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# Effect of storage conditions on 'Deglet Nour' date palm fruit organoleptic and nutritional quality

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## Abstract

The aim of this study was to identify optimal storage conditions able to preserving date palm quality and minimising their loss in the supply chain. Hence, the effect of storage at -18, 0, 2 and 4 °C for 3, 6 and 9 months during two harvest seasons (2017 and 2018) on sugars, organic acids, polyphenols and cell wall yields and composition of 'Deglet Nour' Tunisian dates, were studied. Mid Infrared Spectroscopy (MIR) as a non-targeted method allowed to highlight a year effect on chemical composition and to discriminate samples stored at 4 and 2 °C regarding to major components (moisture, sugars, organic acids..). Cell wall yields were stable during the time. However, galactose from pectin side chains decreased with time, causing an increase of lignin, cellulosic glucose, fucose and rhamnose. Procyanidins, accounting for 98% of total polyphenols, were not affected by storage. Regarding quality parameters stability, stored fruits at -18 °C could be the solution for a long term storage but due to its high energetic costs, 2 °C must be the optimal temperature with a lower time.

**Keywords:** *Phoenix dactylifera* L.; cold storage; cell wall structure; polyphenol

## 1. Introduction

The date palm tree (*Phoenix dactylifera* L.) is cultivated as a food and cover approximately 3% of cultivated areas in the world (Dowson, 1982). Native to the Middle East region, date palm tree is grown extensively in arid and semiarid regions of the world (Ahmed, Al-Gharibi, Daar, & Kabir, 1995). Date (*Phoenix dactylifera* L.) is a fruit of high economic and nutritional relevance and date palm constitutes the basis of economy for the people living in Tunisian Sahara.

Moreover, in human nutrition, date palm fruits are considered as an important part of the Mediterranean diet regardless to their high nutritional value (protein, dietary fibers,

40 sugars, organic acids, antioxidants, vitamins, fatty acids, and minerals) ( Awad et al., 2011 ;
41 Al-Farsi et al., 2005; Elleuch et al., 2008). Date palm fruits are also used in traditional
42 medicine, and studied for their role against hypertension, cancer, infections, heart diseases,
43 etc. (Vayalil, 2012).

44 In Tunisia 'Deglet Nour' date palm is the most produced cultivar, it is also the most
45 appreciated cultivar both locally and internationally. Its production is increasing and reaches
46 241 321 tons from a total date palm production of 305 251 tons in 2018 harvest season
47 (GIFuits, 2018). Moreover, 'Deglet Nour' date palm represent 16% of total agriculture
48 product exportations. However, date palm production is accompanied by a loss in the supply
49 chain due to reducing fruit quality that is a fundamental aspect for the consumer.

50 Storage at low temperature is an efficient approach to maintain quality and increase
51 postharvest life by reducing fruit metabolic activity (Siddiq & Greiby, 2013). Optimal
52 storage conditions for dates at *Tamr stage* are 0 °C for 6 to 12 months, depending on cultivar:
53 semi-soft dates, like 'Deglet Nour' and 'Halawy', have longer storage-life than soft dates, like
54 'Medjhool' and 'Barhee'. For extended storage, the use of temperatures below the highest
55 freezing temperature of -15.7 °C is recommended (Kader & Hussein 2009; Jemni et al., 2019;
56 Ismail et al., 2008). Dates fruits with 20% or lower moisture can be kept at -18 °C for more
57 than one year, at 0 °C for one year, at 4 °C for 8 months, or at 20 °C for one month, relative
58 humidity should be kept at 65-75% for all cases (Kader & Hussein 2009). However, in
59 Tunisia, the storage process is not well mastered and date palm fruits are stored arbitrary
60 between 2 and 5 °C. During storage, the ripening-related loss of firmness or softening is due
61 to the cell wall degradation resulting in a lower quality. Fruit softening is a result of changes
62 of the cell wall components, its involve hydrolysis of neutral sugars from pectin side chains,
63 and depolymerisation, which are associated with cell wall degrading enzymes activities, such
64 as polygalacturonase (PG), pectin methylesterase (PME), cellulase, β-galactosidase (β-GAL),

65 and α-arabinofuranosidase (α-ARF) (Deng et al., 2005; Wei et al., 2010; Wang et al., 2018;
66 Gwanpua et al., 2016; Chen et al., 2017a; Chen et al., 2017b; Awad et al., 2011; Murayama et
67 al., 2002; Hasegawa & Smolensky, 1971). In date palm fruits, the most commonly reported
68 modifications indicate a loss of galactose and uronic acid (Gribaa et al., 2013; Awad et al.,
69 2011). Nutritional compositions of fruits are also modified during storage, and depend on
70 storage conditions such as time and temperature. Total polyphenol amounts in 'Deglet Nour'
71 date palm decrease slightly with storage time (Jemni et al., (2019) but increase with freezing
72 temperatures (A. A. Allaith, Ahmed, & Jafer, 2012; Biglari et al., 2009; Hazbavi,
73 Khoshtaghaza, Mostaan, & Banakar, 2015), it is probably due to a better extractability of
74 phenolic compounds. Since quality parameters are affected by storage, it is very important to
75 understand the effect of such storage conditions on the different characteristics and on
76 consumers' acceptability of the date palm fruit. Many studies are dedicated to the effect of
77 storage on different fruit and vegetable attributes (Ismail et al., 2008; Harker et al., 2003).
78 Unfortunately, few studies are focused on date palm quality parameters. The ideal
79 storage temperature and time for fresh date palm fruit consumption should be evaluated and
80 identified in order to avoid qualitative and quantitative losses. Therefore, the aim of this work
81 was to assess the effect of cold storage conditions, temperature and time on date palm fruit
82 'Deglet Nour' organoleptic and nutritional composition in order to define the optimum
83 storage conditions.

84 ## 2. Material and methods

85 ### 2.1 Chemical

86 Polyphenol standards ((+)-catechin, (-)-epicatechin, 4-cafeoylshikimic acid, 5-
87 cafeoylshikimic acid, rutine, isorhamnetin and chrysoeriol) were purchased from
88 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 ($NH_4OH$) (33%), Sodium borohydride ($NaBH_4$) and acetic acid were from Merck Chimie SAS, an affliate of Merck KGaA, Darmstadt, Germany

**2.2 Plant material**

'Deglet Nour' date (*Phoenix dactylifera* L.) were hand harvested and collected from Kebeli oasis in the South of Tunisia (33° 42' 7" North and 8° 58' 25" East) at the end of October during two harvest seasons (2017 and 2018). Fruits were collected at the fully maturity stage, i.e. *Tamr* stage, which corresponds to the last physiological stage as described by Hussain et El-Zeid, (1975) and Al-Shahib et Marshall (2003) when date palm fruit colour darken with soft and semi-soft texture. Date palm spikelets (about 25 kg) were transported in plastic boxes at ambient temperature to postharvest laboratory in the Higher Agronomic Institute of Chott Mariem, Tunisia. Date palm fruits were manually detached from the spikelets and sorted to discard infested, immature and damaged fruits in the order to have a homogenous and uniform sample. Date palm fruits were stored in small PET containers (190x 115x 58 mm) at -18 °C, 0 °C, 2 °C and 4 °C during 3, 6 and 9 months.

Thirty date palm fruits were considered for each biological replicate and for each condition (temperature/time pair), leading to 4 temperatures x 3 times x 3 replicates, i.e. 36 samples for each year (2017 and 2018).

**2.3 Sample preparations**

112 After each storage time, date palm fruits were pitted, cut into small pieces, dropped in
113 liquid nitrogen and stored at -20 °C until delivery to INRAE PACA, (Avignon, France).
114 Samples were then ground in liquid nitrogen using an IKA®A11 basic analytical mill (Ika
115 Labortechnik, Staufen, Germany) in order to obtain a fine homogeneous powder. Fresh
116 powders using for the determination of soluble sugars and organic acids were conserved at -
117 80 °C until analysis whereas samples used for polyphenols, cell wall isolation as Alcohol
118 Insoluble Solids (AIS) and Mid Infrared Spectroscopy determination (MIR) were freeze-dried
119 and finally stored at -20 °C until analysis.

120 **2.4 Mid Infrared Spectroscopy**

121 Mid Infrared Spectra were acquired at room temperature using ATR Tensor 27 FT-IR
122 spectrometer (Brucker Optics, Wissembourg, France) equipped with a single-reflectance
123 horizontal diamond crystal (Golden Gate, Bruker Optics). All date palm fruit samples were
124 analyzed using the dried powder of whole fruit to compare their spectral quality according to
125 the storage conditions. Freeze-dried powder was placed on the ATR (Attenuated Total
126 Reflection) crystal and was pressed with a system press tip flap. The spectra were acquired
127 from 4000 to 600 $cm^{-1}$ and corrected against the background spectrum of air. Each spectrum
128 was obtained by taking the average of 16 scans. Nine spectra were acquired on different
129 aliquots for each sample to evaluate its heterogeneity. The crystal was cleaned between
130 measurements with deionized water and well dried. Instrument control and spectra collection
131 were performed using OPUS software (version 4.0, Bruker, France) supplied by the
132 equipment manufacturer. The absorption ranged between 2400 and 2200 $cm^{-1}$, due to carbon
133 dioxide, was discarded prior to the calculation. Spectral pre-processing and multivariate data
134 analysis were performed with Matlab 7.5 (Mathworks Inc. Natick, MA) software using the
135 SAISIR package (Bertrand & Cordella, 2008). Principal Component Analysis (PCA) was

136 applied in order to get an overview of the sample discrimination characterized by their
137 infrared spectral data according to storage conditions and was performed using Matlab 7.5.

138 **2.5 Cell walls or Alcohol Insoluble Solids (A.I.S) preparation**

139 Alcohol Insoluble Solids (AIS) were prepared according to previous papers with some
140 modifications (Renard, Voragen, Thibault, & Pilnik, 1990; Renard 2005). Approximately 2 g
141 of freeze-dried date palm powder were dropped in 15 mL of 96% boiling ethanol and let for
142 30 min. Suspension was then transferred to a 50 mL empty Sep-pack column (Interchim,
143 Montlucon, France) equipped with a sinter of porosity 20 µm. The suspension was washed
144 with ethanol 70% at room temperature until the filtrate was sugar-free as shown by the
145 negative reaction of the phenol sulphuric test (Dubois et al., 1956). Sample was then dried by
146 solvent exchange with acetone: water (v/v 60:40, three times), acetone: water (v/v 80:20, two
147 times) and then with acetone 100% until discolouration of the supernatant. The residue was
148 then dried at 37 °C during 48 h and weighted. AIS yields were expressed in mg/g of Fresh
149 Weight (FW).

150 **2.6 Analysis methods**

151 **Sugars and organic acids**

152 Sugars (glucose, fructose and sucrose) and organic acids (malic acid and citric acid)
153 were quantified using colorimetric-enzymatic methods (Boehringer Mannheim Co.,
154 Mannhein, Germany) and expressed in mg/g FW. Absorbance was measured at 340 nm with a
155 SAFAS flx-Xenius XM spectrofluorimeter (SAFAS, Monaco).

156 **Neutral sugar analysis**

157 Neutral sugars from AIS samples were analysed as alditol acetates after acid
158 prehydrolysis and hydrolysis. For the quantification of cellulosic glucose and galacturonic

159 acid, 10 mg of AIS samples were submitted to prehydrolysis by adding 250 µL of 72%
160 sulphuric (1 hour at room temperature) (Saeman, Moore, Mitchell, & Millett, 1954). The
161 solution was then diluted by addition of 1 mL of water and 1 mL of inositol (internal
162 standard). For only neutral sugar quantification no prehydrolysis was carried out, and directly
163 1 mL of 1M sulphuric acid and 1 mL of inositol (internal standard) were added to 10 mg of
164 AIS samples. All Samples were hydrolysed for 3 hours in a heater block at 100 °C. After
165 hydrolysis they were derivatised to volatile alditol acetates (Englyst, Wiggins, & Cummings,
166 1982). Extracts were injected on a GC-FID Clarus 500 (PerkinElmer, Waltham, USA) with a
167 capillary column Optima of 30 m × 0.25 mm, coated with 0.25 µm film thickness
168 (Marcherey-Nagel, Duren,Germany). The conditions were as follows: temperature of
169 injection 250 °C in split mode (1:8 ratio) with injection volume of 1.5 µL, column flow 35
170 cm/s, oven temperature 230 °C, FID detector (250°C, H2 flow 45 mL/min/pressurized air).
171 Results were expressed in mg/g AIS.

172 **Uronic acids assay**

173 After acid prehydrolysis (Seaman procedure), samples were used to measure their
174 uronic acid contents with a spectrophotometric method at 520 nm using a spectrophotometer
175 (V-530 Jasco, Tokyo, Japan), and the m-hydroxydiphenyl (MHDP) assay as described by
176 Blumenkrantz & Asboe-Hansen (1973) with galacturonic acid as external standard, expressed
177 as anhydrouronic acids (AUA). Results were expressed in mg/g AIS.

178 **Methanol assay**

179 Methanol in AIS samples was determined by Headspace-GC-MS (HS-GC–MS) after
180 saponification using $CD_3OH$ as internal standard as described by Renard & Ginies, (2009).
181 The degree of methylation (DM) was calculated as molar ratio of methanol to uronic acid.

182 **Lignin content**

183 Lignin was analyzed in AIS samples as described by Brahem, Renard, Gouble & Le
184 Bourvellec (2017). The amount of lignin was calculated from a linear calibration curve
185 created with commercial alkali lignin.

186 **Polyphenol quantification**

187 Polyphenol were identified by HPLC-ESI-$MS^2$ and their composition was determined
188 by HPLC-DAD with or whitout thioacidolysis as described by Guyot, Marnet, Sanoner &
189 Drilleau (2001). Their identification was performed using an Acquity Ultra performance LC
190 (UPLC) apparatus from Waters (Milford, MA, USA) and their characterization and
191 quantification were performed using an Ultra Fast Liquid Chromatography Prominence
192 system (Shimadzu, Kyoto, Japan) controlled by the LabSolutions software (Version 5.57,
193 Shimadzu, Kyoto, Japan).

194 **Polyphenol identification by HPLC-ESI-$MS^2$**

195 HPLC/ESI-$MS^2$ analysis was performed on an Acquity Ultra performance LC (UPLC)
196 apparatus from Waters (Milford, MA, USA), equipped with a photodiode array detector
197 (detection at 280, 320, 350 and 520 nm) coupled with a Bruker Daltonics (Bremen, Germany)
198 HCT ultra ion trap mass spectrometer with an electrospray ionization source. Separations
199 were achieved using a Kinetex 2.6 µm C18 100A LC column 100x4.6 mm (Phenomenex,
200 Torrence, CA, USA ) protected by a guard column of the same material (C18 100A LC
201 column 100x4.6 mm (Phenomenex, Torrence, CA, USA) operated at 30 °C. The mobile phase
202 consisted of water/formic acid (99:1, mL/mL) (eluent A) and acetonitrile (eluent B). The flow
203 rate was 1 mL/min. The elution program was follows: 3-9% B (0-5 min); 9-16% B (5-15
204 min); 16-50% B (15-45 min); 50-90% B (45-48 min); 90-90% B (48-52 min); 90-3% B (52-
205 55 min); 3-3% B (55–60 min). Samples (crude extracts) were injected at a level of 10 µL. For
206 polyphenol characterization, a capillary voltage of 2 kV was used in the negative ion mode.
207 Nitrogen was used as drying and nebulizing gas with a flow rate of 12 L/min. The desolvation

208 temperature was set at 365 °C and the nebulization pressure at 0.4 MPa. The ion trap was
209 operated in the Ultrascan mode from m/z 100 to 1000.

210 **Polyphenol quantification by HPLC-DAD**

211 Separations were achieved using a Kinetex 2.6 µm C18 100A LC column 100x4.6 mm
212 (Phenomenex, Torrence, CA, USA ) operated at 30 °C. The mobile phase consisted in
213 water:formic acid (99:1, v/v) (eluent A) and acetonitrile (eluent B). The flow rate was 1
214 ml/min. The elution program was as follows: 3-9% B (0-5 min); 9-16% B (5-15 min); 16-50%
215 B (15-45 min); 50-90% B (45-48 min); 90-90% B (48-52 min); 90-3% B (52-55 min); 3-3%
216 B (55–60 min). 20 µl of samples were injected. Quantification was achieved by comparaison
217 with standard solutions of known concentrations at 280 nm for (+)-catechin, (-)-epicatechin
218 and (-)-epicatechin benzyl thioether (quantified as (-)-epicatechin); at 320 nm for
219 cafeoylshikimic hexoside-1, cafeoylshikimic hexoside-2, 4-cafeoylshikimic acid, 5-
220 cafeoylshikimic acid, cafeoylsinapoyl hexoside; at 350 nm for flavonols (quercetin quantified
221 as quercetin-3-rutinoside and isorhamnetin quantified as isorhamnetin rutinoside and
222 isorhamnetin hexoside) and for flavones (quantified as chrysoeriol rhamnosyl hexoside and
223 chrysoeriol hexoside sulfate).The average degree of polymerisation was calculated with the
224 molar ratio of all flavan-3-ol units (thioether adducts plus terminal units) to (-)-epicatechin
225 and (+)-catechin corresponding to terminal units. Results were expressed in mg/g of Fresh
226 Weight and total polyphenol content quantified as the sum of the individual compounds.

227

228 **Statistical analysis**

229 Results are presented as mean values of biological triplicates for each storage
230 temperature and time. Data are reported as pooled standard deviation (Pooled SD). Pooled
231 SDs were calculated for each series of replicates using the sum of individual variances
232 weighted by individual degrees of freedom (Box, Hunter, & Hunter, 1978). Statistical analysis

233 were established using XLSTAT package of Microsoft Excel. Significant differences ($p<0.05$)
234 between means and interactions between variables were evaluated by two-way ANOVA and
235 Tukey's multiple range test.

236 # 3. Results

237 ## 3.1 Mid-infrared spectroscopy

238 A Principal Component Analysis (PCA) was carried out using the spectral data in the
239 range between 1500 and 900 $cm^{-1}$ in order to evaluate the possibility of using these data to
240 discriminate date palm samples according to their storage conditions (Fig. 1). The
241 eigenvectors associated with this PCA were represented in Fig. 2. The first two components
242 (PC1 and PC2) explained more than 85% of the total variance with 66.7 % for the PC1 and
243 18.7% for the PC2 respectively. As regards to the years, the 2017 samples were more
244 gathered than the 2018 ones probably in relation with a highest variability in 2018 than in
245 2017. The storage conditions did not involve change of date palm characteristics in 2017
246 whereas in 2018, samples were separated in two clusters; one of which was overlapped with
247 2017 samples. In 2018, the storage conditions impacted the date palm quality by separating on
248 the left samples stored during 3 and 6 months (T3 and T6) at 2°C and 4°C from the others.
249 This spectral region considered as the fingerprint region (1500 and 900 $cm^{-1}$) corresponds to
250 the absorption of fruit major components, such as sugars, and bands are assigned to C-O, C-C,
251 O-C-H, C-O-H stretching or bending vibrational modes (Talari, Martinez, Movasaghi,
252 Rehman, & Rehman, 2017). This spectral range contains qualitative and quantitative
253 information about sugars, organic acids, cell walls and phenolic compounds, as demonstrated
254 in Bureau et al. (2012) and Canteri et al., (2019). In our work, the main absorptions were
255 observed at 983 $cm^{-1}$ characterizing the samples localized on the left of the map in opposition
256 with a cluster of peaks around 1028 $cm^{-1}$ characterizing the samples on the right (Fig. 3). This

257 area incorporates bands typical of soluble sugars (glucose, fructose, sucrose) such as bands
258 assigned to the C-O and C-OH stretch (1025, 1055 $cm^{-1}$) and of polyphenols (1200 $cm^{-1}$ )
259 which are the abundant chemicals in date palm fruit, bands typical of polysaccharides
260 (cellulose and pectins) assigned to O-C-H stretch (972, 982 $cm^{-1}$).

261 **3.2 Cell wall yields and compositions**

262 The AIS content of 'Deglet Nour' date palm were 104.4 mg/g and 74.3 mg/g Fresh
263 Weight (FW) for 2017 and 2018 respectively (Table 1). They were in the range of those found
264 by Gribba et al., (2013) and Mrabet et al., (2012) but much lower than those reported by
265 Benchabane et al., (2000) which are 346 mg/g and 310.4 mg/g FW of AIS content
266 respectively in 'Deglet Nour' and 'Ghars' cultivars at *Tamr stage*. The difference could be
267 due to factors affecting fruit quality such as year with specific pedoclimatic conditions or
268 agricultural practices or the used of different analytical methods that introduce some
269 variations in the content of extracted components (Myhara et al., 2000; Gribba et al., 2013;
270 Shafiei et al., 2010; Mustafa et al., 1986) Date palm fruits were richer in AIS content than
271 other fruits like apple, i.e. 17 mg/g to 25 mg/g FW (Le Bourvellec et al., 2011), pear, i.e. 28
272 mg/g FW (Le Bourvellec et al., 2013) and apricot, i.e. 30.5 mg/g FW (Femenia et al., 1998a),
273 but contained less than fig flesh cell, i.e. 110 to 160 mg/g FW (Trad et al., 2014).

274 The AIS compositions of whole fruit date palm were characterized by a high amount
275 of lignin (up to 268 mg/g CWM for 2018), cellulosic glucose (up to 137 mg/g CWM for
276 2018) and galacturonic acid (up to 175 mg/g CWM for 2018) (Table 1). Xylose was the main
277 non-cellulosic neutral sugar in the AIS (between 63 and 101 mg/g CWM in 2017 or 2018),
278 followed by arabinose (22-28 mg/g CWM in 2017 and 2018) and galactose (17-22 mg/g
279 CWM for 2017 and 2018). Non-cellulosic glucose, mannose, rhamnose and fucose were only
280 minor components (< 10 mg /g CWM). Mrabet et al., (2012) reported also that in some date

281 palm fruit cultivars such as the 'Deglet Nour' cultivar, lignin was the major component
282 followed by cellulose and uronic acid which is in agreement with our observations. Gribaa et
283 al., (2013) have shown that in date palm cell walls the major non-cellulosic polymers are
284 pectins and not hemicelluloses. Mrabet et al., (2012) found that xylose, arabinose and
285 galactose were the major neutral sugars present in date palm fruit. The composition of date
286 palm cell walls indicated a prevalence of lignin, cellulose, pectins and associated material, the
287 degree of methylation of pectins was >50%, reaching 86% in 2017. Xylose might originates
288 from xylogalacturonans as the other diagnostic sugars for hemicelluloses i.e. non-cellulosic
289 glucose, fucose, and mannose were present in low amounts. These sugar patterns are
290 comparable to those reported by Mrabet et al., (2015) and Elleuch et al., (2008). In our
291 experiment, AIS contents varied with the year and were statistically lower in 2018 than in
292 2017 (Table 1). However, storage time had no effect on AIS yield content meaning that cell
293 wall contents were stable over time whatever the temperature (Table 1), in contrast to other
294 fruits where cell walls contents change considerably during storage (Chen et al., 2015;
295 Femenia, Sánchez, Simal, & Rosselló, 1998b; Murayama, Katsumata, Horiuchi, &
296 Fukushima, 2002; Kim et al., 1999).

297 The year effect was significant for all components, except methanol contents, this
298 could be due to the pedoclimatic conditions. As function of storage time, a significant
299 increase was observed in lignin, cellulosic glucose, fucose, rhamnose, whereas a decrease in
300 galactose content was observed. This tendency may be related to galactose degradation by
301 galactosidase as this enzyme was identified as active enzyme during ripening (Serrano et al.,
302 2001), which resulted to an apparent increase on lignin and other neutral sugars. Gribaa et al.,
303 (2013) also observed a loss of galactose during ripening. In the same way, in our experiments
304 no change was observed for galacturonic acid, xylose and non-cellulosic glucose. A

305 significant slightly increase in mannose and arabinose contents was also observed. This trend
306 in increasing arabinose levels is contrary to other studies showing its decrease (Ahmed &
307 Labavitch, 1980 ; Brahem et al., 2017) or its stability (Gribaa et al., (2013) in different fruit
308 species during ripening. This difference could be due to lower arabinose contents in other date
309 palm fruits (Elleuch et al., 2008), and to specific enzymatic activities of date palm fruit as
310 function of time, like galactosidase which increased with ripening (Serrano et al., 2001) and
311 low degrading arabinofuranosidase and/or arabinanase during the fruit maturation in specific
312 conditions (Gribaa et al., (2013).

313 The storage temperatures also impact the cell wall composition (Table 1) especially pectic
314 polysaccharides. With the temperature increase, galacturonic acid, galactose and arabinose
315 contents decreased whereas an increase in cellulosic glucose content was observed. The other
316 neutral sugar contents were not affected by storage temperatures. Galacturonic acid and
317 neutral sugars changes with increasing temperature could be explained by the pectin
318 depolymerisation and hydrolysis of neutral sugars from pectin side chains (Brummell, 2004;
319 Zhang et al., 2010) due to an increase in both polygalacturonase and β-galactosidase activities
320 during storage (Serrano et al., 2001).

321 **3.3 Sugars and organic acids**

322 Sucrose was the main sugar in 'Deglet Nour' date palm fruit, followed by both,
323 glucose and fructose, almost in the same concentration. Sucrose contents were 268 mg/g in
324 2017 and 353 mg/g FW in 2018) (Table 2) followed by glucose up to 161 mg/g FW in 2017
325 and fructose up to 137 mg/g FW in 2017. Sucrose concentrations were in accordance with
326 other results such as 238 mg/g FW in 'Deglet Nour' date palm (Al-Farsi & Lee, 2008) and
327 239.8 to 350.9 mg/g FW (Ben-Amor et al., 2016). According to Jemni et al., (2019), glucose

328 and fructose concentrations range from 142.8 to 235.8 mg/g FW and from 96.3 to 130.5 mg/g
329 FW respectively, in agreement with the present data.

330 Malic acid was the main organic acid in 'Deglet Nour' date palm fruit, followed by
331 citric acid. Their concentrations were 4.40 mg/g FW in 2017, 2.39 mg/g FW in 2018 for malic
332 acid whereas for citric acid its content did not exceed 1.57 mg/g FW in 2018. This was in
333 agreement with Ghnimi et al., (2018), who found that malic acid is predominant in some
334 Emirati dates ranging from 0.86 to 3.43 mg/g FW, and citric acid ranging from 0.11 to 1 mg/g
335 FW.

336 Characterizing date palm fruit using infrared spectral data showed that the most
337 discriminating region was between 1500 and 900 $cm^{-1}$. This region is well described to
338 contain the bans of absorption of sugars, the main components of date palm fruits (Bureau et
339 al., 2019). The observed data obtained with MIRS (Figure 1) were in accordance with the
340 PCA performed on the sugar and organic acid contents (results not shown), which clearly
341 separated samples according to the year but not to the storage temperature. The sugar and
342 organic acid contents of date palm fruits varied depending on the considered year. Le
343 Bourvellec et al., (2015) also found that year significantly affects primary metabolite contents
344 in tree apple cultivars. This was mainly due to different pedoclimatic conditions as function of
345 year.

346 Sucrose contents were significantly affected by storage time and temperature.
347 Generally, sucrose contents decreased with time for the different temperatures from 268 to
348 134 mg/g FW in 2017 and from 354 to 211 mg/g FW in 2018, except a slight increase or no
349 changes at -18 °C in the two years. These results are in agreement with the study of Jemni et
350 al., (2019) who found the same sucrose decrease in freezing 'Deglet Nour' date palm (0 °C, -
351 40 °C and -80 °C) stored during 10 months. Alhamdan, A. M., & Al-Helal, I. M. (2008)

352 showed also a significant decrease in sucrose content in 'Barhi' freezing date palm for 3, 6
353 and 9 months independently to the storing method. Glucose contents were significantly
354 affected by temperature but not by storage time (Table2). Glucose contents increased
355 generally, with increasing temperature (Table 2). On the contrary, fructose contents were
356 significantly affected by storage time but not by temperature (Table 2). Fructose contents
357 increased with time for the different freezing temperatures and whatever the year. According
358 to Ismail et al., (2008) and Jemni et al., (2019) respiration which could occur during storage,
359 combined with a slowly hydrolysis of sucrose could explain the changes and variation
360 between different sugars (glucose and fructose). An increase of total soluble sugars occurs
361 also in strawberry fruits stored at 6 °C indicating that a new biosynthesis had taken place
362 during storage (Cordenunsi et al., 2005).

363 Citric acid contents presented an opposite trend according to the year. In 2017, citric
364 acid contents increased with storage time whereas in 2018 they decreased. Moreover, this
365 effect was also function of the temperature, especially in 2017. While citric acid contents
366 increased with the storage time at -18 °C, 0 °C and 2 °C, its contents were quite stable at 4 °C.
367 These results are in agreement with those of Jemni et al., (2019) who found that the titratable
368 acidity of 'Deglet Nour' date palm increases after storage at 0 °C from 0.18 to 2.02 g/100 g
369 FW. Malic acid contents were highly affected by storage time. In 2017, they decreased
370 significantly with storage time at the lowest temperatures (-18 °C and 0 °C), and were quite
371 stable at 2 °C and 4 °C. However, in 2018 no change was observed in malic acid contents
372 except a slight decrease at 2 °C and 4 °C after only 6 and 9 months. Other authors also shown
373 opposite trend according to storage as function of fruit botanical origin: Remberg et al.,
374 (2010) found that titratable acidity in 'Summered' apple fruit increases after four months at
375 low temperature (1 °C) while Dziedzic & Blaszczyk, (2019) reported that organic acids in

376 sweet cherry cultivar 'Regina' decrease after a storage at 2°C for two weeks. The results
377 observed could be due to difference in metabolic pathway.

378 So, according to malic and citric acid behaviour, we could estimate that 2 °C and 4 °C
379 were the best temperatures for storing date palm fruits.

380 **3.4 Polyphenols**

381 Four major polyphenol groups were identified in 'Deglet Nour' date palm fruit
382 including flavan-3-ols, flavonols, flavones and hydroxycinnamic acids (Table 3). A total of 11
383 individual compounds were identified and quantified (Table 3). These groups coincide with
384 those found previously in Deglet Nour date palm cultivar (Hammouda, Chérif, Trabelsi-
385 Ayadi, Baron, & Guyot, (2013)). The content of polyphenols ranged between 13.9 (2017) and
386 12.1 (2018) mg/g of FW, in accordance with Hammouda, Chérif, Trabelsi-Ayadi, Baron, &
387 Guyot, (2013). Among the four major groups, procyanidins were the predominant class
388 accounting for 98% of total polyphenols, i.e. 13.5 (2017) and 11.9 (2018) mg/g of FW, close
389 to the 12.44 mg/g FW found by Hammouda, Chérif, Trabelsi-Ayadi, Baron, & Guyot, (2013).
390 (–)-Epicatechin was always the predominant constitutive unit, accounting between 97% and
391 98% of total constitutive units in 'Deglet Nour fruit whereas (+)-catechin was only present as
392 terminal unit and accounted from 0.1% to 0.5% of the total constitutive units. The average
393 degree of polymerization (DPn) of procyanidins ranged between 32 (2017) and 38 (2018).
394 This DPn varies depending on the fruit type, variety, maturation stage and fruit tissue
395 (Hammouda, Chérif, Trabelsi-Ayadi, Baron, & Guyot, 2013) and is highly linked to
396 astringency perception (Lea & Arnold, 1978). However date palm fruit at *Tamr stage* (full
397 ripe) and especially 'Deglet Nour' are not perceived as astringent (Myhara et al., 2000) even
398 if their DPn is high (Haslam and Lilley, 1988).

399 This discrepancy between analytical characterization and perception can be explained
400 by the complexity of the date palm fruit matrix, its high sugar contents, and interactions
401 occurring between procyanidins and cell wall polysaccharides after cellular rupture during
402 mastication (Renard, Baron, Guyot, & Drilleau, 2001) which compete with formation of
403 adducts with proteins and so with sensory perception. Concerning flavan-3-ols, any
404 monomers were detected, specifically in the Deglet Nour fruit.

405 In our study, the DPn of procyanidins were affected by year, storage time and
406 temperature. A significant increase was found after 6 and 9 months of storage at 0, 2 and 4 °C
407 (From 32 in fresh date palm before storage to 45 after 9 months at 0 °C) (Table 3). This could
408 be due to a preferential degradation of low molecular weight procyanidins. Compared to
409 procyanidins, the other polyphenol classes (i.e., hydroxycinnamic acids, flavonols and
410 flavones) were present in very low concentrations (Table 3). Hydroxycinnamic acids
411 accounted for less than 2% of total polyphenols in the fruits. Hammouda, Chérif, Trabelsi-
412 Ayadi, Baron, & Guyot, (2013) have shown that hydroxycinnamic acids account for 0.7 % of
413 total polyphenols in date palm fruit ('Deglet Nour' and 'Ftimi' cultivar). The main component
414 of this class was 5-cafeoylshikimic acid followed by 4-cafeoylshikimic acid as previously
415 reported in 'Deglet Nour' date palm (Hammouda, Chérif, Trabelsi-Ayadi, Baron, & Guyot,
416 2013). The other hydroxycinnamic acid compounds, i.e. the two cafeoylshikimic hexoside
417 and cafeoylsinapoyl hexoside were present in lower amount.

418 Flavonols in Deglet Nour date palm fruit were mainly quercetin and isorhamnetin
419 glycosides. Only one quercetin glycoside and two isorhamnetin glycosides were found, i.e.
420 isorhamnetin hexoside which was in higher concentration than isorhamnetin rutinoside.

421 Flavones were mainly chrysoeriol (luteolin 3′-methylether) glycosides. Two
422 chrysoeriol glycosides were found, i.e. chrysoeriol rhamnosyl hexoside and chrysoeriol
423 hexoside sulfate present in the same contents.

424 All these concentrations and relative composition of each class are consistent with
425 previous works (Hammouda, Chérif, Trabelsi-Ayadi, Baron, & Guyot, 2013; Mansouri et al.
426 2005 and Hong et al., 2006).

427 Polyphenol contents were significantly affected by year but not by storage time and
428 temperature. The storage did not provide a significant loss of total polyphenol compounds.
429 This was mainly due to the fact that procyanidins were stable during storage (Table 3).
430 However, the other minor phenolic compounds tended to decrease with storage time and
431 temperature probably because of their susceptibility to browning or due to their low content,
432 their slightest variation may induce an effect. Le Bourvellec et al., (2018) also found that
433 apricot phenolic contents were not affected by storage. Total phenolics and flavonols were
434 also stable at low temperature (6 °C) in strawberry fruits (Cordenunsi et al., 2005).

435 Storing date palm at low temperatures did not affect polyphenol amounts. In contrast
436 to many fruits that tend to lose stability over storage (Kevers et al., 2007), dates are relatively
437 stable. Thus, based on this experiment, date palm fruits could be stored at the highest
438 temperatures (2 or 4°C) in the aim to guarantee the maximal shelf life with minimal costs.

439 **4. Conclusion**

440 The use of a non-destructive and non-targeted method as infrared spectroscopy and
441 specific chemical characterizations such as sugars, organic acids, polyphenols and cell walls
442 allowed to evaluate the behavior of 'Deglet Nour' date palm fruits during storage at different
443 temperatures during two years. The principal results concerned a good stability of the date
444 palm fruits during storage. However, significant differences were highlighted between the

445 two-harvest years for all studied parameters and spectra, which can be attributed to the effect
446 of agronomic and climatic conditions. The main polyphenols, i.e. procyanidins, were stable
447 with time and temperature, some losses were observed only for minor compounds. The
448 changes of cell wall during storage were linked to the depolymerisation of pectins and the loss
449 of their side chains, whereas the total content of cell wall was stable.

450 Then, in order to prolong the shelf-life of dates for a long-term period and minimize
451 global costs, 2 °C must be considered as the optimal temperature.

**Table 1. AIS yields (mg/g fresh weight), neutral sugars and lignin content (mg/g AIS) of 'Deglet Nour' date fruit during storage at different temperatures in the two harvest seasons (2017 and 2018).**

433

| | Yields | Rha | Fuc | Ara | Xyl | Man | Gal | NC Glc | C Glc | MeOH | AUA | DM (%) | Lig |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Year: 2017 | | | | | | | | | | | | | |
| **Before storage** | 104.4 | 4 | 3 | 22 | 63 | 9 | 19 | 9 | 115 | 16 | 102 | 86% | 118 |
| **After 3 months** | | | | | | | | | | | | | |
| **-18°C** | 91.2 | 4 | 2 | 23 | 81 | 9 | 17 | 8 | 117 | 15 | 101 | 85% | 126 |
| **0°C** | 90.2 | 4 | 2 | 22 | 63 | 10 | 18 | 7 | 131 | 16 | 116 | 74% | 179 |
| **2°C** | 86.5 | 4 | 2 | 22 | 68 | 9 | 17 | 9 | 111 | 16 | 107 | 83% | 99 |
| **4°C** | 91.2 | 4 | 2 | 22 | 75 | 8 | 16 | 7 | 104 | 17 | 99 | 94% | 134 |
| **After 6 months** | | | | | | | | | | | | | |
| **-18°C** | 89.3 | 5 | 3 | 24 | 74 | 9 | 17 | 11 | 110 | 14 | 92 | 85% | 177 |
| **0°C** | 91.2 | 4 | 3 | 24 | 72 | 9 | 18 | 4 | 112 | 14 | 74 | 103% | 110 |
| **2°C** | 94.9 | 5 | 3 | 23 | 67 | 8 | 18 | 7 | 112 | 14 | 81 | 97% | 113 |
| **4°C** | 91.5 | 4 | 3 | 23 | 67 | 8 | 17 | 8 | 108 | 15 | 87 | 95% | 199 |
| **After 9 months** | | | | | | | | | | | | | |
| **-18°C** | 83.6 | 6 | 3 | 23 | 67 | 9 | 18 | 7 | 118 | 14 | 92 | 83% | 135 |
| **0°C** | 84.8 | 5 | 5 | 25 | 77 | 10 | 19 | 7 | 119 | 15 | 114 | 71% | 131 |
| **2°C** | 91.3 | 6 | 5 | 26 | 68 | 9 | 22 | 6 | 126 | 15 | 124 | 68% | 99 |
| **4°C** | 92.4 | 6 | 5 | 23 | 69 | 9 | 19 | 7 | 114 | 15 | 108 | 78% | 138 |
| Year: 2018 | | | | | | | | | | | | | |
| **Before storage** | 74.3 | 6 | 8 | 25 | 74 | 10 | 21 | 7 | 134 | 17 | 175 | 53% | 170 |
| **After 3 months** | | | | | | | | | | | | | |
| **-18°C** | 72.7 | 6 | 6 | 24 | 74 | 10 | 21 | 6 | 135 | 18 | 160 | 62% | 204 |
| **0°C** | 80.3 | 6 | 5 | 24 | 87 | 9 | 20 | 6 | 125 | 17 | 158 | 60% | 172 |

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | **2°C** | 83.6 | 5 | 5 | 23 | 67 | 9 | 20 | 6 | 127 | 16 | 137 | 64% | 137 |
| | **4°C** | 81.8 | 5 | 6 | 23 | 71 | 9 | 19 | 6 | 124 | 16 | 125 | 88% | 148 |
| **After 6 months** | | | | | | | | | | | | | | |
| | **-18°C** | 78.3 | 5 | 3 | 25 | 101 | 9 | 19 | 5 | 127 | 17 | 171 | 53% | 154 |
| | **0°C** | 83.6 | 5 | 3 | 23 | 76 | 9 | 19 | 6 | 116 | 14 | 153 | 51% | 132 |
| | **2°C** | 79.5 | 6 | 3 | 22 | 84 | 8 | 17 | 5 | 117 | 13 | 147 | 47% | 186 |
| | **4°C** | 83.9 | 5 | 3 | 23 | 91 | 8 | 17 | 7 | 116 | 13 | 162 | 44% | 122 |
| **After 9 months** | | | | | | | | | | | | | | |
| | **-18°C** | 76.7 | 6 | 4 | 28 | 89 | 10 | 22 | 6 | 137 | 14 | 161 | 48% | 262 |
| | **0°C** | 87.2 | 7 | 4 | 24 | 81 | 9 | 19 | 10 | 121 | 13 | 156 | 45% | 175 |
| | **2°C** | 90.8 | 7 | 5 | 25 | 86 | 10 | 20 | 8 | 135 | 12 | 131 | 50% | 151 |
| | **4°C** | 88.8 | 7 | 3 | 23 | 76 | 9 | 18 | 7 | 119 | 13 | 141 | 50% | 257 |
| **SD Pooled** | | 3.4 | 0.4 | 0.4 | 0.9 | 5.3 | 0.5 | 0.6 | 0.9 | 5.4 | 0.6 | 6.6 | 0.1 | 13.4 |
| **Year F-value** | | 64.6** | 33.4** | 97.6** | 4.2* | 24.5** | 5.5* | 21.4** | 6.1* | 24.7** | 0.8 | 381.8** | 199.2** | 52.3** |
| **S.Time F-value** | | 1.0 | 24.5** | 22.5** | 8.6* | 2.5 | 7.5* | 20.8** | 1.5 | 5.9* | 42.1** | 2.6 | 18.2** | 5.5* |
| **S.Temperature *F*-value** | | 4.2* | 1.0 | 0.3 | 3.8* | 2.4 | 2.3 | 5.8* | 0.3 | 3.7* | 2.6 | 3.3* | 2.8 | 13.2** |
| **Year*S.Time *F*-value** | | 3.7* | 1.8 | 42.5** | 2.1 | 4.3* | 0.6 | 12.4** | 5.9* | 0.3 | 10.4* | 20.2** | 18.8** | 21.7** |
| **Year*S.Temperature *F*-value** | | 1.2 | 0.3 | 2.1 | 1.7 | 0.2 | 2.7 | 6.7* | 5.0* | 2,7 | 12.5** | 7,6* | 0,0 | 4.1* |
| **S.Time*S.Temperature *F*-value** | | 0.7 | 0.9 | 1.8 | 0.8 | 1.1 | 0.4 | 2.2 | 3.9* | 1.3 | 0.6 | 3.4* | 3.8* | 6.5** |
| **Year*S.Time*S.Temperature *F*-value** | | 0.9 | 0.5 | 2.0 | 1.7 | 3.2* | 0.9 | 1.7 | 1.9 | 0.6 | 0.5 | 1.6 | 1.9 | 8.1** |

Rha: rhamnose, Fuc: fucose, Ara: arabinose, Xyl: xylose, Man: mannose, Gal: galactose, NC Glc: Non-Cellulosic glucose determinated without Seaman hydrolysis, C Glc: Cellulosic glucose deteriminated with Saeman hydrolysis AUA: anhydrous uronic acids, MeOH: methanol, DM: degree of methylation, Lig: lignin, S.Time: Storage time, S.Temperature: Storage temperature.

Pooled SD: pooled standard deviation, F-value: Fisher's value

* Significant at $p$ <0.0001; ** Significant at $p$ <0.05

460 **Table 2. Sugars and organic acids (mg/g FW) variation of 'Deglet Nour' date fruit during storage at different**
461 **temperatures in the two harvest seasons (2017 and 2018). Statistical results (Pooled SD, two-way ANOVA) and**
462 **interaction effects between variables.**
463

| | | Sugars | | | Organic acids | |
|---|---|---|---|---|---|---|
| | | **Glucose** | **Fructose** | **Sucrose** | **Citric acid** | **Malic acid** |
| Year: 2017 | | | | | | |
| Before storage | | 169 | 137 | 268 | 0.9 | 4.4 |
| After 3 months | | | | | | |
| | -18 °C | 126 | 139 | 311 | 0.8 | 4.4 |
| | 0 °C | 172 | 170 | 208 | 0.3 | 3.5 |
| | 2 °C | 168 | 153 | 207 | 1.0 | 4.6 |
| | 4 °C | 167 | 176 | 231 | 1.3 | 4.1 |
| After 6 months | | | | | | |
| | -18 °C | 137 | 154 | 192 | 1.2 | 3.4 |
| | 0 °C | 194 | 130 | 134 | 0.6 | 3.6 |
| | 2 °C | 142 | 147 | 171 | 1.3 | 4.2 |
| | 4 °C | 147 | 149 | 355 | 1.0 | 4.3 |
| After 9 months | | | | | | |
| | -18 °C | 103 | 195 | 384 | 1.5 | 3.5 |
| | 0 °C | 209 | 150 | 223 | 1.8 | 3.5 |
| | 2 °C | 163 | 137 | 202 | 0.2 | 4.3 |
| | 4 °C | 154 | 141 | 324 | 1.2 | 4.1 |
| Year: 2018 | | | | | | |
| Before storage | | 134 | 87 | 354 | 1.6 | 2.4 |
| After 3 months | | | | | | |
| | -18 °C | 138 | 95 | 333 | 1.7 | 2.5 |
| | 0 °C | 124 | 110 | 350 | 0.9 | 2.3 |
| | 2 °C | 135 | 132 | 326 | 0.3 | 2.4 |
| | 4 °C | 151 | 136 | 253 | 0.1 | 2.0 |
| After 6 months | | | | | | |
| | -18 °C | 100 | 107 | 373 | 0.0 | 2.2 |
| | 0 °C | 148 | 14 | 236 | 0.0 | 2.1 |
| | 2 °C | 135 | 118 | 252 | 0.0 | 1.7 |
| | 4 °C | 156 | 78 | 211 | 0.0 | 1.9 |
| After 9 months | | | | | | |
| | -18 °C | 107 | 115 | 340 | 0.0 | 2.1 |
| | 0 °C | 130 | 128 | 290 | 0.0 | 2.2 |
| | 2 °C | 142 | 178 | 272 | 0.0 | 2.1 |
| | 4 °C | 137 | 166 | 256 | 0.0 | 1.8 |
| SD Pooled | | 7.8 | 7.0 | 12.7 | 0.05 | 0.1 |
| Year F-value | | 59.0** | 114.1** | 98.5** | 436.1** | 1834.4** |
| S.Time F-value | | 0.6 | 21.6** | 29.5** | 56.3** | 45.1** |
| STemperature *F*-value | | 35.7** | 2.1 | 57.4** | 48.7** | 17.0** |
| Year*S.Time *F*-value | | 0.6 | 11.4** | 16.1** | 192.1** | 7.3* |
| Year*S.Temperature *F*-value | | 14.0** | 14.9** | 53.8** | 36.6** | 54.3** |
| S.Time*S.Temperature *F*-value | | 4.8* | 7.6** | 12.6** | 44.0** | 5.6* |
| Year*S.Time*S.Temperature *F*-value | | 3.8* | 14.7** | 17.8** | 115.1** | 5.3* |

464 S.Time: Storage time, S.Temperature: Storage temperature
465 Pooled SD: pooled standard deviation, F-value: Fisher's value
466 * Significant at $p$ <0.0001; ** Significant at $p$ <0.05

**Table 3. Total polyphenols, procyanidins (mg/g of FW) and minor phenolic compounds (µg/g of FW) variation of 'Deglet Nour' date fruit during storage at different temperatures in two harvest seasons (2017 and 2018). Statistical results (Pooled SD, two-way ANOVA) and interaction effects between variables.**

| | Procyanidins | | | | | Hydroxycinnamates | | | | | Flavonols | | | Flavones | | Total PP |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | **PCA** | **DP** | **CAT %** | **EC %** | **$EC_{ext}$ %** | **CSH 1** | **CSH2** | **CSA4** | **CSA5** | **CSpH** | **QR** | **IhR** | **IhH** | **ChRh** | **ChhS** | |
| **Year: 2017** | | | | | | | | | | | | | | | | |
| **Before storage** | 13.5 | 32 | 0.4 | 2.7 | 96.9 | 13 | 18 | 98 | 116 | 30 | 9 | 12 | 24 | 5 | 5 | 13.9 |
| **After 3 months** | | | | | | | | | | | | | | | | |
| **-18 °C** | 12.6 | 34 | 0.4 | 2.6 | 97.0 | 13 | 16 | 94 | 108 | 29 | 9 | 15 | 27 | 6 | 6 | 13.0 |
| **0 °C** | 11.9 | 33 | 0.4 | 2.7 | 96.9 | 12 | 15 | 87 | 108 | 28 | 8 | 12 | 23 | 7 | 5 | 12.2 |
| **2 °C** | 12.4 | 30 | 0.4 | 2.9 | 96.7 | 12 | 14 | 97 | 109 | 27 | 10 | 14 | 26 | 7 | 5 | 12.7 |
| **4 °C** | 13.2 | 29 | 0.4 | 3.1 | 96.5 | 14 | 17 | 110 | 129 | 31 | 10 | 15 | 29 | 6 | 5 | 13.5 |
| **After 6 months** | | | | | | | | | | | | | | | | |
| **-18 °C** | 14.9 | 38 | 0.1 | 2.5 | 97.4 | 12 | 14 | 81 | 104 | 30 | 6 | 9 | 20 | 5 | 4 | 15.1 |
| **0 °C** | 14.7 | 40 | 0.1 | 2.4 | 97.5 | 9 | 9 | 57 | 72 | 17 | 9 | 10 | 21 | 4 | 3 | 15.0 |
| **2 °C** | 14.2 | 38 | 0.2 | 2.5 | 97.4 | 11 | 12 | 73 | 93 | 19 | 11 | 10 | 22 | 5 | 3 | 14.5 |
| **4 °C** | 14.4 | 37 | 0.2 | 2.5 | 97.3 | 12 | 15 | 82 | 103 | 28 | 11 | 10 | 22 | 5 | 3 | 14.7 |
| **After 9 months** | | | | | | | | | | | | | | | | |
| **-18 °C** | 13.0 | 39 | 0.2 | 2.4 | 97.5 | 12 | 14 | 81 | 104 | 30 | 6 | 9 | 20 | 5 | 4 | 13.3 |
| **0 °C** | 13.6 | 45 | 0.1 | 2.1 | 97.8 | 9 | 8 | 43 | 59 | 13 | 6 | 9 | 20 | 4 | 3 | 13.8 |
| **2 °C** | 14.4 | 41 | 0.1 | 2.3 | 97.5 | 11 | 12 | 70 | 87 | 21 | 11 | 10 | 25 | 6 | 4 | 14.6 |
| **4 °C** | 14.6 | 39 | 0.2 | 2.4 | 97.5 | 13 | 15 | 74 | 93 | 28 | 6 | 10 | 20 | 5 | 3 | 14.9 |
| **Year:** 2018 | | | | | | | | | | | | | | | | |
| **Before storage** | 11.9 | 38 | 0.4 | 2.2 | 97.3 | 8 | 9 | 53 | 0.082 | 21 | 5 | 12 | 17 | 2 | 4 | 12.1 |
| **After 3 months** | | | | | | | | | | | | | | | | |
| **-18 °C** | 13.8 | 35 | 0.5 | 2.3 | 97.2 | 8 | 12 | 68 | 0.110 | 26 | 6 | 12 | 21 | 3 | 5 | 14.1 |

| | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **0 °C** | 12.3 | 45 | 0.4 | 1.8 | 97.8 | 7 | 6 | 30 | 57 | 11 | 6 | 12 | 18 | 3 | 4 | 12.4 |
| **2 °C** | 12.4 | 40 | 0.4 | 2.1 | 97.5 | 6 | 6 | 41 | 64 | 14 | 6 | 12 | 20 | 3 | 4 | 12.5 |
| **4 °C** | 13.4 | 40 | 0.4 | 2.1 | 97.5 | 8 | 6 | 31 | 57 | 12 | 6 | 12 | 17 | 2 | 4 | 13.5 |
| **After 6 months** | | | | | | | | | | | | | | | | |
| **-18 °C** | 12.6 | 37 | 0.5 | 2.2 | 97.3 | 7 | 8 | 51 | 95 | 18 | 5 | 12 | 20 | 1 | 4 | 12.8 |
| **0 °C** | 11.7 | 46 | 0.5 | 1.7 | 97.8 | 10 | 7 | 28 | 49 | 14 | 3 | 13 | 15 | 2 | 3 | 11.8 |
| **2 °C** | 11.9 | 47 | 0.5 | 1.6 | 97.9 | 10 | 7 | 24 | 41 | 14 | 4 | 13 | 15 | 3 | 3 | 12.0 |
| **4 °C** | 11.0 | 46 | 0.5 | 1.6 | 97.8 | 10 | 7 | 26 | 46 | 14 | 5 | 12 | 16 | 3 | 3 | 11.1 |
| **After 9 months** | | | | | | | | | | | | | | | | |
| **-18 °C** | 12.7 | 37 | 0.5 | 2.2 | 97.3 | 7 | 9 | 58 | 102 | 19 | 10 | 12 | 20 | 2 | 5 | 12.9 |
| **0 °C** | 11.9 | 47 | 0.5 | 1.6 | 97.9 | 10 | 7 | 26 | 48 | 15 | 6 | 13 | 16 | 3 | 3 | 12.0 |
| **2 °C** | 12.0 | 48 | 0.5 | 1.6 | 97.9 | 10 | 8 | 29 | 46 | 14 | 5 | 13 | 15 | 3 | 3 | 12.1 |
| **4 °C** | 11.6 | 48 | 0.5 | 1.6 | 97.9 | 10 | 7 | 23 | 39 | 14 | 4 | 11 | 14 | 2 | 3 | 11.7 |
| **SD Pooled** | 0.5 | 1.3 | 0.02 | 0.08 | 0.08 | 0.5 | 0.5 | 2.2 | 3.0 | 1.2 | 0.7 | 0.6 | 1.3 | 0.4 | 0.3 | 0.5 |
| **Year *F*-value** | 56.3** | 136.4 | 1125.8* | 391.5** | 153.7** | 296.6** | 798.6** | 2098.3** | 744.5** | 343.2** | 150.2** | 10.7* | 112.6** | 314.6** | 4.6* | 64.7* |
| **S.Time *F*-value** | 1.7 | 70.7* | 86.9** | 67.1** | 91.6** | 0.8 | 19.5** | 176.5** | 109.1** | 15.0** | 5.4* | 29.5** | 23.5** | 17.4** | 54.6** | 1.4 |
| **S.Temperature *F*-value** | 1.7 | 22.9* | 5.6* | 17.5** | 19.1** | 11.4** | 42.4** | 147.2** | 183.2** | 61.9** | 4.3* | 0.5 | 3.9* | 4.3* | 9.3** | 2.1 |
| **Year*S.Time *F*-value** | 25.8** | 7.0* | 259.4** | 3.3* | 19.7** | 34.5** | 26.7** | 48.5** | 12.0** | 11.0* | 18.1** | 37.1** | 2.2 | 9.1* | 2.6 | 25.0* |
| **Year*S.Temperature *F*-value** | 3.2* | 22.8* | 6.2 | 21.8** | 23.8** | 20.1** | 23.2** | 73.3** | 103.5** | 17.7** | 22.9** | 3.4* | 7.3* | 1.7 | 1.6 | 3.6* |
| **S.Time*S.Temperature *F*-valu** | 1.8 | 2.9* | 1.5 | 4.0* | 3.4* | 3.6* | 4.6 | 4.4* | 6.4** | 1.8 | 7.2** | 1.3 | 1.0 | 0.5 | 0.9 | 1.8 |
| **Year*S.Time*S.Temperature** | 0.7 | 1.0 | 1.7 | 0.3 | 0.6 | 3.9* | 8.7** | 8.3** | 6.3** | 12.6** | 4.6* | 0.6 | 2.0 | 2.2 | 1.5 | 0.7 |

PCA: procyanidins, , DP: average degree of polymerization of procyanidins, %CAT:percentage of (+)-catechin as terminal unit, % EC: percentage of (-)-epicatechin as terminal unit, $\%EC_{ext}$: percentage of (-)-epicatechin as extension unit, , CSH1: Cafeoylshikimic hexoside_1, CSH2: Cafeoylshikimic hexoside_2, CSA4: 4-cafeoylshikimic acid, CSA5: 5-cafeoylshikimic acid, CSpH: cafeoylsinapoyl hexoside, QR: Quercetin-3-rutinoside, IhR: isorhamnetin rutinoside, IhH: isorhamnetin hexoside ChRh: chrysoeriol rhamnosyl hexoside, , ChhS: chrysoeriol hexoside sulfate, Total PP: total: total polyphenols, S.Time: Storage time, S.Temperature: Storage temperature, F-value: Fisher's value, * Significant at $p$ <0.0001; ** Significant at $p$ <0.05

**Fig 1. PCA results on mid-infrared spectral data between 1500 and 900 cm$^{-1}$ based on storage conditions of Deglet Nour'. The code corresponds to the year (17 : 2017 ; 18 : 2018), to storage time (T0 : initial time ; T3 : 3 months storage ; T6 : 6 months storage and T9 : 9 months storage) and to the temperature (18 : -18 °C ; 0 : 0 °C ; 2 : 2 °C ; 4 : 4 °C and te : control). A) as function of the year B) as function of the storage time C) as function of the storage temperature**

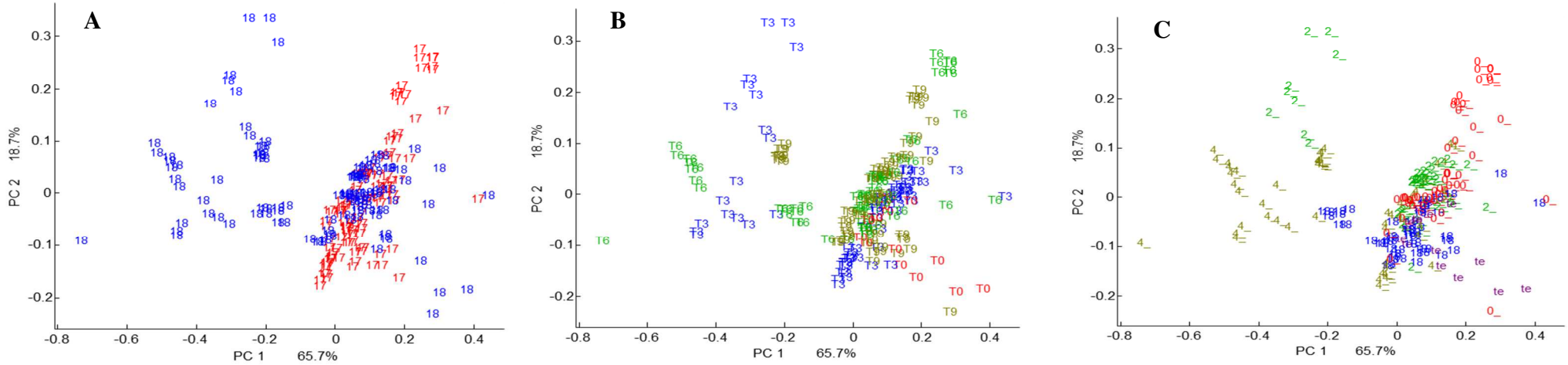
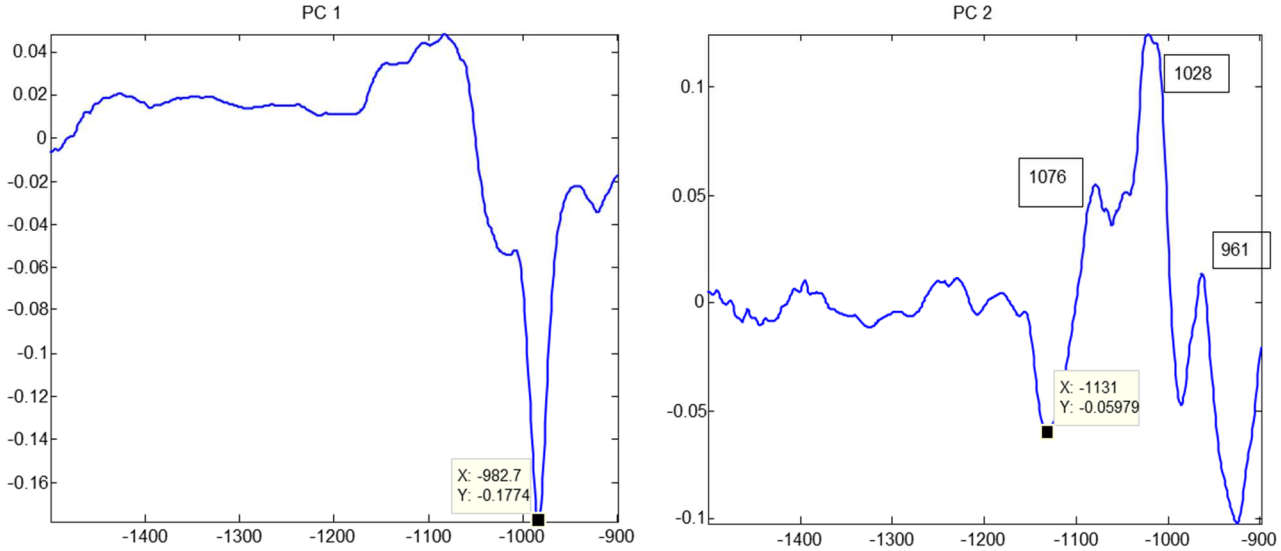



**Fig. 2: Eigenvectors associated to PCA results (A), (B) and (C) on FT-IR spectra (1500-900 cm$^{1}$)**



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