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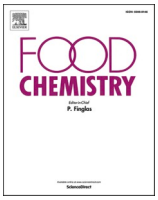

Does hydration of 'Deglet Nour' date palm fruits improve their quality and help to reduce waste?

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ABSTRACT

This work investigates the quality change of date palm fruits after hydration treatment which is commonly applied to enhance the hard textured 'Deglet Nour' fruits that are unacceptable for consumption. Date palm fruits were treated at 60–62 °C with saturated steam for 4 h in three different processing units (DPU). Mid Infrared Spectroscopy (MIR) giving a global spectral evaluation discriminates samples from the three DPUs and highlights date palm fruits of the first DPU regarding hydration treatment. Treatment led to a decrease of fruit firmness, skin lightness, and of sucrose and malic acid contents whereas citric acid and procyanidins contents and procyanidins 'degree of polymerization increased. Thermal treatment had no effect on glucose and fructose contents, on cell wall content and composition and on minor phenolic groups. Significant differences existed on dates from the three DPUs, discriminating dates presenting high firmness. Hydration treatment improve dates texture as expected while nutritional parameters were quite stable, confirming that is very promising and could be highly recommended to valorise fruit that are currently not commercialized. However, optimisation is needed for the very hard-type dates.

1. Introduction

Date palm fruits (*Phoenix dactylifera* L.) have high nutritional, biochemical and physico-chemical characteristics (Ahmed, Al-Gharibi, Daar, & Kabir, 1995; Al-Shahib & Marshall, 2003; Ismail, Haffar, Baalbaki, Mechref, & Henry, 2006). They are consumed fresh or in various processed forms (Besbes, Drira, Blecker, Deroanne, & Attia, 2009; Jridi et al., 2015).

Date palm fruits, and in particular 'Deglet Nour' cultivar, do not ripe at the same time, even in the same bunch, which leads to several harvests during the harvesting season (Awad, 2007). Moreover, combined with irregular climatic conditions and no proper timing of harvest, the harvested fruits can present a poor commercial quality because of their unacceptable texture (over-dried or very soft), pest infestation and damages (Kader & Hussein, 2009). Hence, almost 30% of the date palm production is lost or wasted at some steps along the food supply chain (Masmoudi et al., 2008) because they do not meet market specification and consumers' expectations especially for texture.

To valorise these secondary class dates (discarded dates with low commercial quality) and minimize wastes generated during processing, hydration treatment is commonly applied to too firm fruits to make them softer. To be declared as good quality, 'Deglet Nour'date fruits must be semi-soft presenting 30% of moisture content (Codex, 1985) and must be slightly to moderately elastic and chewy with smooth texture and mouth feel (Ismail, Haffar, Baalbaki, & Henry, 2001). Among the different treatments applied to dried dates, hot hydration treatment induces texture softening (Boubekri, Benmoussa, Courtois, & Bonazzi, 2010; Kader & Hussein, 2009; Yahia, Lobo, & Kader, 2014).

It is therefore necessary to evaluate the properties of the date palm quality according to the processing conditions. Fruit softening is always related to cell wall modifications (Awad, Al-Qurashi, & Mohamed, 2011; Brummell, 2006), especially pectins which are modulated by the action of cell wall associated enzymes (El-Zoghbi, 1994). Fruit colour is also modified after hot treatments, dates becoming susceptible to darken

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(Ben-Amor, Dhouibi, & Aguayo, 2016) which could be explained by an oxidative browning of phenolic compounds by polyphenol oxidase (PPO) in relation with the tissue destructuration. However, Deglet Nour' sucrose, glucose and fructose concentrations are stable after soaking at 45 °C (Boubekri et al., 2010). Concerning bioactive compounds, specifically total phenolic compounds, different studies have shown that they increased with heat treatment in many fruits i.e. apricot (Le Bourvellec et al., 2018), date palm (Siddiq & Greiby, 2013), apple, orange and grape (He et al., 2016) as cellular degradation after heat treatment make them more extractable.

No research evaluating the physical and chemical quality changes after hydration treatments currently applied in Tunisian date palm processing units is published to our knowledge. Thus, the aim of this study was to evaluate the organoleptic and nutritional quality changes of 'Deglet Nour' hard textured dates after hydration treatment applied in Tunisian date palm processing units for their better valorisation and to lower wastes.

2. Material and methods

2.1. Chemical

Polyphenol standards ((+)-catechin, (−)-epicatechin, 4-cafeoylshikimic acid, 5-cafeoylshikimic acid, rutin, isorhamnetin and chrysoeriol) were purchased from Extrasynthese (Lyon. France). Acetonitrile of HPLC grade and methanol were from Carlo Erba Reagents S.A.S (Val de Reuil. France), formic acid was from Sigma-Aldrich (Deisenhofen. Germany). Ethanol, acetone and sulfuric acid were from Fisher Scientific (Fair Lawn. NJ. USA). Neutral sugar standards (rhamnose, fucose, arabinose, xylose, mannose, galactose, and glucose) were from Fluka (Buchs. Switzerland). *N*-methylimidazole and acid anhydride were from Acros Organics (Geel. Belgium). Ammonium hydroxide solution (NH4OH) (33%). Sodium borohydride (NaBH4) and acetic acid were from Merck Chimie SAS (an affliate of Merck KGaA, Darmstadt, Germany).

2.2. Plant material including hydration treatment

Date palm (*Phoenix dactylifera* L.) samples of the 'Deglet Nour' cultivar were from Kebeli oasis (33° 42′ 7" North and 8° 58' 25" East) in the south of Tunisia. Fruits were picked at *Tamar* stage during the 2018 harvest season (October–December). Fruits were provided from three different date palm processing units (DPU) at Bni Khalad delegation (Tunisia), before and after treatment. The three DPUs used the same treatment devices with the same industrial parameters (time, temperature and humidity) described below in 2.3. Only hard-type dates (very dry dates with wrinkled skin) were visually selected. Once dates were received at the DPU, they entered the processing chain following basic steps as described by Yahia et al. (2014).

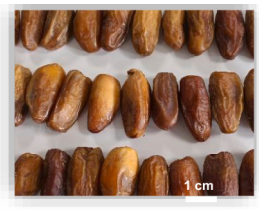
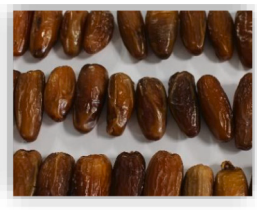
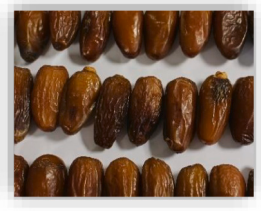
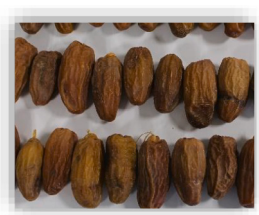
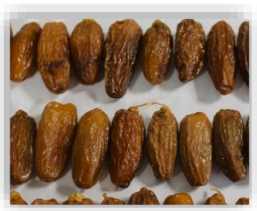
Firmer dates discarded after sorting in the supply chain were chosen according to their visual quality (Table 1). They were exposed per batch to saturated steam (100% humidity) at 60–62 °C during 4 h in a 30–50 m^3 capacity semi-automatic hydration room (KINKAI, Model JK10RD, Guangdong, China) with 2380 × 1370 × 1690 mm (L × W × H) dimension. Heating capacity was 35 kW allowing a hydration treatment of 40 L/h and about 400–500 kg per one batch. About 80 fruits (~1000 g) from the whole batch from each DPUs were collected at random as usually practiced, before and after hydration treatment, and were further divided in three biological replicates. Thus, 18 sample lots were selected from each DPU before and after treatment, and for each replicate. Date palm samples were then transported immediately after treatment in small plastic boxes to INRAE PACA (Avignon. France), where they were kept one night at 4 °C until characterization.

2.3. Sample characterization

2.3.1. Fruit firmness and colour

Colour and firmness were measured on the whole fruits the day after their reception. Whole fruit firmness was determined at room temperature, as a compression force on the two flat sides of 10 fruits chosen as representative among the 18 sample lots using a texturometer (Texture

Table 1
'Deglet Nour' dates from industrial batches before and after hydration treatment.

	Before hydration treatment	After hydration treatment
Date Processing Unit 1		
Date Processing Unit 2		
Date Processing Unit 3		

analyser TAplus, Ametek, Lloyd Instruments Ltd., Fareham, UK). Firmness was defined as the maximal force required to penetrate 3 mm in the date palm fruits with a 2 cm diameter probe at a descending speed of 20 mm/min, and was expressed in Newton (N). The CIE L* a* b* and hue (h*) and Chroma C* colour coordinates of the skin samples were measured on the two opposite sides of the 10 same fruits as the firmness test, using a CR-400 chromameter (Minolta Co. Ltd., Osaka, Japan). In order to estimate how human eye perceives the colour difference between samples, the colour differences ΔE*, ΔC* and ΔH* were calculated for each DPU between before and after treatment, and between each DPU two by two before and after treatment (DPU1 v. DPU2, DPU1 v. DPU3, DPU2 v. DPU3). ΔE* is defined as the arithmetic distance between the coordinates of two samples, ΔC* is defined as the difference between two samples chroma C, and ΔH* was calculated as:

$$\Delta H^{*} = \sqrt{\Delta E^{*2} - \Delta L^{*2} - \Delta C^{*2}} \quad (1)$$

with ΔL the difference between two samples lightness L.

2.3.2. Samples preparation

After colour and firmness measurement, samples were ground in liquid nitrogen using an IKA®A11 basic analytical mill (Ika Labortechnik, Staufen, Germany) in order to obtain a fine homogeneous powder. The powder was then frozen and stored at −80 °C until analysis for soluble sugars and organic acids. Samples used for polyphenols, cell walls and Mid Infrared Spectroscopy determination (MIR) were freeze-dried and stored at −20 °C until analysis.

2.3.3. Mid infrared spectroscopy

MIR Spectra were acquired at room temperature using ATR Tensor 27 FT-IR spectrometer (Bruker Optics, Wissembourg, France) equipped with a single-reflectance horizontal diamond crystal (Golden Gate. Bruker Optics) as described by Bureau, Ścibisz, Le Bourvellec, and Renard (2012).

2.3.4. Cell walls or Alcohol Insoluble Solids (A.I·S) preparation

Alcohol Insoluble Solids (AIS) were prepared according to previous works (Renard, 2005). AIS yields were expressed in mg/g of Fresh Weight (FW).

2.3.5. Analysis methods

2.3.5.1. Sugars and organic acids. Sugars (glucose, fructose and sucrose) and organic acids (malic acid and citric acid) were quantified as described in Bureau et al. (2012). Absorbance was measured at 340 nm and results were expressed in mg/g FW.

2.3.5.2. Neutral sugars, uronic acids, methanol and lignin contents of AIS. Neutral sugars, uronic acids and methanol were analysed as described by Renard and Ginies (2009). Results were expressed in mg/g AIS. The degree of methylation (DM) was calculated as the molar ratio of methanol to uronic acids. Lignin was analysed in AIS samples as described by Brahem, Renard, Gouble, and Le Bourvellec (2017). The amount of lignin was calculated from a linear calibration curve created with commercial alkali lignin. Results were expressed in mg/g AIS.

2.3.5.3. Polyphenols. Polyphenol were identified by HPLC-ESI-MS^2 and their quantification was determined by HPLC-DAD with or without thioacidolysis as described by Cherif, Le Bourvellec, Bureau, and Benabda (2021).

2.4. Statistical analysis

Data are reported as the mean ± pooled standard deviation (Pooled SD; Box, Hunter, & Hunter, 1978). Statistical analyses were established using XLSTAT package of Microsoft Excel. Significant differences ($p < 0.05$) between means and interactions between variables were evaluated by two-way ANOVAs. Principal Component Analyses (PCA) was applied to get an overview of the infrared spectral data discrimination according to hydration treatment and DPU and to interpret variable relationships.

3. Results and discussion

3.1. Global characterization of date palm by mid-infrared spectroscopy

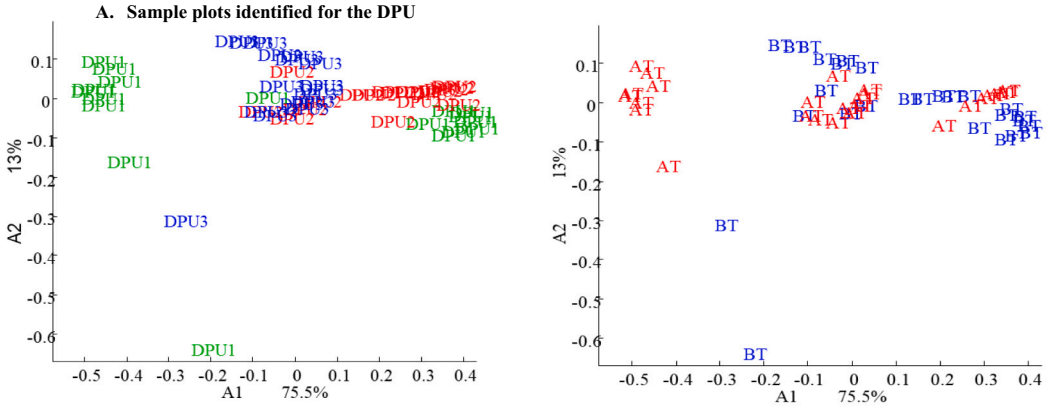
A Principal Component Analysis (PCA) was applied on the spectral data (2000–800 cm^{-1}) to discriminate date palm fruits according to the studied factors i.e., hydration treatment and DPU (Fig. 1 A and B). PC1 and PC2 components explained >88% of the total variance (75.5% for the PC1 and 13% for the PC2). PC1 discriminated samples as regards to the date palm processing unit. Samples of DPU_2 and DPU_3 were grouped, indicating a weak effect of DPU without any effect of the treatment. However, for DPU_1, two groups were observed with the first completely on the right corresponding to dates before treatment and the second completely on the left corresponding to dates after treatment. The eigenvectors allowed to identify the most discriminant spectral wavenumbers explaining the discrimination according to DPU and hydration treatment (Fig. 1B). The most discriminant wavenumbers explaining the discrimination of samples on PC1 were 987 cm^{-1} and 925 cm^{-1} for samples on the right and the minor bands at 1083, 1009 and 772 cm^{-1} for samples on the left (Fig. 1B). These wavenumbers illustrated the changes of the main components which are in dates in decreasing order: sugars, fibers, polyphenols, organic acids, minerals, proteins and fats (Abbès et al., 2011; Al-Farsi, Alasalvar, Morris, Baron, & Shaihdi, 2005; Al-Farsi & Lee, 2008; Elleuch et al., 2008). These wavenumbers incorporate typical bands of soluble sugars such the ones assigned to the C—O and C-OH stretch (900–1250 cm^{-1}), and organic acids assigned to O—C—H stretch (1180–1400 cm^{-1}) (Bureau, Cozzolino, & Clark, 2019). MIR global characterization showed variability of the dates due to the different DPUs, probably in link to their dry matter content. MIR highlighted an effect of hydration treatment only for dates from DPU_1, probably regarding their higher dry matter content and particularly to sucrose content as chemicals analysis had revealed below.

3.2. Effect of treatment on date palm physical properties and appearance

3.2.1. Firmness

Firmness values of fresh fruits ranged between 16 and 36 N depending on the DPU (Table 2). These firmness ranges are comparable to those reported by Boubekri et al. (2010) for 'Deglet Nour' dried dates. DPU_3 presented the firmest samples. The fruit origin significantly affected fruit firmness. These large differences could be related to specific sampling methods of each DPU, to agricultural practices depending on the oasis' farmers and to the fruit physiology at harvest.

In our experiment, as dates were hard textured type, hydration treatment affected significantly firmness values. Firmness decreased significantly after treatment for each DPU (Table 2). The highest significant decrease was by 40% for DPU_1 giving the softest dates after treatment. Ben-Amor et al. (2016) also reported a decrease in 'Deglet Nour' date palm firmness after hot water treatment at 50 °C for 10 min. For the hardest dates of DPU_3, the firmness decrease was only 13% (Table 2). Hydration treatment used in our study (60–62 °C during 4 h), led to soften dried dates to fit to human consumption (Table 1). However, this treatment was not effective enough for the very hard dates, which might need different temperature or more time under steam exposition. Since the relationship between date palm softening after hydration and taste acceptability by consumers are lacking and are not studied in our work, we tried to investigate sensorial analysis data from the comparative study of Ismail, Djendoubi, Kodia, Hassine, and Slama (2013). Tunisian'Deglet Nour' cultivar was the most appreciated and designed as a soft cultivar. So, based on this latter study, we could probably estimate that the hardest dates in our study (DPU_3), which still



With codes: BT: before treatment; AT: After treatment in the 3 Date Processing Units, DPU1, DPU_2 and DPU_3

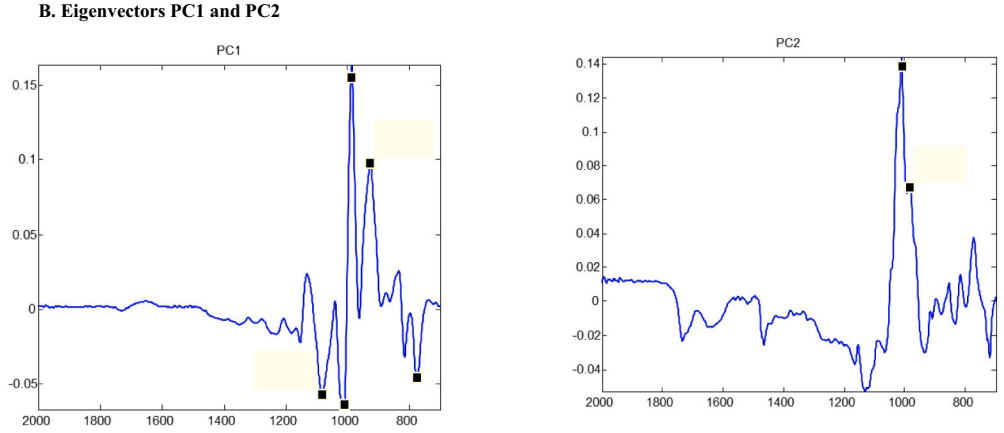


Fig. 1. PCA results on mid-infrared spectral data (2000 and 800 cm^{-1}) of 'Deglet Nour' date fruits before and after hydration treatment (HT) for the three Date Processing Units (DPU).
With codes: BT: before treatment; AT: After treatment in the 3 Date Processing Units, DPU1, DPU_2 and DPU_3.

Table 2
Firmness (N) and CIELAB colour parameters: Lightness (L*), Redness/Greenness (a*), Yellowish/Blueness (b*) of 'Deglet Nour' date fruits before and after hydration treatment (HT) for the three Date Processing Units (DPU). Statistical results (Pooled SD, two-way ANOVAs) and interaction effects between variables.

	Firmness	L*	a*	b*	h* (°)	C*
		Before treatment				
DPU 1	17.7	36.0	11.2	15.7	53.2	19.5
DPU 2	16.1	34.8	10.8	15.2	53.8	18.7
DPU 3	35.9	40.1	12.7	20.8	57.9	24.5
		After treatment				
DPU 1	10.7	33.1	11.4	15.9	53.3	19.7
DPU 2	12.4	32.5	12.2	15.7	51.7	20.0
DPU 3	31.0	35.0	13.5	20.2	55.9	24.4
Pooled SD	1.34	0.71	0.33	0.64	1.28	0.59
		Statistics: F-value and significance				
DPU	138	17	17	39	6	48
	***	**	**	***	*	***
HT	23	35	9	0.02	2	1
	**	***	*	ns	ns	ns
DPU*HT	0.8	2.1	1.6	0.4	0.4	0.8
	ns	ns	ns	ns	ns	ns

Pooled SD: pooled standard deviation, F-value: Fisher's value, *Significant at $p \leq 0.05$.
** Significant at ≤0.01, Significant at *** $p \leq 0.001$.

have a low commercial value although the hydration treatment, might conserve an acceptable taste and could therefore be incorporated in functional foods such as meat products, dairy products, and pastries (Martín-Sánchez et al., 2014).

3.2.2. Colour

The fruit visual appearance plays an important role in determining consumer acceptance. Before treatment, variability in date palm colour was observed depending on DPUs with the lightness degree (L*) between 34 and 40, the a* coordinate between 10.8 and 12.7 and b* coordinate between 15.2 and 20.8, h* between 53.2 and 57.9 and finally C* between 18.7 and 24.5 (Table 2). The colours here were similar to those reported by Ben-Amor et al. (2016) for 'Deglet Nour' date palm fruits, but were darker and less brown than those reported by Djouab, Benamara, Gougam, Amellal, and Hidous (2016) and brighter with red colour tendency than those reported by Hazbavi, Khoshtaghaza, Mostaan, and Banakar (2015). The difference observed could be due to date palm cultivars. The lightness difference between samples were slightly detected visually as shown in Table 1, where DPU_2 samples seems to be the darkest, may be because their wrinkled skin limiting their flat surface. L*, a*, b* values of DPU_3 were significantly different compared to other DPUs. This difference could be due to usual sampling practices in each industry but also to the hardest type of DPU_3 fruits having a very wrinkled skin causing a different surface examination (Table 1). When estimating the colour shift between DPUs two by two (Table 3), DPU1 and DPU2 showed similar colour ($\Delta E < 5$, considering 5 as the threshold above which the colour difference is considered significant and visible), whereas DPU3 stood out from the others ($\Delta E > 6.5$).

After hydration treatment, L* parameter was significantly reduced, especially in DPU_1 and DPU_3 (Table 2). Visually, date palm fruit colour changed from a light brown to a slight dark brown. Date palm samples from DPU_3, with the clearest skin colour, were the most affected by hydration treatment, showing the lowest L* value (decrease by 13%). The use of high temperatures (50–55 °C) usually increase colour

Table 3
Colour differences resulting from comparisons between processing unit before and after heat treatment, and between before and after hydration treatment for each fruit lot (processing unit and replicate).

Processing unit	Replicate	Hydration Treatment	Comparison	ΔE*	ΔH*	ΔC*
–	–	BT	DPU1 *v.* DPU2	1.38	0.00	0.80
–	–	BT	DPU1 *v.* DPU3	6.65	1.53	5.01
–	–	BT	DPU2 *v.* DPU3	7.91	1.00	5.80
–	–	AT	DPU1 *v.* DPU2	0.99	0.73	0.26
–	–	AT	DPU1 *v.* DPU3	5.16	1.11	4.66
–	–	AT	DPU2 *v.* DPU3	5.34	1.62	4.40
DPU1	1	–	BT *v.* AT	7.33	1.63	0.69
DPU1	2	–	BT *v.* AT	9.50	3.02	1.65
DPU1	3	–	BT *v.* AT	8.07	3.07	1.60
DPU2	1	–	BT *v.* AT	5.55	2.10	2.03
DPU2	2	–	BT *v.* AT	8.00	2.65	1.58
DPU2	3	–	BT *v.* AT	7.60	2.30	0.19
DPU3	1	–	BT *v.* AT	11.47	3.03	0.19
DPU3	2	–	BT *v.* AT	8.25	2.44	0.17
DPU3	3	–	BT *v.* AT	7.46	2.97	0.38

darkening in date palm fruit (Kader & Hussein, 2009). Ben-Amor et al. (2016) observe a decrease in lightness degree after date palm hot water treatment at 50 °C, 55 °C and 60 °C for 3 min. In our study, a significant increase was observed in a* value after heat treatment indicating a rise in red colour as a result of darkening skin. An increase of a* values was also reported after heating dates (İzlï, G., 2016). No significant differences were detected in b* coordinate. These results were not in accordance with those reported by Hazbavi et al. (2015), who shown a decrease in b* values after heating of 'Stamaran' dates. This variability could be due to the treatment conditions as well as the fruits and cultivars used. Ismail et al. (2013) demonstrated that Tunisian 'Deglet Nour' cultivar was the most preferred one, having an attractive colour. Thus, hydration treatment should be adapted to preserve this colour acceptability rate. This colour modification could be due to oxidative decomposition either enzymatically by polyphenol oxidase and peroxidase, or by non-enzymatic phenolic autoxidation or by thermal degradation of phenolic compounds, as a consequence of tissular and cellular disruption during thermal treatment. However, total phenolic contents in dates increased after treatment (Table 5). Thus, native compounds may not be involved in colour modification. Another explanation could be non-enzymatic reactions and/or degradation by temperature of maturation-induced compounds, i.e., compounds occurring during fruit maturation by oxidation of phenolics by PPO, hence already oxidized phenolic compounds, not identified nor quantified by HPLC-DAD with or without thioacidolysis due to their specific structure compare to native compounds. Their degradation leading to both L* and a* colour coordinates modification. Moreover, the differences in colour between DPUs remained after hydration (ΔE > 5 between DPU1 and DPU3, and between DPU2 and DPU3). The hydration treatment always induced a significant change of colour, as the lowest ΔE value was 5.55, however the hue (ΔH*) and chroma (ΔC*) remained similar before and after hydration treatment (Table 3).

3.3. Effect of treatment on fruit composition

3.3.1. Sugars and organic acids

Sucrose was the main sugar in 'Deglet Nour' date palm fruits followed by fructose and glucose in the same concentration (Table 4). Sucrose contents before treatment ranged from 372 (DPU_3) to 308 mg/g FW (DPU_1) followed by fructose up to 135 mg/g FW and glucose up to 105 mg/g FW for DPU_1. Our results are in the range of those published by Ben-Amor et al. (2016) for fresh date palm fruit, but higher than those reported by Al-Farsi and Lee (2008) and lower compared to those found by Elleuch et al. (2008) and Besbes et al. (2009) for date palm by-product. The differences observed could be due to fruit type and cultivar, locality and pedoclimatic conditions. Significant differences were observed between DPUs. The date palm fruits from DPU_3 presented the highest sucrose content and the lowest fructose and glucose contents before and after treatment (Table 4). These components participated to the already observed variability of date palm composition and appearance.

Table 4
Sugars, organic acids contents (mg/g FW) and dry matter (%) of 'Deglet Nour' date fruits before and after hydration treatment (HT) for the three Date Processing Units (DPU). Statistical results (Pooled SD, two-way ANOVAs) and interaction effects between variables.

	Sugars			Organic acids		Dry Matter
	Glucose	**Fructose**	**Sucrose**	**Citric acid**	**Malic acid**	
			Before treatment			
DPU 1	105	135	308	1.28	4.31	89.1
DPU 2	98	133	313	2.33	4.24	84.8
DPU 3	65	76	372	1.84	4.67	86.6
			After treatment			
DPU 1	125	114	172	2.60	3.22	86.8
DPU 2	103	130	256	2.95	3.98	85.5
DPU 3	83	81	335	1.44	5.12	87.4
Pooled SD	5.4	10.0	11.5	0.15	0.11	3.2
			Statistics: F-value and significance			
DPU	29 ***	16 **	49 ***	24 ***	57 ***	39 ***
HT	10 **	1 ns	66 ***	18 **	12 **	1 ns
DPU*HT	1 ns	1 ns	10 **	17 **	26 ***	15 **

Pooled SD: pooled standard deviation, F-value: Fisher's value, *Significant at $p \leq 0.05$.
** Significant at ≤0.01, Significant at *** $p \leq 0.001$.

Sucrose contents were affected by both hydration treatment and DPUs. They decreased after treatment for all DPUs. The highest decrease observed for DPU_1 was in accordance with the discrimination of the date palm samples before and after treatment obtained from their MIR spectral data (Fig. 1A). The sucrose decrease was accompanied only by glucose increase, fructose was stable, and not by both fructose and glucose increase as expected due to the action of invertase activity (Fayadh & Al-Showiman, 1990). This fact could probably be due to respiration which could be accelerated with heating combined with a slowly hydrolysis of sucrose, thereby explaining the variation between reducing sugars. Ismail, Haffar, Baalbaki, and Henry (2008) also shown the same trend on date palm fruit sugars behaviour during storage. Another indirect consequence of this phenomenon could be related to the decrease of dry matter after samples hydration, which was apparent only for DPU_1. Moreover, dry matter was only affected by DPUs and not by hydration treatment (Table 4). Ben-Amor et al. (2016) also report the same trend. Boubekri et al. (2010) also found a decrease in sucrose contents in 'Deglet Nour' dates. This decrease, contrary to our results, is accompanied by an expected simultaneous increase in fructose and glucose contents. The differences could be explained by the treatment applied, by the origin of date palm samples and/or by a basic metabolism pathway stimulated by hydration treatment conditions.

Malic acid, the main organic acid in 'Deglet Nour' date palm fruits, varied from 4.2 (DPU_2) to 4.7 mg/g FW (DPU3) before treatment, whereas citric acid contents did not exceed 2.3 mg/g FW for DPU_2 (Table 4). The predominance of malic acid is also revealed in both Egyptian (Youssef, El-Geddawy, El-Rify, & Ramadan, 1992) and

Emirates (Ghnimi et al., 2018) date palm cultivars but with lower contents. As the previous quality traits, significant differences were observed between DPUs for organic acid contents illustrating their variability.

Both malic and citric acids were affected by hydration treatment, but with opposite behaviours. For DPU_1, malic acid content decreased significantly after treatment whereas citric acid content increased. The decrease of malic acid may be due to its consumption as a respiratory substrate. Kim, Smith, and Lee (1993) reported lower total acidity in heated apple slices than the non-heated fruits, caused probably by the high respiration rate induced by the heat treatment. Titratable acidity decreases also after a hot water treatment for 15 min at 35, 45 or 55 °C of strawberries (Garcia, Aguilera, & Albi, 1995). Organic acids were also significantly affected by DPU after treatment which could be explained by the different responses of dates regarding their locality and pedo-climatic conditions.

3.3.2. Cell wall yields and composition

The AIS content (Table 5) of 'Deglet Nour' fresh date palm fruits ranged between 99.1 (DPU_2) and 121.4 mg/g FW (DPU_3) which was consistent with previously published works (Cherif et al., 2021; Mrabet et al., 2012). The two-way ANOVAs analysis showed significant differences in AIS content between the three DPUs ($DPU_2 < DPU_1 < DPU_3$). This difference could be due to the variability of sample quality belonging to different DPU as mentioned before for firmness and colour and also to DPUs dry matter values.

Lignin was the major component of fresh date palm AIS of the three DPUs, i.e., up to 173 mg/g AIS (DPU_3), followed by galacturonic acid (up to 140 mg/g CWM for DPU_2) and glucose coming from cellulose (up to 100 mg/g CWM for DPU_3) (Table 5). The main non-cellulosic neutral sugars in the AIS were xylose, arabinose and galactose whereas the minor ones were glucose, mannose, rhamnose and fucose (< 10 mg/g CWM) in accordance with previous studies (Cherif et al., 2021; Mrabet et al., 2012).

Even if DPU did not influence significantly the cell wall composition, dates from DPU_3 were the richest samples on AIS contents and lignin was their main component. These results might explain their highest firmness value (36 N before treatment) since lignin provides rigidity and structural support to cell wall polysaccharides (Kärkönen & Koutaniemi, 2010; Vance, Kirk, & Sherwood, 1980). According to Shafiei, Karimi, and Taherzadeh (2010), lignin and galacturonic acid can be the key compounds in determining the quality of dates. High lignin and low pectin contents could indicate a low quality of date palm sorted for a use in food industrial processing (Mrabet et al., 2015). Neutral sugar patterns in our study are comparable to those reported by Mrabet et al. (2015).

No significant difference was observed in AIS contents after hydration treatment, meaning that this treatment had no effect on cell wall yields whatever the DPU. However, significant differences existed between the three DPUs, only in cell wall yield and rhamnose content, where DPU_3 still with the highest AIS contents after treatment in accordance with the highest firmness value (31N) of these dates.

Moreover, after hydration treatment no significant change was observed on cell wall composition in the different DPUs. In the contrary, Mrabet et al. (2015) show significant increase in lignin and cellulose with a decrease of galacturonic acid after hydration treatments. This might be due to the hydration methods used in their experiment which leads to pectin degradation and an apparent increase in lignin and cellulose. The interaction between hydration treatment and DPU had a significant effect only on rhamnose content which was essentially due to the high effect of DPU factor.

Fruit softening is related to changes of the cell wall components (Awad et al., 2011; Brummell, 2006), and specifically to enzyme activities (Awad et al., 2011; Hasegawa & Smolensky, 1971). In our case, hydration treatment induced firmness modification without significant change neither on cell wall content nor on their composition. These phenomena could be due to the treatment temperature which is responsible for slowing pectin methyl esterase and polygalacturonase activities.

According to our results, date palm fruit cell walls appeared to be stable after treatment.

3.3.3. Polyphenols

Four major polyphenol groups were identified in 'Deglet Nour' dates including flavan-3-ols, flavonols, flavones and hydroxycinnamic acids (Table 6). Dates are rich in polyphenols independently of their type (Al-Farsi et al., 2007; Awad et al., 2011; Besbes et al., 2009; Cherif et al., 2021; Hammouda, Chérif, Trabelsi-Ayadi, Baron, & Guyot, 2013; Mansouri, Embarek, Kokkalou, & Kefalas, 2005) with content higher than 12 mg/g in the fresh edible part (flesh and peel) of the fruit. Date palm fruits are richer in polyphenols than other fruits like nectarine flesh, i.e., 0.14 to 1.02 mg/g FW (fresh weight), peach flesh, i.e., 0.21 to 0.61 mg/g FW (Gil, Tomás-Barberán, Hess-Pierce, & Kader, 2002), and dessert apple flesh, i.e., from 0.6 to 1.6 mg/g FW (Le Bourvellec et al., 2011).

Total polyphenol contents quantified as the sum of the individual compounds ranged from 12.5 (DPU2) to 15.9 mg/g FW (DPU3, Table 6).

Table 5
AIS yields (mg/g fresh weight), neutral sugars, galacturonic acids and lignin content (mg/g AIS) of 'Deglet Nour' date fruits before and after hydration treatment (HT) for the three Date Processing Units (DPU). Statistical results (Pooled SD, two-way ANOVAs) and interaction effects between variables.

	Yields	Rha	Fuc	Ara	Xyl	Man	Gal	NC Glc	C Glc	MeOH	AUA	DM (%)	Lignin
						Before treatment							
DPU 1	107.2	5	3	25	79	13	23	8	97	15	134	60%	159
DPU 2	99.1	4	3	26	96	9	19	7	94	14	140	58%	148
DPU 3	121.4	5	3	24	85	8	21	7	100	16	123	82%	173
						After treatment							
DPU 1	104.4	6	4	25	79	9	22	9	104	15	112	78%	133
DPU 2	93.1	4	3	25	76	9	22	7	99	14	120	68%	161
DPU 3	126.5	4	3	25	85	9	21	9	86	15	102	89%	153
Pooled SD	**3.0**	**0.2**	**0.6**	**0.7**	**5.1**	**1.8**	**1.2**	**0.9**	**3.9**	**0.6**	**19.2**	**0.1**	**8.3**
						Statistics: F-value and significance							
DPU	43	16	0.4	2	1	1	2	1	2	3	0.4	2	2
	***	**	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
HT	0.3	0.001	0.2	0.001	3	1	1	2	0.1	0.3	2	1	3
	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
DPU*HT	2	6	1	1	2.4	1	1	1	4	0.6	0.0	0.1	3
	ns	*	ns	ns	ns	ns	ns	ns	*	ns	ns	ns	ns

Rha: rhamnose, Fuc: fucose, Ara: arabinose, Xyl: xylose, Man: mannose, Gal: galactose, NC Glc: Non-Cellulosic glucose determined without cellulose hydrolysis, C Glc: Cellulosic glucose, AUA: anhydrous uronic acids, MeOH: methanol, DM: degree of methylation, Pooled SD: pooled standard deviation, F-value: Fisher's value, *Significant at $p \leq 0.05$ ** Significant at ≤0.01, Significant at *** $p \leq 0.001$.

Table 6
Total polyphenols, procyanidins (mg/g of FW and their characterization) and minor phenolic compounds (μg/g of FW) of 'Deglet Nour' date fruits before and after hydration treatment (HT) for the three Date Processing Units (DPU). Statistical results (Pooled SD, two-way ANOVAs) and interaction effects between variables.

	Procyanidins					Hydroxycinnamic acids					Flavonols			Flavones		Total PP
	PCA	DP	CAT %	EC %	EC_{ext} %	CSH1	CSH2	CSA4	CSA5	CSpH	QR	IhR	IhH	ChRh	ChhS	
Before treatment																
DPU 1	12.8	31.6	0.7	2.5	96.8	12	14	63	96	25	8	7	3	14	2	13.0
DPU 2	12.3	36.1	0.6	2.2	97.2	12	14	58	94	23	8	9	2	20	3	12.5
DPU 3	15.6	32.8	0.6	2.5	97.0	14	15	76	122	28	9	10	3	17	3	15.9
After treatment																
DPU 1	14.4	36.2	0.5	2.2	97.2	14	15	61	107	25	9	10	2	18	3	14.7
DPU 2	13.1	38.8	0.5	2.1	97.4	15	17	79	130	34	11	11	2	22	3	13.4
DPU 3	16.6	34.7	0.5	2.4	97.1	12	13	62	108	25	8	9	2	14	3	16.8
Pooled SD	0.62	1.42	0.04	0.09	0.12	1.28	1.28	5.37	9.41	2.38	0.89	0.80	0.36	1.83	0.25	0.62
Statistics: F-value and significance																
DPU	16	4	3	5	4	0.1	1	1	1	1	0.4	2	3	5	0.1	16
	**	*	ns	*	ns	ns	ns	ns	ns	ns	ns	ns	ns	*	ns	*
HT	5	7	12	4	7	3	0.3	0.1	2	2	2	4	0.3	0.5	2	5
	*	*	*	ns	*	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	*
DPU*HT	0.3	0.5	1	1	1	2	2	5	4	5	2	2	2	2	2	0.2
	ns	ns	ns	ns	ns	ns	ns	*	ns	*	ns	ns	ns	ns	ns	ns

PCA: procyanidins, DP: average degree of polymerization of procyanidins, %CAT:percentage of (+)-catechin as terminal unit, % EC: percentage of (−)-epicatechin as terminal unit, %ECext: percentage of (−)-epicatechin as extension unit, CSH1: Caffeoylshikimic hexoside_1, CSH2: Caffeoylshikimic hexoside_2, CSA4: 4-caffeoylshikimic acid, CSA5: 5-caffeoylshikimic acid, CSpH: caffeoylsinapoyl hexoside, QR: Quercetin-3-rutinoside, IhR: Isorhamnetin-3- rutinoside, IhH: Isorhamnetin-3-hexoside, ChRh: Chrysoeriol rhamnosyl hexoside, ChhS: Chrysoeriol hexoside sulfate, Total PP: total polyphenols, DPU: Date Processing Unit, HT: Hydration Treatment. Pooled SD: pooled standard deviation, F-value: Fisher's value, *Significant at $p \leq 0.05$ ** Significant at ≤ 0.01, Significant at *** p.

These values are much higher than those reported in the majority of studies (Al-Farsi & Lee, 2008; Ben-Amor et al., 2016; Mansouri et al., 2005;) as the total phenolic content in dates is usually estimated using the colorimetric Folin-Ciocalteu method and varies greatly according to the phenolic standards and to the cultivar used. Moreover, in our study thioacidolysis was directly applied to fruit powders without prior solvent extraction followed by HPLC-DAD analysis of the reaction medium, which enabled the determination of total polyphenol concentration including both extractable and nonextractable procyanidins which are not quantified when a colorimetric assay is performed on a methanol extract. Using phloroglucinolysis prior to HPLC-DAD analysis, Hammouda et al. (2013) also show that total concentration of polyphenols in 'Deglet Nour' date palm fruit accounts for an average of 14 mg/g FW.

Among the four major groups, procyanidins were the predominant class accounting for 98% of total polyphenols and the other polyphenol classes (i.e., hydroxycinnamic acids, flavonols and flavones) were present in very low amount (Table 6). (−)-Epicatechin was always the predominant procyanidin constitutive unit, representing between 97% and 98% of total constitutive units in 'Deglet Nour' fruit whereas (+)-catechin was only present as terminal unit and accounted from 0.4% to 0.8% of the total constitutive units. The average degree of polymerization (DPn) of procyanidins ranged between 31.6 and 36.1 with no significant difference between DPUs. This DPn is linked to astringency perception (Lea & Arnold, 1978), however date palm fruits at *Tamar* stage are not perceived as astringent (Myhara, Al-Alawi, Karkalas, & Taylor, 2000) even if their DPn is higher than 30. This phenomena could be linked to interactions occurring between procyanidins and cell wall polysaccharides after cellular rupture during mastication (Renard, Baron, Guyot, & Drilleau, 2001), inhibiting their physicochemical association to salivary proteins responsible to the astringency sensation.

Five compounds were identified as hydroxycinnamic acids which was the second polyphenol group accounted from 0.9 to 2.5% of total polyphenols in 'Deglet Nour' date fruits. Hammouda et al. (2013) quantified hydroxycinnamic acids as 0.7% of total polyphenols in 'Deglet Nour' and 'Ftimi' cultivars. The major component of this class was 5-caffeolshikimic acid followed by 4-caffeolshikimic acid as previously reported in 'Deglet Nour' date palm (Hammouda et al., 2013). The other hydroxycinnamic acid compounds were present in lower amount.

In 'Deglet Nour' date palm, flavonols were mainly quercetin and isorhamnetin glycosides (quercetin 3′-methylether) and flavones were mainly chrysoeriol (luteolin 3′-methylether) glycosides in accordance with Mansouri et al. (2005) and Hammouda et al. (2013) studies. Flavonols accounted from 0.23 to 0.30% of total polyphenols in 'Deglet Nour' date palm fruits. Flavones only accounted from 0.03 to 0.04% of total polyphenols in 'Deglet Nour' date palm fruits. Hammouda et al. (2013) quantified flavonols as 0.6% of total polyphenols in 'Deglet Nour' and 'Ftimi' cultivars.

Before treatment, significant differences between DPU were observed only for procyanidin contents where fruits of DPU3 presented the highest contents. This difference could be explained by sample heterogeneity due to agricultural practices depending on farm and ripening stage. Contrary to procyanidins, no significant differences were observed for hydroxycinnamic acids, flavonols and flavones between the three DPUs, probably in relation with their low contents inducing some difficulties to evaluate their variability between DPU versus their variability between triplicates.

The average of total polyphenol contents of the three DPU increased significantly after hydration treatment, which is mainly due to the increase of procyanidin contents. Hydration treatment may promote fruit softening increasing the extraction efficiency and leading to the release of polyphenols from their intracellular compartments making them more available for quantification (Wen, Prasad, Yang, & Ismail, 2010). DPn was slightly affected by DPU and heat treatment. In general, DPn increased after heat treatment independently of DPU which could be due to the matrix degradation leading to a better extractability of procyanidins of higher DPn knowing for their capacity to interact with cell wall polysaccharides (Le Bourvellec & Renard, 2012; Renard et al., 2001) or to the degradation of low molecular weight procyanidins. These results are in accordance with orther authors (Mrabet et al., 2015). In contrary to our results, Ben-Amor et al. (2016) reported a higher significant loss of total phenol content after hot water treatment at 60 °C for 3 min. This difference could be due to the origin and the physiological state of date palm fruits.

Neither treatment nor DPU affected minor class phenolic compounds. This could be explained by their lower contents and their higher variability between samples making difficult to observed some significant variations.

No polyphenol losses were detected leading to the conclusion that softening hard-type dates with hydration treatment did not alter their nutritional quality, which is a good advantage promoting date palm

marketability.

4. Conclusion

Characterizing hard-type 'Deglet Nour' dates from three different date palm processing units, before and after hydration treatment, using both, mid infrared spectroscopy as a non-targeted method, and the characterization of appearance (colour, texture) and organoleptic and nutritional compositions allowed to obtain a good overview of the date palm fruit qualities depending on location and fruit treatment. Dates from the different DPUs showed significant variability before and after treatment. These differences are an important factor to take in consideration during sampling and especially on sorting step in the date palm industry supply chain, since it could be determinant on date palm quality and on the best choice of the optimum treatment.

After hydration treatment, date palm fruits became, as expected, softer. However, this treatment was not very suitable for the very hard textured dates (DPU_3) since it decreased their commercial value, as they are designated for direct human consumption. Otherwise, this date palm type could be used for intermediate food products (Martín-Sánchez et al., 2014) in agri-food industries as an economical source of bioactive compounds that would compensate their economic value loss. On the other side, sucrose was the major components discriminating samples from DPU_1 regarding to treatment which were in accordance with MIRS date palm spectra. Thus, infrared spectroscopy being a good evaluative method for date palm quality after treatment, we suggest that it is adopted by Tunisian DPUs as a non-destructive and predictive technique.

Finally, 'Deglet Nour' date palm fruits showed a good nutritional stability during treatment. No changes were detected on cell wall yields and compositions, despite the decrease in firmness, and no loss was observed on the main polyphenols, i.e., procyanidins. The current hydration treatment used in Tunisian date palm processing industries, in the same conditions, seems to be a good solution to enhance the fruits marketability by reaching an appreciated texture while preserving their initial nutritional quality. However, further work will be required to optimize hydration conditions and methods, especially for the very hard-type dates, to evaluate fruit safety and microbiological stability, especially water activity which can support the growth of bacteria, yeast and mold, even if it has been shown that using steam for hydration improves resistance to microbial pathogens (Kader, 2003) lowering the water content (aw = 0.60), as well as their storage after processing and eventually to investigate consumers' sensory acceptance before and after treatment.

CRediT authorship contribution statement

Sarra Cherif: Writing – original draft, Investigation, Formal analysis. **Alexandre Leca:** Writing – review & editing, Supervision, Conceptualization. **Sylvie Bureau:** Writing – review & editing, Supervision, Conceptualization. **Carine Le Bourvellec:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. **Jameleddine Ben Abda:** Writing – review & editing, Supervision, Conceptualization, Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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