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5	Valorization of date palm biodiversity: Physico-chemical composition,
6	phenolic profile, antioxidant activity, and sensory evaluation of date pastes
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Abstract

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This work is a contribution to the valorization of the Algerian date palm agrobiodiversity by studying some nutraceutical properties of date pastes from less important cultivars. A commercial date paste. prepared from Ghars variety, was compared to two date pastes prepared from the secondary cultivars Tamjouhert and Tazarzeit. Physico-chemical characteristics, phenolic profile (HPLC-DAD), and antioxidant activity were assessed for both date fruits and pastes. The sensory evaluation of date pastes was conducted through triangular, ranking, and hedonic tests. The results revealed that the transformation of date fruits into pastes caused a significant increase in phenolic content and antioxidant capacity, a decrease in sugar content in parallel with the appearance of hydroxymethylfurfural and furfural. The elaborated date pastes were characterized by a relatively low level of sugar (72.17-74.14 mg/100g), a high amount of phenolic compounds (>290 mg/kg DW), and a potent antioxidant activity. Seventeen phenolic compounds were identified by HPLC with global quantities for date fruits of 113 (Tamjouhert cv.) and 100 mg/kg DW (Tazarzeit cv.) and that were increased twofold after transformation to date pastes. The sensory analysis for the obtained date pastes indicated high scores of acceptability in comparison with the commercial paste. The obtained results showed clearly that these secondary cultivars possess several potentialities; this may help to valorize them by turning a part of their production into several added-value products such as date past.

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- Keywords Agrobiodiversity; Date Paste; Physico-chemical Composition; Phenolic Profile; Antioxidant
- 45 Activity; Sensory Evaluation.

Introduction

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Apart from the nutritional point of view; date fruits (*Phoenix dactylifera* L.) represent one of the most important commercial crops worldwide by an annual world production of 8.16 million tons [1]. Thereby, this high production has recently opened new opportunities to turn the surplus traditionally and/or industrially into a wide range of products and by-products; counting: alcohol, yeast, juice, jam, flour, mayonnaise, polyol, syrup, jelly, vinegar, and date paste [2]. The increasing interest in date fruit and its derived products is due to their highly nutritious composition that is rich in sugar, vitamins, minerals as well as fibers [3, 4]. On the other hand, it contains low amounts of fat and protein with no starch [5]. Moreover, these fruits are rich in phenolic compounds possessing several biological properties; including antimicrobial, antioxidant, and anti-inflammatory activities [3, 5, 6]. Among all the processed dates listed above, date paste is the most widespread considering its profitability, simplicity of preparation, and wide use. Date paste was initially prepared in order to conserve soft date fruits that were steamed and mixed to get a paste that can be stored for more than two years [7-9]. This paste is commonly used in bakery and confectionery; it can be also incorporated in several food preparations; precisely bologna-type meat, fruit bars, and muffin [10-13]. Admittedly, date pastes possess many nutraceutical properties since date fruits, used as raw material, are rich in essential nutrients and bioactive compounds [1, 4-6, 14]. Interestingly, it constitutes a novel and attractive vehicle of probiotics [15]. Indeed, date paste had been poorly studied; to our knowledge, limited data is available regarding their phenolic content [16]. However, their phenolic pattern and antioxidant activity had not been studied. The existing works focused on their physico-chemical composition [17, 18], microbial, technological, rheological, and textural characteristics [7, 16, 19, 20]. Harrak et al. [19] evaluated and improved the traditional process of making date paste by focusing on physic-chemical, sensory (texture; flavor, color, taste, and acceptability), and microbial characterization of the obtained paste. Abekhti et al. [7] described the dynamic changes of microbial and chemical parameters (pH, water content) of dates during transformation to paste and its storage. On the other hand, many recent works focused on the potential use of date paste as sugar substitute in several food preparations including muffin [12] and fruit bars [13],

jam [21], bread [22], and yogurt [23]. The fortified foods were then analyzed for their nutraceutical and organoleptic properties.

Algeria is among the first date-producing countries (1.09 million tons) with a high agrobiodiversity potential but not all the date cultivars were evaluated for their performance and quality. *Deglet-Nour* and *Ghars* cultivars are widely cultivated in Algeria due to their high commercial values, which cause the biodiversity erosion of other cultivars. Hence, paste production from secondary cultivars could be viewed as an interesting way to contribute to the date palm agrobiodiversity preservation. In this context, by studying date pastes preparation, the current study was initiated in the framework of the valorization of two endemic cultivars, *Tamjouhert* and *Tazarzeit* grown in M'zab valley oases compared with *Ghars* cultivar mainly used for date paste production in Maghreb (Algeria, Morocco, and Tunisia). Thus, our work reports the proximate composition, the antioxidant activity, and the phenolic profile of two date fruits and pastes; as well as sensory evaluation of the prepared pastes compared to an industrial one. In addition, the effect of the preparation process on the composition and antioxidant activity evolution from fruit to the paste was assessed. All these issues are considered as a challenge with high nutritional, social, environmental, and economic stakes.

Materials and methods

Chemicals

Acetone (99%, CAS: 67-64-1), petroleum ether (99.9%, CAS: 64742-49-0), and methanol (99.80%, CAS: 67-56-1) were from VWR Prolabo (Fontenay-sous-Bois, France). Sodium carbonate (NaCO₃ 99.5%, CAS: 497-19-8), sodium nitrate (NaNO₃ 99.0%, CAS: 7631-99-4), aluminium chloride (AlCl₃ 97.0%, CAS: 7446-70-0), sodium hydroxide (NaOH 97.0%, CAS: 1310-73-2), potassium ferricyanide (K₃(Fe(CN)₆) 99.0%, CAS: 13746-66-2), iron trichloride (FeCl₃ 98.0%, CAS: 7705-08-0); iron II chloride (FeCl₂ 98.0%, CAS: 13478-10-9), and trichloroacetic acid (TCA 98.0%, CAS: 76-03-9) were from Biochem Chemopharma (Georgia, USA). Folin-Ciocalteu's reagent was from Biochem Chemopharma (Montreal, Quebec). 2,2-Diphenyl-1-picrylhydrazyl (DPPH 90%, CAS:1898-66-4), hydrogen peroxide (H₂O₂, CAS: 7722-84-1), and ferrozine (98%, CAS: 63451-29-6) were from Sigma-Aldrich (Sternheim,

- 100 Germany). Gallic (99.5%, CAS: 149-91-7), p-coumaric (98%, CAS: 501-98-4), ferulic (99%, CAS: 537-
- 101 98-4), and caffeic acids (98%, CAS: 331-39-5), rutin (94%, CAS: 207671-50-9), luteolin (98%, CAS:
- 102 491-70-3), isoquercetrin (97%, CAS: 482-35-9), quercetrin (95%, CAS: 522-12-3), and quercetin (98%,
- 103 CAS: 117-39-5) were from Sigma-Aldrich Co (Saint-Louis, USA).

Plant material

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- The study was carried out on two local date fruit cultivars: *Tamjouhert* (DFT) and *Tazarzeit* (DFZ);
- 106 collected from M'zab Valley, Ghardaia, Algeria (32°29'N 3°41'E). Full ripe fruits (1 Kg) with uniform
- size, free of physical damage, insect injury, and fungal infection were selected.

Preparation of date pastes

- The preparation of the date paste was carried out according to the modified method of Ahmed *et al.* [17].
- 110 Two date pastes were aseptically prepared from Tamjouhert (DPT) and Tazarzeit (DPZ) cultivars
- following the industrial procedure. Briefly, about 500g of pitted dates were cooked under steam for 15
 - minutes. After cooking, the whole paste was mixed with 5 ml of sunflower oil and 5 ml of orange
- blossom water and then blended at speed 2000 rotations per minute (rpm) for 4 min (Fig. 1A). Three
- 114 replicates were prepared of each cultivar. In addition to these two pastes, a third date paste, freshly
 - prepared from *Ghars* cultivar (DPG) in accordance with the preparation process used for the preparation
- of DPT and DPZ, was obtained from an Algerian producer and was also studied as a standard for
- 117 comparison. The use of a commercial date paste as a reference will make it possible to compare the two
- other elaborated date pastes (DPT and DPZ) with a marketed product and to get closer to the consumer,
- especially concerning the sensory perception, and the samples (Fig. 1B) were conserved in sterilized glass
- iars at 4°C for further analyses.

Physico-chemical composition

- The physico-chemical characterization was performed on both date fruits and date pastes. The pH was
- measured according to Abekhti et al. [7] using a pH meter (Inolab, Munich, Germany) at 20°C. The
- moisture content was determined by drying two grams (2 g) of each sample in a vacuum oven (Memmert
- GmbH + Co.KG, Schwabach, Germany) at 70 ± 2 °C for 48h [24]. The total ash was measured according
- to El Arem *et al.* [25], One gram of each sample was incinerated in a muffle furnace (Nabertherm GmbH,

Lilienthal, Germany) at 530°C for 5h. Total sugar content was determined spectrophotometrically by phenol-sulfuric acid reaction [26]. Total nitrogen was determined by Kjeldahl method, which is used to determine protein content using a conversion factor of 6.25 [24]. The lipid fraction was extracted with petroleum ether at 60°C for 6 h using Soxhlet apparatus (Velp Scientifica, Milan, Italy). The solvent was finally removed using a rotary evaporator (Buchi, Flawil, Switzerland) for determining the fat content [27]. All the analyses were carried out by triplicates and the results were expressed as mg/100g of dried weights.

Determination of bioactive compounds

Extraction of bioactive compounds

The extraction of bioactive compounds was carried out according to Al-zoreky and Al-tahar [28] with slight modifications. Briefly, four grams of each date fruit or date paste were homogenized with 20 ml of methanol: water (65:35, v:v) for 2 min using an ultra-turax T25 homogenizer (IKA-labortechnik, Breisgau, Germany). The extracts were then centrifuged at 4000g (Sigma 2-16K, Osterode, Germany) for 5 min, paper filtered, and finally stored at 4°C for further analyses.

Determination of total phenolic content

The date fruits and date pastes total phenolic content (TPC) were determined according to Hachani *et al*.

[6] by using the Folin-Ciocalteu spectrophotometric method. Two hundred microliters of each extract were mixed with 1.5 ml of Folin-Ciocalteu reagent (1:10). After 5 minutes, 1.5 ml of sodium carbonate (Na₂CO₃ 7.5%) was added. The reaction mixture was incubated at room temperature in dark for one hour. The absorbance was measured at 760 nm. Results were expressed as milligrams of gallic acid equivalent per 100g of dried weight (mg GAE/100g DW).

Determination of flavonoid content

Total flavonoids content (TFC) of both date fruits and date pastes were measured calorimetrically according to Al Juhaimi *et al.* [29]. Extract (500 μL) was added to 150 μL NaNO₂ (5%) and 300 μL AlCl₃ (10%), respectively. After incubation at room temperature for 5 min, 1 ml of NaOH (1 M) was added. The absorbance was measured at 510 nm. Results were expressed as milligrams of quercetin equivalent per 100g of dried weight (mg QE/100g DW).

Determination of phenolic profile

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To determine the date fruits and date pastes phenols, twenty microliters (20 µL) of each extract was injected into HPLC-DAD (Teknokroma, Barcelona, Spain). The Analytical separation was carried during 55 min out on a reversed phase equipped with C18 column (Teknokroma Tracer Extrasil ODS-2, 250 mm-4.6 mm, i.d. 5 µm) in gradient system (eluent A: water/TCA 0.01%, eluent B: acetonitrile). The flow rate of the mobile phase was 1 ml/min [12]. The identification of compounds was done by comparing their retention times and UV spectra to standards as well as by running the samples after the addition of pure standards. The quantification of the tentatively identified phenolics was done using standard calibration curves [30]. The results were expressed as milligrams per kilogram referred to dry weight (mg/kg DW). The same protocol was also used for the determination of the two sugar degradation products (SDP): hydroxymethylfurfural (HMF) and furfural.

Determination of antioxidant activity

- Prior to determining the antioxidant activity (AOA), the obtained methanolic extracts were evaporated
- using N₂ and then diluted in water and finally filtered. The resultant extracts were used to assess
- antioxidant activity through different tests:

169 Ferric reducing power (FRP)

- 170 The FRP was carried out using the method of Oyaizu [31]. Five hundred microliters of each extract were
- added to 1.25 ml of potassium ferricyanide (1%) and 1.25 ml of phosphate buffer (0.2 M, pH 6.6). The
- solution was incubated at 50°C for 20 min, and then 1.25 ml of TCA (10%) was added. The mixture (1.25
- 173 ml) was combined with 1.25 ml of distilled water and 0.25 ml of FeCl₃ (1%) before the absorbance
- 174 recording at 700 nm. The results were expressed as milligrams gallic acid equivalent per 100g (mg
- 175 GAE/100g DW).

176 **DPPH** radical scavenging capacity

- 177 The free radical scavenging capacity was carried out as described by Al Juhaimi et al. [29]. Sixty
- microliters of each extract were added to 1.5 ml of DPPH solution (6 10⁻⁵ M). The mixture was incubated
- in the dark for 30 min at room temperature. The absorbance was recorded at 515 nm. The DPPH radical
- inhibition was determined using the following formula:

- 181 I % = $[(A_0 A_1) / A_0] * 100$
- I %, inhibition percentage; A_0 , absorbance of the control; A_1 , absorbance of the sample extract.
- 183 Ferrous ion chelating capacity (FIC)
- The FIC was determined as reported by Ramchoun et al. [32]. Each extract (250 μL) was mixed with 25
- 185 μL of FeCl₂ (2 mM) and 800 μL of distilled water. The mixture was left for 5 min at room temperature
- then 50 µL of ferrozine (5 mM) were added, mixed, and left another 5min. The absorbance was measured
- at 562 nm. The ability of the sample to chelate ferrous ion was calculated using the following formula:
- 188 CE % = $[1 (A_1 A_2) / A_0] * 100$
- 189 CE, chelating percentage; A₀, absorbance of the control; A₁, absorbance of the sample extract; A₂,
- absorbance of the sample without ferrozine.
 - Sensory analysis

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- 192 The sensorial characterization of the three date pastes was realized according to ISO 4120 [33], which
- requires that n/3 should be greater than 5. Triangular, ranking, and hedonic tests were carried out, each
- one, on eighteen (18) panelists, aged from 18 to 60, and randomly selected among the students and the
- staff of the University.
- The three tests were realized according to Watts *et al.* [34]. To make it easier and to reduce the tasters'
- 197 confusion, we avoided the discussion about the samples but we explained to them the method and the
- protocols that must be used. We also recommended them not only to avoid using products with strong
- odors (soaps, lotions, and perfumes) but also to avoid eating, drinking, or smoking at least 30 minutes
- before proceeding to the tests. We provided them a survey that everyone had to fill in. Just one tasting
- session has been done. However, the different tests were done separately with a time difference of one
- 202 hour; during which we presented the samples, explained the test, distributed the survey, and answered the
- 203 questions asked by the tasters. Moreover, samples re-tasting was allowed for all three tests. The three date
- 204 pastes submitted for these analyses were labeled as follows:
 - "A": The reference date paste prepared from *Ghars* variety (DPG).
- "B": The date paste prepared from *Tamjouhert* variety (DPT).
 - "C": The date paste prepared from *Tazarzeit* variety (DPZ).

Triangular test

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- 209 The triangular test is commonly used to determine the ability of tasters to distinguish the differences in
- 210 the appearance, smell, flavor, or texture of foods [34]. We presented simultaneously three samples,
- among which one was repeated, and the trained participants were asked to determine the unrepeated one.
- The pastes were submitted according to the following positions: (A-A-B), (A-A-C), (B-B-A), (B-B-C),
- 213 (C-C-A), (C-C-B), (A-B-A), (A-C-A), (B-A-B), (B-C-B), (C-A-C) and (C-B-C) in identical sample
- 214 containers coded with 3-digit random numbers. All three code numbers on the samples presented to each
- 215 panelist were different, even though two of the samples are identical.
- 216 Two triangular proofs were carried out, the first relating to the external aspect and the second to the
- taste. These proofs use a binomial approximation that is based on calculating a " μ " value as follows:
- 218 $\mu = (|3x n| 1.5) / \sqrt{2n}$
- N, number of tasters; x, number of correct answers; the statistical decision is taken considering the
- following possibilities: if $\mu > 1.64$: Differences are significant at 5%; if $\mu > 2.33$: Differences are
- significant at 1%; if $\mu > 2.81$: Differences are significant at 1 %.

222 Ranking test

- 223 The ranking test aims to determine the degree to which the consumer accepts a product and therefore its
- real consumption. We submitted the three samples simultaneously in independent sample containers,
- labeled A, B, and C, then we asked the trained tasters to evaluate them singly by giving them rank from
- one to three ascendingly according to their acceptance, taking into account that equality is not accepted.
- The sample that has the lowest rank sum is the most preferred and vice versa. Three classification criteria
- were used: consistency, sweetness, and aroma.
- The ranks assigned to each sample were summed and then the differences between all possible pairs in
- 230 the summed rankings are compared to a critical value, cited in the table of critical absolute rank sum
- differences for "all treatments" comparisons at 5% [35].

232 Hedonic test

- 233 This test is done to establish a sensory profile for each sample, based on descriptors mainly: the texture
- (sliceable, spreadable, brittle, or liquid), the taste (bitter, sweet, salty, and acid), the color, and the smell.

Samples were submitted separately and the untrained tasters were asked to taste them and to give each one a score from 0 to 9 depending on the intensity of each descriptor mentioned above. The simultaneous presentation of the samples was adopted to allow re-evaluating the samples if desired and make comparisons between the samples.

Statistical analysis

All analyses were carried out in triplicate and the results were expressed as means \pm standard deviation. Statistical analyses were performed using XLSTAT 7.1 (Addinsoft, USA) and the differences at p< 0.05 were considered statistically significant. The Student-t test was performed to compare the results of two fruit samples. Analysis of variance (ANOVA_T-Tuky) followed by Dunnett test was used to determine the differences between the elaborated date pastes and the reference one. The Principal Component Analysis (PCA) served to visualize the correlation between the physico-chemical parameters and the sensory profile as well as between bioactive compounds and antioxidant capacity of the samples.

Results and discussion

Physico-chemical characterization

The proximate composition of the analyzed samples is given in Table 1. The two date fruits presented globally the same physic-chemical characteristics. The transformation to date pastes was accompanied by some modifications particularly a significant (p<0.05) augmentation of pH and moisture and fat contents well sugar level was decreased. Carbohydrates were the predominant components with concentrations varying between 66.10 and 74.14 mg/100g DW in date pastes and from 79.82 to 82.53 mg/100g DW in date fruits. Moisture content ranged from 10.63 (DFZ) to 17.17 mg/100g DW (DPG). Both date fruits and pastes were characterized by low amounts of proteins and fat. Some SDP such as HMF (27.14 - 45.82 mg/kg) and furfural (0.57 - 1.31 mg/kg) were also detected in pastes.

It was observed that the fat and the moisture contents increased significantly in date pastes in comparison with fruits, with about 47 and 30%, respectively. Moreover, the transformation caused a slight increase of pH (4.3%). On the other hand, the sugar content diminished considerably (10%) while protein and ash contents remain stable.

The physico-chemical composition of the analyzed fruits is within the previously published values. Rahman *et al.* [24] highlighted that Omani date fruits are slightly acid (5.6 - 5.8) while their sugar fraction is around 62%. Chibane-Amellal [8] reported that water and protein contents of Algerian cultivars are ranging from 13.03 and 2.18% (*Mech Degla*) to 14.80 and 3.50% (*Frezza*), respectively. All the other studies reported that fat and ash contents do not exceed 2%.

Regarding the proximate composition of the date pastes, Sánchez *et al.* [16] reported similar protein, fat, and mineral contents (2%); lower sugar content (53%), and higher moisture content (34%). On the other hand, the pastes analyzed by Abekhti *et al.* [7] are characterized by nearby water contents (9.09-11.58%) and slightly acidic pH (5.45-5.84).

The same trend of variation in the physico-chemical composition after transformation of date fruits into pastes was reported in the literature. Abekhti *et al.* [7] reported also a significant increase in pH and moisture contents by 2.51, 1.83, 4.02, and 4.95, 4.32, 4.04%, respectively in *Lmaiz*, *Abani*, and *Mtafra* Algerian date pastes after transformation of the fruits. With regard to fat content, Parn *et al.* [13] highlighted an average increase of 2.54% in fruit bars made with date paste. In their studies about Tunisian date pastes, Mrabet *et al.* [18] reported also the diminution of the sugar fraction by 25%.

Moreover, the changes in the physico-chemical composition after transformation are certainly due to the preparation process. Indeed, the increase of pH may be attributed to blending during which organic acids (citric, malic, and oxalic acids) may be released [10]. Similarly, the increasing amounts of fat and water contents are due to the addition of sunflower oil and moisturization by steam, respectively. However, the diminution of sugar content could be explained by the cooking process which may cause their caramelization and Maillard reaction [5] as well as the degradation of a little fraction of carbohydrates to HMF and furfural [14]. Likewise, the diminution of ash and protein contents is probably due to the addition of the organic material from the sunflower oil and the orange blossom water and moisturization. The apparition of SDP in date pastes could be explained by the degradation of sugars under the thermal treatment applied when preparing them. According to Mrabet *et al.* [12], high amounts of HMF were found when using the steam treatment. The lowest amounts of furfural could be explained by its volatility [14].

Total Phenolic content

The results of total phenolic and flavonoid contents were summarized in Table 2. Date fruits presented similar levels on TPC and TFC. The transformation of date fruits to pastes was accompanied by a significant increase in TPC by about 10% but no effect was observed for TFC. Regarding elaborated date pastes, their TPC were higher than the reference (DPG) while their TFC were lower.

Very high polyphenol amounts were previously detected in Algerian cultivars that varied from 247 to 1394 mg GAE/100g [30, 36, 37, 38]. According to Ghnimi *et al.* [5], the variations observed between cultivars may be due to genetic, stage of ripening, storage conditions, and extraction procedure. Moreover, the phenolic fraction of our date pastes is higher than that reported by Sánchez *et al.* [16] who found only 225 mg GAE/100g. Regarding the effect of the transformation on the phenolics, its increase after heating and steaming was previously reported [14, 26]. Likewise, these findings were also detected for other fruits and vegetables such as tomato [39], eggplants [40], strawberry [41], and cocoa [42]. Actually, the hydrothermal treatment, employed during date paste preparation, helps to solubilize a higher quantity of phenols from date fruits [43]. In addition, heating of dates may facilitate the leakage of some phenols, mainly phenolic acids and anthocyanins [2].

Antioxidant activity

whatever was the test.

The antioxidant potential was assessed using FRP, DPPH, and FIC and the results were given in Table 2. As clearly shown in the table, all the samples exhibited a potent AOA, which varied significantly (p<0.05) among date fruits as well as among date pastes that exhibited the strongest antioxidant potential

Date fruits of *Tamjouhert* cultivar demonstrated higher DPPH scavenging capacity and ferrous ion chelating capacity than the fruit of *Tazarzeit* cultivars. Whereas, this latter had a higher ferric reducing power than the first one. The elaborated date pastes presented higher significant antioxidant activity measured by FRP and FIC than corresponding date fruits, but DPPH scavenging activity remained stable after the transformation.

The Algerian date fruit cultivars are known to possess strong AOA as mentioned by several studies. The AOA ranges from 32.4 to 86%, from 25.29 to 41.67 mg EAG/100g, and from 170.4 to 948.1 mg EAA/100g respectively, as tested by DPPH scavenging capacity [28, 36, 37].

It was reported that antioxidant activity of thermally treated dates increased significantly compared to the date fruit [43-45]. However, these results disagree with those of Parn *et al.* [13] who reported a slow decrease of antioxidant capacity of date paste, as tested by DPPH scavenging capacity, upon its incorporation in fruit bars. Similarly, numerous studies established that heat treatment enhanced the antioxidant activity of tomato, eggplants, strawberry, and cocoa [39-42].

Admittedly, the strong AOA of date fruits is due to the presence of enzymatic antioxidants (catalase, peroxidase, and superoxide dismutase) and non-enzymatic antioxidants, mainly phenolic compounds [46]. Hence, it is admitted that these compounds are responsible for the antioxidant power of date paste especially, non-enzymatic activity. This is confirmed by the increase of the AOA in date pastes, compared to date fruits, in parallel with the increase of their phenols (TPC and changes in phenolic profiles).

This was confirmed by strong correlations between bioactive compounds (TPC and TFC) and AOA (DPPH, FRP, and FIC) of analyzed samples with coefficients of determination ranged from 0.70 to 0.95 (Table I, supplementary material). These results are in line with those of Mansouri *et al.* [47] and Biglari *et al.* [48] who reported that the antioxidant potential of date fruit is due to the presence of phenolic compounds and consequently their potent contribution to the antioxidant capacity of date pastes. Nevertheless, this activity may depend on the available concentration of an individual antioxidant compound or on the potential synergistic interaction that occurs in various constituents of plants [49, 50].

Phenolic profile

The phenolic profiles of date fruits and pastes were determined using HPLC-DAD and the results were presented in Table 3. All the analyzed samples shared quietly the same phenolic pattern. The main identified compound was gallic acid (2.44-29.36 mg/kg DW). *Tamjouhert* cultivar was distinguished by significantly high contents on fives compounds (catechin and vanillic, syringic, sinapic, and ferulic acids) than *Tazarzeit* cultivar. This latter exceeded DFT only in tyrosol content. The other phenolic compounds

were equal for the two date fruits. Interestingly, the transformation of date fruits to date paste caused a significant increase in almost all of the phenolic compounds, mainly gallic acid, PHBA, vanillin. In parallel luteolin decreased significantly, especially in DPZ with 80% loss. It can be seen also that the two elaborated date pastes were globally more concentrated on phenolics than the reference.

Unlike date fruits, date pastes' phenolic profile was poorly studied. In this context, more or less similar phenolic patterns have been listed for other date fruit varieties. Mansouri *et al.* [47] identified *p*-coumaric, ferulic, sinapic, and cinnamic acids; however, they have not been quantified. Similarly, Benmeddour *et al.* [30] identified and quantified some flavonoids including isoquercetin (13.33-51.03%), rutin (19.10% - 39.90%), quercetin (16.05- 53.78%), and luteolin (0.64- 4.49%). Likewise, Mrabet *et al.* [12] detected certain phenolic acids, namely gallic (major compound), protocatechuic, vanillic, and *p*-coumaric acids as well as tyrosol, while Hachani *et al.* [6] detected 23 phenolic compounds, most of them are hydroxycinnamic and formic acid derivatives. These authors did not detect gallic acid except in methanolic extracts while luteolin was only detected in acetone-aqueous extracts.

These results were in line with those of Allaith $et\ al.\ [43]$ and Mrabet $et\ al.\ [12]$ who indicated the increase of some date phenols after steam and heat treatments. In addition, Rubio-Sentent $et\ al.\ [51]$ detected new phenols in thermally treated olive oil that were not present in the untreated reference sample. According to Homayouni $et\ al.\ [2]$, the increasing level of phenolic acids is due to chain-breaking effect of heat treatment, the breakdown of cell walls, and hydrolysis of linkages between bound acid and lignin or arabinoxylans caused the release of phenolic acids. On the other hand, Ghnimi $et\ al.\ [5]$ explained the diminution of some phenols by their destruction under high temperatures because they are heat-labile. Nevertheless, Mrabet $et\ al.\ [12]$ reported that gallic acid contents decrease in thermal extracts at very high temperatures $(180-200^{\circ}C)$.

Sensory analysis

Triangular test

The statistical decisions and μ values for both the external appearance and the taste of the three date pastes were given in Table II (supplementary material). It is indicated that DPG (A) had an external appearance that was significantly different from that of DPZ (C), regardless of its position of presentation.

On the other hand, its external appearance was significantly different from that of DPT (B), especially when it was presented after. However, DPT (B) and DPZ (C) had similar external appearances.

Regarding the taste, sample "A" had a taste that is significantly different from that of sample "C", regardless of its position of presentation. On the other hand, its taste was significantly different from that of DPT (B) only when it was presented before. Finally, the DPT (B) and DPZ (C) had similar tastes (Sensory records of the triangular test were given in table III of supplementary material).

Ranking test

The participants ranked the three samples according to their consistency, sweetness, and aroma, and the attributed ranks were presented in Table 4. For the three sensory descriptors, the panelists assigned the first rank to DPZ (C) followed by DPT (B) while the sample DPG (A) is ranked the last one.

In parallel, the differences between the paired rank totals (A - B), (A - C), and (B - C) were calculated and the results were mentioned in Table 5. According to Friedman's table, the critical value for 18 tasters and 3 samples is 15. Thus, differences between the pairwise classification totals (D) show that there is no significant difference (D < 15 differences) between the consistencies of the pairs (DPT-DPZ) and (DPG-DPT). For the other pair (DPG-DPZ), the difference was significant $(D \ge 15)$. In terms of sweetness and aroma, the three samples show no significant differences since the differences between the total paired ranks were all less than 15 (Sensory records of the ranking test were given in table IV of supplementary material).

Hedonic test

Sensory profile

The obtained results, presented in Fig. 2, showed that panelists perceive that sensory attributes describing texture and color were the most intense in contrast with those describing smell and taste where a low intensity was noted for each of their descriptors, except the sweet taste. Indeed, DPZ had a sliceable and spreadable texture and a considerable sweetness. On the other hand, DPT had a brittle texture, low spreadability, and the best color, while DPG had a semi-solid texture, an acid taste and best smell.

The statistical analysis showed significant differences (p< 0.05) between the three samples. The Dunnett test mentioned clearly that the texture, taste and the smell of DPT and DPZ were significantly (p< 0.05)

share the same color, which was significantly different from that of DPT. Furthermore, the mean classification test (T-Tukey) reveals their separation into two heterogeneous groups. The first group includes the sample "A" while the second group includes the samples "B" and "C". This means that DPG was significantly different from DPT and DPZ, which were similar in terms of almost all the studied descriptors (Sensory records of the hedonic test were given in table V of supplementary material).

Correlation between the sensory profile and the proximate composition

- The PCA allows visualizing the correlation between physico-chemical parameters and sensory attributes.
- The obtained results (Fig. 3) showed that they are closely correlated as it was clearly appearing that the
 - sweety taste was positively correlated with the sugar content (r = +0.958). Admittedly, carbohydrates
 - were the main molecules that were responsible for the food's sweetness. Being the major components of
 - the date fruit, sugars render the date paste very sweet.
- The color had also a positive correlation with sugar content (r = +0.855). Indeed, in addition to
- 408 pigments such as carotenoids, anthocyanins, and tannins, carbohydrates contribute to date color through
- 409 Maillard and caramelization reactions [5]. Regarding texture, the spreadable texture and pH were
- 410 positively correlated (r = +0.866). In fact, after heating, when the pH increased the protein-protein,
- 411 protein-fat, and protein-water interactions increase. Hence, the product may have a soft and elastic texture
- 412 [52].

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- On the other hand, the pH is negatively correlated (r = -0.953) with the brittle texture. When the pH
- decreases, the friable texture could result from the weakness of protein-protein and protein-water
- 415 interactions [53].
- Likewise, a positive correlation (r = +0.782) is observed between the liquid texture and the moisture
- 417 content. Actually, the decrease in moisture content causes poor protein hydration and results in a less
- elastic paste that is more susceptible to breaking forces [24]. Thus, this was explained by the plasticizing
- 419 effect of water contained in dates fruit itself and that acquired during the steaming preparation of date
- 420 paste.

Furthermore, the variations of the salty taste can be attributed to samples' ash content (r = +0.609), which presents a more intense salty taste with the increase in the mineral content, especially NaCl [54].

A similar trend of positive correlation was observed between odor and fat content (r = +0.951). Since volatile compounds responsible for the aroma were mainly lipophilic, it is recognized that the intensity of the flavor increased with the increase of fat content. In fact, the volatile compounds including alcohol, aldehydes, esters, terpenes, and ketones are the key volatiles determinants of the date aroma [55]. These flavor volatiles are derived from an array of compounds, via different pathways such as terpenes pathway and some of them (alcohols) are mainly generated as reaction products of lipid oxidation [25].

Conclusion

The preparation of date paste from date fruits resulted in a dough with higher bioactive compounds amounts and lower sugar content as well as appreciated texture, taste, color, and smell. In fact, both date fruits and pastes exhibited promising nutraceutical potentialities. The prepared date pastes showed high scores in consumer's acceptability and preference in comparison with the commercial paste. Relying upon the obtained results; the transformation of secondary date varieties, mainly *Tazarzeit* and *Tamjouhert*, into paste constitutes a useful boost for their valorization.

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Conflict of interest

- The authors declare that there is no conflict of interest.
- 449 Availability of data and material
- 450 Not applicable
- 451 Code availability
- 452 Not applicable
- 453 **Authors' contributions**
- 454 *Malika Tassoult:* Conceptualization, Methodology, Software, Validation, Formal analysis, Investigation,
- Data curation, Writing- Original draft, Visualization.
- 456 *Djamel Edine Kati:* Conceptualization, Methodology, Resources, Supervision; Project administration.
- 457 *Mustapha Bachir bey:* Software, Validation, Formal analysis, Visualization.
- 458 Ali Benouadah: Validation, Resources, Project administration.
- 459 Guillermo Rodriguez-Gutierrez: Conceptualization, Methodology, Resources, Supervision; Project
- administration.
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541	Figure Captions
542	
543	Fig. 1: Diagram of date paste preparation from date fruits (A) and photographs of date fruits and pastes
544	(B).
545	DPG, Reference date paste prepared from Ghars cultivar (DFG); DPT, date paste prepared from Tamjouhert cultivar (DFT);
546	DPZ, date paste prepared from Tazarzeit cultivar (DFZ).
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550	Fig. 2: Hedonic profile of date pastes.
551	The hedonic profile of the three date pastes (DPG, DPT, and DPZ) was studied using a 9- point scale for the each descriptor of
552	the following sensory attributes: texture, taste, color and smell. DPG, Reference date paste prepared from Ghars cultivar;
553	DPT, date paste prepared from Tamjouhert cultivar; DPZ, date paste prepared from Tazarzeit cultivar, For each attribute,
554	different letters indicate statistical significant difference (ANOVA-Dunnett test, p <0.05, b< a).
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558	Fig. 3: Principal Component Analysis between sensory profile and physico-chemical composition of date
559	paste.
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Table 1. Physico-chemical composition of date fruits and pastes

	Date f	ruits	Date pastes			
	DFT	DFZ	DPT	DPZ	DPG	
рН	5.77 ± 0.04^{b}	5.74 ± 0.10^{b}	6.02 ± 0.02^{a}	$5.98 \pm 0.02^{a(-)}$	6.03 ± 0.02	
MC (%)	11.75 ± 0.35 ^{b(*)}	10.63 ± 0.03^{b}	15.52 ± 0.29 ^{a(-}	13.77 ± 0.25 ^{a(-}	17.17 ± 0.48	
SC (%)	79.82 ± 2.14^{a}	82.53 ± 1.58^{a}	72.17 ± 1.52 ^{b(+)}	74.14 ± 2.94 ^{b(+)}	66.10 ± 0.88	
PC (%)	1.87 ± 0.04^a	1.50 ± 0.32^a	$1.82 \pm 0.08^{a(-)}$	1.44 ± 0.04^{a}	1.25 ± 0.14	
FC (%)	1.31 ± 0.07^{b}	1.33 ± 0.07^{b}	$1.90 \pm 0.04^{a(-)}$	1.97 ± 0.10^{a}	2.11 ± 0.04	
AC (%)	2.54 ± 0.25^{a}	2.45 ± 0.10^{a}	$2.48 \pm 0.02^{a(-)}$	$2.37 \pm 0.02^{a(+)}$	2.30 ± 0.03	
HMF (%)	nd	nd	3.01 ± 0.21	$4.58 \pm 0.12^{(+)}$	2.71 ± 0.32	
Furfural (%)	nd	nd	$0.05 \pm 0.01^{(-)}$	$0.08 \pm 0.01^{(-)}$	0.13 ± 0.02	

Each value in the table is the mean ± standard deviation (n=3); Result of date fruit with asterisk (*) is statistically higher than that of the other one (Student-t test); Results of the same cultivar (fruit/paste) with different letters are statistically different (Student-t test, p>0.05, a>b); Results of date pastes (DPT or DPZ) and control (DPG) were compared with Dunnett test (-/+ significantly lower/higher at p<0.05); MC, moisture content; SC, sugar content; PC, protein content; FC, fat content; AC, ash content; DFT, date fruit of *Tamjouhert* cultivar; DFZ, date fruit of *Tazarzeit* cultivar; DPG, control date paste prepared from *Ghars* cultivar; DPT, date paste prepared from *Tamjouhert* cultivar; DPZ, date paste prepared from *Tazarzeit* cultivar; nd, net detected.

Table 2. Total phenolic and flavonoid contents and antioxidant activities of date fruits and pastes

	Date fi	ruits	Date pastes			
Parameter						
	DFT	DFZ	DPT	DPZ	DPG	
Phenolic content (mg/10	0g DW)					
TPC (mg GAE/ 100g DW)	268.04 ± 6.50^{b}	264.87 ± 6.12^{b}	$288.37 \pm 0.99^{a(+)}$	$298.11 \pm 7.44^{a(+)}$	281.47 ± 1.54	
TFC (mg QE/ 100g DW)	16.11 ± 0.19^{a}	17.36 ± 1.38^{a}	$17.17 \pm 0.95^{a(-)}$	$18.15 \pm 1.92^{a(-)}$	28.27 ± 0.89	
Antioxidant activity						
FRP (mg GAE /100g DW)	766.97 ± 9.16^{b}	$927.55 \pm 0.39^{b*}$	809.29 ± 9.73^{a}	$1005.16 \pm 2.60^{a(+)}$	806.16 ± 2.60	
DPPH (%)	$69.89 \pm 1.98^{a^*}$	55.77 ± 0.53^{a}	$70.39 \pm 1.38^{a(-)}$	$58.52 \pm 3.42^{a(-)}$	75.46 ± 0.60	
FIC (%)	$71.20 \pm 0.03^{b*}$	70.39 ± 0.18^{b}	$78.49 \pm 3.51^{a(+)}$	$75.60 \pm 1.87^{a(+)}$	69.17 ± 1.45	

Each value in the table is the mean ± standard deviation (n=3); Result of date fruit with asterisk (*) is statistically higher than that of the other one (Student-t test); Results of the same cultivar (fruit/paste) with different letters are statistically different (Student-t test, p>0.05, a>b); Results of date pastes (DPT or DPZ) and control (DPG) were compared with Dunnett test (-/+ significantly lower/higher at p<0.05); TPC, total phenolic content; TFC, total flavonoids content; FRP, Ferric reducing power; DPPH, free radical scavenging activity; FIC, Ferrous ion chelating capacity; DFT, date fruit of *Tamjouhert* cultivar; DFZ, date fruit of *Tazarzeit* cultivar; DPG, control date paste prepared from *Ghars* cultivar; DPT, date paste prepared from *Tamjouhert* cultivar; DPZ, date paste prepared from *Tazarzeit* cultivar; GAE, gallic acid equivalent; QE, Quercetin equivalent; DW, dried weight.

 Table 3. Phenolic profile of date fruits and pastes

Content (mg/kg DM)	Date fr	ruits	Date pastes			
_	DFT	DFZ	DPT	DPZ	DPG	
Gallic acid	2.49±0.11 ^b	2.44±0.38	29.36±0.20 ^{a(+)}	24.09±1.32 ^a	25.02±0.18	
O-Coumaric acid	2.44±0.02 ^b	Traces	12.18±0.13 ^{a(+)}	4.88±0.07 ⁽⁻⁾	8.25±1.00	
Protocatechuic acid	9.34±0.42 ^a	6.69±3.16 ^b	12.62±2.02 ^a	16.9±0.07 ^{a(+)}	12.68±1.29	
Tyrosol	8.17±0.2 ^b	9.63±0.42 ^{b*}	16.37±0.23 ^a	16.12±0.29 ^a	16.54±0.08	
РНВА	5.22±0.27 ^b	3.79±4.85 ^b	14.70±0.19 ^a	18.89±0.05 ^{a(+)}	14.86±0.93	
Vanillic acid	$2.55 \pm 0.28^{b^*}$	1.45 ± 0.22^{b}	5.55 ± 0.14^{a}	5.31 ± 1.56^{a}	5.35 ± 0.09	
Syringic acid	$4.89 \pm 0.46^{a^*}$	3.7 ± 0.2^a	4.16 ± 2.11^{a}	4.81 ± 1.84^{a}	5.56 ± 0.11	
Caffeic acid	5.12 ± 1.16^{a}	4.71 ± 0.29^{b}	$6.53 \pm 0.05^{a(+)}$	$7.56 \pm 0.38^{a(+)}$	5.94 ± 0.24	
P-Coumaric acid	1.41 ± 0.07^{b}	0.8 ± 0.46^b	3.01 ± 0.09^{a}	$4.48 \pm 0.06^{a(+)}$	3.16 ± 0.06	
Sinapic acid	$6.7 \pm 0.22^{a^*}$	4.3 ± 0.70^{b}	$6.61 \pm 0.14^{a(-)}$	7.24 ± 0.01^{a}	7.45 ± 0.22	
Ferulic acid	$4.5 \pm 0.34^{b*}$	2.27 ± 1.33^{b}	7.77 ± 1.93^{a}	$7.53 \pm 0.41^{a(+)}$	6.05 ± 0.04	
Cinnamic acid	12.59 ± 0.28^{b}	11.79 ± 0.48^{b}	$18.68 \pm 0.16^{a(+)}$	$17.79 \pm 1.69^{a(+)}$	14.35 ± 0.54	
Catechin	$12.93 \pm 0.07^{b*}$	7.56 ± 0.06^{b}	$21.56 \pm 0.36^{a(-)}$	$15.86 \pm 0.43^{a(-)}$	22.91 ± 0.50	
Vanillin	3.01 ± 0.37^{b}	3.47 ± 0.54^{b}	$15.62 \pm 0.05^{a(+)}$	$13.45 \pm 0.16^{a(+)}$	12.12 ± 0.02	
Rutin	14.25 ± 2.04^{a}	19.35 ± 3.40^{a}	12.25 ± 0.19^{a}	13.93 ± 1.73^{a}	10.18 ± 3.26	
Luteolin	16.27 ± 1.04^{a}	16.23 ± 0.43^{a}	$14.27 \pm 0.16^{b(+)}$	9.02 ± 0.48^{b}	10.4 ± 1.27	
Quercetin	1.21 ± 0.05^{b}	1.27 ± 0.16^{b}	$6.25 \pm 0.02^{a(-)}$	$4.81 \pm 0.01^{a(-)}$	7.10 ± 0.02	

587	Each value in the table is the mean ± standard deviation (n=3); Result of date fruit with asterisk (*) is statistically higher than that of the
588	other one (Student-t test); Results of the same cultivar (fruit/paste) with different letters are statistically different (Student-t test, p>0.05,
589	a>b); Results of date pastes (DPT or DPZ) and control (DPG) were compared with Dunnett test (-/+ significantly lower/higher at p<0.05)
590	DFT, date fruit of Tamjouhert cultivar; DFZ, date fruit of Tazarzeit cultivar; DPG, control date paste prepared from Ghars cultivar; DPT,
591	date paste prepared from Tamjouhert cultivar; DPZ, date paste prepared from Tazarzeit cultivar; PHBA, phosphor-hydroxybenzoic acid.
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Table 4. Ranks attributed to the three date pastes

Samples		DPG			DPT			DPZ	
Aspects	Consistency	Sweetness	Aroma	Consistency	Sweetness	Aroma	Consistency	Sweetness	Aroma
Ranks sum	48	43	44	37	34	32	23	33	31
Rank	3	3	3	2	2	2	1	1	1

DPG, Reference date paste prepared from *Ghars* cultivar; DPT, date paste prepared from *Tanjouhert* cultivar; DPZ, date paste prepared from *Tazarzeit* cultivar.

Table 5. Differences between rank total pairs of pastes

Pairs	DPG – DPT	DPG - DPZ	DPT – DPZ
Consistency	11*	25**	14*
Sweetness	09*	10*	01*
Aroma	12*	13*	01*

DPG, Reference date paste prepared from *Ghars* cultivar; DPT, date paste prepared from *Tanjouhert* cultivar; DPZ, date paste prepared from *Tazarzeit* cultivar; * insignificant; ** significant (p>0.05)

Figure 1

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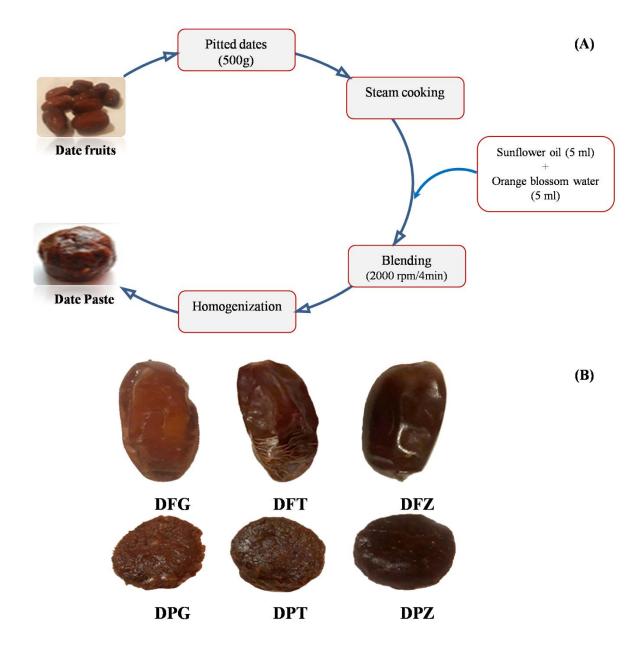


Figure 2

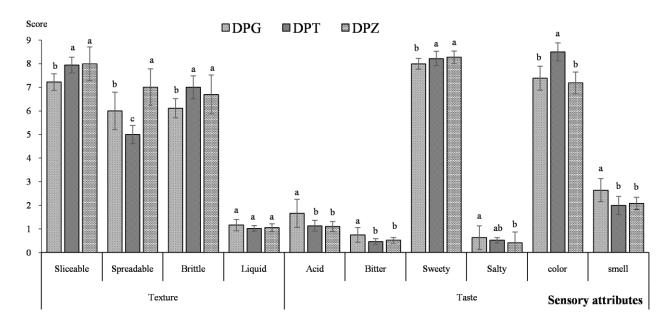


Figure 3



