we considered that these compounds could confer additional antioxidant activity that was not measured with the soluble extracts. In **Figure 1**, the antiradical activity of date fibre is presented. In this figure, the higher slope in the regression line indicates the higher antioxidant activity. The assayed fibres were soluble phenol-free, so their activities should be due exclusively to compounds present in the fibre fraction. There was a group of five varieties (Deglé Nour, Garen Gaze, Smeti, Mermella, and Eguwa) that had the highest activities. Bouhattam, Limsi, Kenta, Rochdi, and Korkobbi had an intermediate level of free-radical scavenging activity, Matteta being the least active variety. In **Table 4**, these levels of activity are expressed as mmols Trolox/Kg fibre. The variety with the highest activity linked to fibre was Smeti; the order of the remaining samples with regard to this activity was as follows: Smeti > Mermella > Garen Gaze = Eguwa > Deglé Nour >> Rochdi > Kenta > Korkobbi > Limsi = Bouhattam >> Matteta. In the fibre fraction, there was no correlation between the polyphenol content and antiradical activity. In prepared food, which undergoes a thermal process, a non-enzymatic brown reaction takes place, leading to the formation of a complex series of compounds called Maillard reaction products. The antioxidant activity of these compounds has been reported in several studies³³. In date palm fruits, this reaction was mentioned by Barneveld (1993)³⁴. In our work, these compounds

could also contribute to the antioxidant activity of the fibre fraction; thus, a more detailed study on date Maillard reaction products should be conducted. The most interesting thing is that four Tunisian varieties had high antiradical activity, even higher than that of the Deglé Nour variety. The fibre from these date varieties could be considered “antioxidant DF”, as defined by Saura-Calixto (1998)³⁵ because they have a free radical scavenging capacity higher than 50 mg of vitamin E (measured by the DPPH· method), and this activity is derived from natural components of the material. **Figure 2** shows the total antioxidant activity expressed on a fresh weight basis. It is clear that Deglé Nour is the best variety, with the highest activities (soluble and linked to fibre). However, fibre from Garen Gaze, Smeti, Mermella, and Eguwa cultivars had an activity similar to or even higher than that of Deglé Nour. These are the Tunisian secondary varieties that could be promising from a technological point of view.

In summary, the DF content of ten Tunisian date varieties from Gabès coastal oases have been studied for the first time and compared to that of Deglé Nour, the most commercially accepted variety. Although Tunisian secondary varieties have similar DF content and composition, they are not very suitable for human consumption. Therefore, our study also focused on possible technological applications. The most important characteristic supporting the use of some of these varieties as food ingredients is their antioxidant activity. The level of activity found in the fibre of Garen Gaze, Smeti, Mermella and Eguwa varieties makes them valuable as potential sources of antioxidant fibre. Further studies on the responsible compounds (maybe Maillard reaction products) for this characteristic are needed, and also a technological approach for the obtaining of this antioxidant DF from date fruit. The use of these secondary varieties in the food industry as healthy ingredients could help in the fight against the reduction in vegetal genetic variability. In addition, the growth of these cultivars for technological purposes

388 may play an important role in the economic and social level of the people from this
389 developing region.

390

391 **Abbreviations Used**

392 DF: dietary fibre; MES: 4-Morpholineethanesulfonic acid; TRIS:

393 tris(hydroxymethyl)aminomethane; DPPH: 2,2-diphenyl-1-picrylhydrazyl free radical;

394 GA: gallic acid; FW: fresh weight.

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FIGURE CAPTIONS

Figure 1.- Dose-response lines of antiradical capacity of date fibres from different cultivars. Antiradical capacity is expressed as percent DPPH· remaining in solution after 30 min of reaction. Each individual point in the graph is the average value of three replicates.

DN-Deglé Nour, Bh-Bouhattam, GG-Garen Gaze, L-Limsi, Mt-Matteta, Kt-Kenta, Rch-Rochdi, St-Smiti, Mr-Mermella, Kor-Korkobbi, Eg-Eguwa.

Figure 2.- Total antiradical activity expressed as mmol Trolox/Kg fresh weight of date fruits from different cultivars.

DN-Deglé Nour, Bh-Bouhattam, GG-Garen Gaze, L-Limsi, Mt-Matteta, Kt-Kenta, Rch-Rochdi, St-Smiti, Mr-Mermella, Kor-Korkobbi, Eg-Eguwa.

Table 1.- Dietary fibre content (expressed as % fresh pulp) and composition (g/100g fibre) of different date fruit cultivars.

Cultivar	Dietary fibre	Neutral sugars	Uronic acids	Cellulose	Lignin
Rochdi	4.76 ± 0.02 ^a	20.23 ± 1.45 ^b	16.60 ± 1.60 ^f	21.81 ± 1.91 ^{de}	35.03 ± 1.82 ^{ab}
Matteta	4.79 ± 0.09 ^a	25.06 ± 1.89 ^{de}	14.88 ± 1.62 ^{de}	24.82 ± 1.59 ^e	33.35 ± 1.27 ^a
Korkobbi	5.11 ± 0.06 ^b	20.38 ± 0.49 ^b	13.25 ± 0.73 ^{bc}	21.63 ± 1.41 ^{cd}	42.27 ± 0.33 ^{de}
Eguwa	5.85 ± 0.07 ^c	15.56 ± 0.39 ^a	13.40 ± 0.85 ^{bc}	17.01 ± 1.51 ^a	50.17 ± 0.44 ^g
Bouhattam	6.05 ± 0.16 ^d	20.44 ± 1.35 ^b	14.12 ± 1.01 ^{cd}	19.12 ± 1.21 ^{bc}	38.67 ± 0.28 ^c
Mermella	6.53 ± 0.02 ^e	20.50 ± 0.04 ^b	16.71 ± 0.87 ^f	19.80 ± 1.39 ^{bc}	39.19 ± 0.46 ^c
Limsi	6.62 ± 0.16 ^{ef}	25.71 ± 2.75 ^e	13.04 ± 0.85 ^b	18.16 ± 1.16 ^a	42.95 ± 0.09 ^e
Kenta	6.71 ± 0.03 ^{ef}	22.23 ± 0.60 ^{bcd}	15.50 ± 1.34 ^e	20.86 ± 2.41 ^{cd}	35.92 ± 2.02 ^b
Deglé Nour	7.23 ± 0.10 ^g	16.22 ± 0.16 ^a	10.74 ± 0.90 ^a	19.67 ± 1.66 ^c	50.37 ± 1.28 ^g
Garen Gaze	7.24 ± 0.11 ^g	24.89 ± 2.34 ^{cde}	12.90 ± 1.05 ^b	22.38 ± 1.86 ^{de}	46.08 ± 1.65 ^f
Smeti	7.26 ± 0.03 ^g	21.78 ± 1.50 ^{bc}	13.44 ± 0.90 ^{bc}	18.20 ± 1.33 ^b	40.58 ± 0.23 ^{cd}

Values are means of triplicate assays.

Means bearing the same letter are not significantly different at 5% level as determined by the Duncan multiple range test.

Table 2.- Neutral sugar composition of date fruit dietary fibre (expressed as molar percentage).

Cultivar	Rha	Fuc	Ara	Xyl	Man	Gal	Glu
Rochdi	3.62 ^e	2.05 ^c	20.52 ^{bcd}	50.28 ^{ab}	4.47 ^{abcd}	14.12 ^{cd}	4.92 ^a
Matteta	1.97 ^{ab}	1.69 ^{ab}	20.44 ^{bcd}	55.22 ^{bcd}	6.07 ^d	9.42 ^{ab}	5.19 ^{ab}
Korkobbi	2.42 ^{bc}	1.66 ^{ab}	19.03 ^{abc}	52.46 ^{abc}	4.48 ^{abc}	13.97 ^c	5.98 ^{abc}
Eguwa	2.82 ^{cd}	1.74 ^b	17.2 ^a	51.35 ^{abc}	5.13 ^{bcd}	16.37 ^{ef}	5.32 ^a
Bouhattam	2.07 ^{ab}	1.40 ^a	22.80 ^d	48.34 ^a	3.83 ^{abc}	16.22 ^{def}	5.34 ^{abc}
Mermella	2.83 ^{cd}	1.71 ^{ab}	17.40 ^{ab}	52.78 ^{abcd}	3.75 ^{abc}	16.52 ^{ef}	5.02 ^a
Limsi	1.57 ^a	1.61 ^{ab}	17.68 ^a	54.64 ^{cd}	8.03 ^e	8.46 ^a	8.01 ^c
Kenta	2.59 ^{bc}	1.69 ^{ab}	17.8 ^{ab}	49.29 ^a	5.20 ^{cd}	15.31 ^{cde}	8.08 ^{bc}
Deglé Nour	2.58 ^{bcda}	1.63 ^{ab}	21.27 ^{cd}	54.78 ^{bcd}	4.50 ^{abcd}	10.63 ^b	4.61 ^a
Garen Gaze	3.33 ^{de}	2.04 ^c	21.42 ^{cd}	57.26 ^d	3.38 ^a	8.99 ^{ab}	3.57 ^a
Smeti	2.67 ^{bcd}	1.48 ^{ab}	16.9 ^a	53.89 ^{abcd}	3.62 ^{ab}	17.42 ^f	3.99 ^a

Values are means of triplicate assays.

Means bearing the same letter are not significantly different at 5% level as determined by the Duncan multiple range test.

Rha: rhamnose; Fuc: fucose; Ara: arabinose; Xyl: xylose; Man: mannose; Gal: galactose; Glu: glucose.

Table 3.- Functional properties of date fruit fibre from different cultivars.

Cultivar	WHC	% SOL	OHC
	mL water/g fibre		mL oil/g fibre
Rochdi	16.23 ± 0.24 ^{def}	0.23 ± 0.01 ^f	3.84 ± 0.29 ^{cd}
Matteta	15.96 ± 1.25 ^{def}	0.15 ± 0.01 ^c	3.53 ± 0.07 ^{bc}
Korkobbi	14.77 ± 0.63 ^{bcd}	0.15 ± 0.00 ^c	4.21 ± 0.08 ^{ef}
Eguwa	12.65 ± 0.75 ^a	0.10 ± 0.01 ^a	4.46 ± 0.11 ^f
Bouhattam	14.56 ± 0.41 ^{bc}	0.18 ± 0.00 ^d	3.83 ± 0.38 ^{cd}
Mermella	16.45 ± 0.95 ^{ef}	0.20 ± 0.01 ^e	4.11 ± 0.18 ^{de}
Limsi	15.63 ± 0.49 ^{cde}	0.13 ± 0.02 ^b	3.72 ± 0.16 ^{bc}
Kenta	17.22 ± 0.92 ^f	0.14 ± 0.01 ^{bc}	3.38 ± 0.09 ^b
Deglé Nour	16.54 ± 1.47 ^{ef}	0.17 ± 0.01 ^d	3.64 ± 0.28 ^{bc}
Garen Gaze	13.01 ± 0.64 ^a	0.11 ± 0.01 ^a	4.25 ± 0.14 ^{ef}
Smeti	13.48 ± 1.05 ^{ab}	0.23 ± 0.00 ^f	3.01 ± 0.31 ^a

Values are means of triplicate assays.

Means bearing the same letter are not significantly different at 5% level as determined

by the Duncan multiple range test.

WHC: water holding capacity; SOL: solubility; OHC: oil holding capacity.

Table 4.- Phenol contents and antioxidant activities of different date fruit cultivars.

Cultivar	Soluble fraction		Fibre fraction				
	Phenol content mg GA/100 g FW	Antiradical activity mmol Trolox/Kg FW	Vanillin mg/100g	<i>p</i> -coumaric acid mg/100g	Ferulic acid mg/100g	Total phenols mg/100g	Antiradical activity mmol Trolox/Kg fibre
Rochdi	28.96±2.30 ^a	3.07±0.63 ^a	39.57±7.63	26.98±11.11	49.27±7.64	115.82±11.10 ^h	104.32±2.71 ^d
Matteta	84.72±7.17 ^{cd}	11.58±0.98 ^{cd}	10.39±0.95	17.39±1.33	56.35±3.04	84.12±0.77 ^{ef}	38.75±4.13 ^a
Korkobbi	145.01±8.17 ^f	28.68±5.32 ^f	42.99±1.08	5.62±2.23	21.25±0.82	69.86±1.97 ^{cd}	73.98±0.67 ^{bc}
Eguwa	146.65±13.41 ^f	9.95±2.13 ^{bcd}	50.74±7.48	8.64±0.99	35.51±1.79	94.88±8.28 ^g	232.30±2.39 ^f
Bouhattam	164.50±13.40 ^g	24.60±1.51 ^e	25.83±1.81	6.52±1.04	31.43±1.38	63.77±4.23 ^{bc}	68.74±0.23 ^b
Mermella	41.20±3.79 ^a	3.18±0.51 ^a	23.37±0.96	24.10±2.37	28.81±1.53	76.28±4.86 ^{de}	278.68±3.03 ^g
Limsi	94.48±11.70 ^{de}	8.82±1.25 ^b	25.85±1.46	15.90±0.12	45.40±0.46	87.15±0.88 ^{fg}	71.94±2.07 ^b
Kenta	96.62± 9.57 ^e	9.66±1.13 ^{bc}	25.67±0.15	12.01±0.52	35.33±0.88	73.01±0.51 ^{cd}	79.45±1.50 ^c
Deglé Nour	221.32±18.35 ^h	50.82±4.80 ^g	11.90±0.43	8.11±0.07	14.35±0.63	34.36±0.13 ^a	203.21±0.81 ^e
Garen Gaze	79.40±6.60 ^c	12.00±2.81 ^d	25.23±0.97	6.07±1.32	33.16±0.22	72.12±1.95 ^{cd}	230.06±6.25 ^f
Smeti	66.22±3.23 ^b	8.04±0.50 ^b	17.92±0.83	13.89±1.51	27.39±0.29	59.19±2.63 ^b	301.36±10.23 ^h

Values are means of at least duplicate assays.

Means bearing the same letter are not significantly different at 5% level as determined by the Duncan multiple range test.

Figure 1

Figure 2

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