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1 Caprification modifies polyphenols but not cell wall 2 concentrations in ripe figs

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26 **Abstract**

27 Figs, when ripe, move to their final colour and lose radically of their firmness, and
28 caprification (artificial pollination) leads to increased fruit softening during maturity. To
29 identify effects of caprification on cell wall amounts and phenolics composition, cell wall
30 material and polyphenols were investigated in five Tunisian figs. Cell walls were isolated as
31 alcohol insoluble solids (AIS) content. AIS yield was not affected by caprification in all
32 cultivars. Pectin polymers decreased as the fruit fulfil its development after being caprifified.
33 The pectin in figs exhibited high degree of methylation (> 50%) which increased more with
34 caprification. Major neutral sugars components of the AIS were glucose, from cellulose,
35 followed by arabinose. Caprification had no effect on neutral sugars composition of the cell
36 wall. Methanol extraction followed by HPLC-DAD analysis revealed two anthocyanins, three
37 flavonols and one hydroxycinnamic acid. For all varieties the total polyphenols were lower in
38 the flesh (up to 1.27 mg/kg FW) than in the peel (up to 12.64 mg/kg FW). Anthocyanins were
39 the major compounds described in figs representing more than 98% of total polyphenols in
40 both dark and white coloured fruit. Textural components and properties of figs are moderately
41 affected by caprification, while Anthocyanins biosynthesis appears to be stimulated by
42 pollination with higher concentrations of total polyphenols in caprifified fig fruits.

43 **Keywords:** figs *Ficus carica*; pectin; anthocyanins; caprification; textural properties;
44 nutritional composition.

45
46
47
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49
50

51 Abbreviations

52 BHL¹

53 ZD²

54 THG³

55 BD⁴

56 KHD⁵

57 AIS⁶

58 HPLC-DAD⁷

59 MHDP⁸

60 GC-MS⁹

61 GC-FID¹⁰

62 PTFE¹¹

63 SPSS¹²

64 GLM¹³

65 DM¹⁴

66 PG¹⁵

67 AUA¹⁶

68

¹ Bouhouli

² Zidi

³ Thgagli

⁴ Bidhi

⁵ Khedri

⁶ Alcohol insoluble solids

⁷ High performance liquid chromatography - diode array detector

⁸ *m*-hydroxydiphenyl

⁹ Gas chromatography - mass spectrometry

¹⁰ Gas chromatography - flame ionisation detector

¹¹ Polytetrafluoroethylene

¹² Statistical Package for the Social Sciences

¹³ General linear model

¹⁴ Degree of methylation

¹⁵ Degree of methylation

¹⁶ Anhydro-uronic acid

69 1. Introduction

70 *Ficus carica* is one of the oldest known fruit crop and used for fruit production (Beck and
71 Lord, 1988; Kislev et al., 2006). Beside olive tree, fig specie is considered a biomarker of
72 Mediterranean ecosystem. Tunisia, a cradle for common fig tree, exhibits an attractive
73 biodiversity and provides a large array of genotypes (Baraket et al., 2009). Figs have been
74 used for human consumption for centuries and recently their laxative and nutritive values
75 have been investigated (Çaliskan and Polat, 2011; Trad et al., 2013). Fig cultivars are several
76 and well adapted to local agrosystems. Some are of the *Common* type that produces figs
77 without pollination. Many others are of *Smyrna* type that need caprification (Mars et al.,
78 1998). The caprification is quite a common practice in all regions and was cited as an
79 important factor affecting the quality of the fruit (Mars et al., 2009). The general practice of
80 caprification is to distribute the *profichis* (male syconia) at intervals of few days over a period
81 of about three weeks corresponding really to the receptivity of female figs (Rahemi and Jafari,
82 2008). In Tunisia, the general tendency is to connect two to six caprifigs with a wire or a stick
83 passed through their neck and hung onto branches of female tree. It is important for fig
84 growers and consumers to understand the incidence of caprification on quality of figs at
85 maturity. Analysis of fig quality in response to caprification is limited to physical and
86 chemical aspects (Condit, 1947; Gaaliche et al., 2011) or aromatic profile released by the fruit
87 at full ripeness (Trad et al., 2012). Caprifiged figs are usually larger, greener and more prone to
88 splitting, and have a darker interior pulp color (Oukabli et al., 2003; Michailides et al., 2008).
89 Increased risk of splitting, i.e. a textural modification, could be linked to the cell wall
90 compositions and/or of the relative ratios of peel, the resistant tissue, to pulp. Little is known
91 about fig cell walls, though dried fruits are known to be rich in dietary fibres (Marlett and
92 Vollendorf, 1994; Vinson, 1999). Modifications in colour could be related in terms of
93 chemical composition to the polyphenols composition. The main polyphenols of figs are

94 anthocyanins, flavonols and phenolic acids which have been identified and quantified in
95 several varieties of figs with different colour (black, red, yellow and green) (Solomon et al.,
96 2006; Del Caro and Piga, 2008; Veberic et al., 2008). Duenas et al. (2008) identified the main
97 anthocyanin in figs as cyanidin-3,5-diglucoside, cyanidin-3-glucoside, cyanidin-3-rutinoside
98 and pelargonidin-3-rutinoside. Crisosto et al. (2010) studied the influence of fruit variety and
99 harvest season on the phenolic compound content. Some others have been made on the
100 health-promoting potential of figs due to their phenolics they contain (Harnly et al., 2006;
101 Duenas et al., 2008; Veberic et al., 2008).

102 To understand the difference in texture and colour between caprifried and non caprifried fig
103 fruit, we have studied the impact of caprification on morphology, texture and tissular
104 repartition, on cell wall contents and composition, and on phenolics in fig syconia of five
105 Tunisian (dark and light) fruit cultivars.

107 2. Materials and methods

108 2.1. Fruit sampling and preparation

109 Figs from five cultivars (Bouhouli 'BHL' and Zidi 'ZD': dark coloured figs; Thgagli 'THG',
110 Bidhi 'BD' and Khedri 'KHD': white coloured figs) were sampled and prepared for AIS and
111 polyphenols determination. Fruits were harvested from two regions well-known by fig tree
112 growing: 'Thibar' (governorate of 'Béjà') in the North West and 'Bekalta' (governorate of
113 'Monastir') representing the Central East of Tunisia. Samples of thirty homogenous fruits
114 (three replicates of 10 fruits each) were picked for each variety and from caprifried and non-
115 caprifried trees (distant about 300 m from the rest of the orchard). Fruits were selected fully
116 ripe from the main crop at the last of july in 'Bekalta' for 'BD' and 'KHD' cultivars and the
117 last of august in 'Thibar' for 'BHL', 'ZD' and 'KHD' during the two cropping season 2009
118 and 2010. General aspects of the five fig cultivars are given in table 1. Fruit weight, flesh

119 weight, fruit size, skin thickness, firmness and dry matter content were measured to disclose
120 caprification impact on morphological aspects of quality in figs. Firmness was determined
121 using durofel (Duro10, SETOP GIRAUD Technology, Cavaillon, France). Four grams of
122 fresh powder of the fruit were dried (70°C - 96 hours) and removed to be weighed again for
123 dry matter determination.

124 Samples were stored at -20°C then thawed for peel/flesh separation. Cell wall and phenolics'
125 characterization was carried out in the two separate tissues (peel and flesh). Peel samples,
126 meticulously removed using a scalpel, were ground after being soaked in liquid nitrogen.
127 Fresh powder obtained was conserved at -80°C until analysis. Flesh samples were ground in
128 liquid nitrogen using an IKA® A11 basic analytical mill (Ika Labortechnik, Staufen, Germany)
129 and the powder was sieved to remove seeds and other debris mainly composed by the ostiole
130 scales. Samples intended for polyphenols determination were lyophilised and the freeze-dried
131 powder was finally stored at -20°C.

133 2.2. Cell wall isolation and analysis

134 Alcohol insoluble solids (AIS) were prepared according to Renard (2005). Uronic acids were
135 measured spectrophotometrically by the *m*-hydroxydiphenyl (MHDP) assay as described by
136 Blumenkrantz and Asboe-Hansen (1973) with galacturonic acid as external standard and
137 expressed as anhydro-uronic acids (AUA). Sample preparation for this analysis consists in
138 sulphuric acid hydrolysis according to Saeman et al. (1954) as described below. Methanol was
139 determined by Headspace-GC-MS after saponification as described by Renard and Ginies
140 (2009). Neutral sugars were analysed as alditol acetates after acid hydrolysis with 2 options.
141 For cell walls including cellulose analysis samples (c.a. 10 mg of AIS) were submitted to
142 prehydrolysis with 250 µl 72% sulphuric acid for 1 hour at room temperature (Saeman et al.,
143 1954) and then diluted to 1M sulphuric acid by addition of water and internal standard

144 (inositol). For extracted polysaccharides no prehydrolysis was carried out, samples were
145 dissolved in 1M sulfuric acid with internal standard. All samples were placed in oven at
146 100°C for 3 hours for hydrolysis. After hydrolysis they were derivatised to volatile alditol
147 acetates (Englyst et al., 1982). They were injected on a GC-FID HP 5890 Serie II (Agilent,
148 Inc, Palo Alto, USA) with capillary column of 30 m x 0.25 mm i.d. coated with DB225 MS,
149 0.25 µm film thickness (J&W Scientific, Agilent, Inc, Palo Alto, USA). The conditions were:
150 temperature of injection 250°C in split mode (ratio 1:25); hydrogen as carrier gas at 45 cm/s
151 (at 215 °C), column flow was 1.3 ml/min and the oven temperature was isothermal at 215 °C.

153 2.3. Polyphenols in figs

154 Polyphenols were extracted by suspension of the freeze-dried fig powder (circa 200 mg) in
155 1200 µL acidic methanol (1% acetic acid, v/v) and 15 min sonication in a melting ice bath as
156 described by Guyot et al. (2001), followed by filtration (PTFE, 0.45 µm) before HPLC-DAD
157 analysis. Polyphenols were measured by HPLC as described by Guyot et al. (2001). Phenolic
158 compounds were separated in an Agilent 1050 separation system (Agilent Technologies,
159 Santa Clara, CA, USA) including a quaternary pump coupled to a diode array detector and
160 controlled by Chemstation A.10.02 software. Separations were achieved using a (250 × 4mm
161 i.d.) Licrospher PR-18 5 µm column (Merck, Darmstadt, Germany) with a guard column
162 (Licrospher PR-18 5 µm column, Merck, Darmstadt, Germany) operated at 30 °C. The mobile
163 phase consisted of water/formic acid (98:2, v/v) (eluent A) and acetonitrile (eluent B). The
164 flow rate was 1 ml/min. The elution programme was as follows: 3–9% B (0–5 min); 9–16% B
165 (5–20 min); 16–50% B (20–45 min); 50–100% B (45–50 min); 100–100% B (50–52 min);
166 100–3% B (52–55 min). Triplicate samples were injected at a level of 20 µl. The column
167 effluent was monitored at 280, 320, 350 and 520 nm. Quantification was achieved by
168 injection of standard solutions of known concentrations. Standard of 5-cafeoylquinic acid was

169 ordered from Sigma-Aldrich (Deisenhofen, Germany). Quercetin-3-rutinoside, quercetin-3-
170 glucoside, cyanidin-3-glucoside and cyanidin-3-rutinoside were obtained from Extrasynthese
171 (Lyon, France).

172

173 **2.4. Statistics**

174 Data analysis was established using SPSS package software (*version* 13.0; SPSS *Inc.*). The
175 results presented are means (N=3) \pm standard deviations (SD) from samples harvested over
176 two years. The means of cultivars were compared by Duncan's multiple range test ($p < 0.01$)
177 and the General Linear Model (GLM) procedure ($p < 0.01$) was used for multivariate analysis
178 including three fixed factors: cultivar, caprification and compartment of the fruit in order to
179 check out the effect of each factor and their interactions on cell wall and polyphenol
180 concentrations in figs.

181

182 **3. Results and discussion**

183 **3.1. Morphological aspects of figs and caprification effect**

184 The incidence of caprification was clear on fruit size, flesh thickness and fresh weight (p
185 < 0.01) (Table 2). Average fruit weight varied from 48 g ('ZD') to 112 g ('BHL'). Caprifried
186 figs were almost twice the weight of non-caprifried fruits (112 g versus 65 g in 'BHL' and 82
187 g versus 48 g in 'ZD' figs). The fruit skin, though relatively thick compared to e.g. peach or
188 apple, represented in average 14.4% of total fruit weight; for fruits that developed without
189 being caprifried, this proportion was higher in conjunction with flesh mass reduction (Table 2).
190 Caprification increased fig weight with proportionally a greater increase in flesh of the fruit.
191 Flesh thickness increased with caprification to reach 23 mm in 'ZD' fruit versus 15 mm in
192 non caprifried figs and this undoubtedly contributes to improve consistency, taste and flavour
193 of figs when ripe. In contrast, non caprifried figs showed an important internal cavity, a mark

194 of pedicels development collapse and lack of seeds and the fruit was significantly firmer (p
195 <0.05) at harvest time. In addition, the lack of caprification was responsible of dry matter
196 content reduction in the fruit (Table 2).

197 Figs are climacteric fruits that show a drastic loss of firmness when ripe and in general
198 become very soft at harvest. Firmness measured at full ripeness was almost the same in the
199 five cultivars. 'BD' and 'KHD' figs were however firmer than the other fruits (Table 2). Both
200 caprifried and non caprifried figs lose of their firmness, but it is important to mention that
201 caprification enhanced more softening of the fruit when ripening.

202

203 3.2. Changes in cell wall of figs

204 AIS content in figs showed marked differences between peel and flesh. AIS concentrations in
205 the flesh were almost three times the values recorded in the peel (Table 3). AIS represented,
206 average values for the five cultivars, 56 and 131 mg/g FW in peel and flesh respectively.
207 However the presence of seeds must be taken into account when compare the flesh (which
208 includes the seeds and their resistant outer layer, very rich in cell walls) to the peel. In the
209 peel, 'KHD' fruit had the highest concentrations on dietary fibres among the five cultivars (65
210 mg/g FW). In the flesh, AIS yields reached 162 mg/g FW in 'THG' fruit. The lowest values
211 were found in 'BHL' fruit (51 and 110 mg/g FW in peel and flesh respectively) and
212 differences were significant between cultivars (Table 3). Caprification had no effect on AIS
213 content of figs ($p <0.05$) which therefore is not sufficient to explain the textural difference.
214 Mohamed and Mrak (1942) reported that total fibres content were somewhat fewer in
215 caprifried figs compared to non-caprifried fruit. Caprifried fruits, having better pollination,
216 contain less aborted flowers and more seeds: the development of the flesh thus appeared to be
217 proportional to the number of seeds.

218 Pectin content was estimated by the uronic acid concentration as galacturonic acid is the main
219 component of pectins. The peel was a bit richer in uronic acids and thus pectin than the flesh
220 with 296 and 285 mg/g AIS respectively in peel and flesh (Table 3). Higher concentrations of
221 galacturonic acid in the outer part of the fruit have been reported for many other species
222 (Gross and Sams, 1984). As indicated in table 3, pectin in figs was highly methylated (degree
223 of methylation (DM) exceeded 50% in the five cultivars) resulting in high properties for gel
224 conversion. This parameter is important as the fruit is utilized for drying, heating or other
225 industrial purposes. Figs have the property to keep their coherent structure and preserve their
226 initial form and shape after storage or conservation and for this reason the fruit is among
227 '*adequate stored foodstuff*'. Regardless of caprification, figs keep high methylated pectin.
228 Pollination may act as enzymatic activity trigger. A good pollination is responsible of a
229 perfect unfolding of ripening process and enzymes like polygalacturonase (PG) or pectin-
230 methyl esterase efficiently proceed to cell wall split and fruit softening as a result of cleavage
231 of 1,4-galacturonosyl linkages caused by hydrolytic enzyme (PG) (Gross and Sams, 1984).
232 Neutral sugars composition was different between the two compartments of the fig receptacle.
233 Neutral sugars represented a high proportion of the AIS in the peel (Table 3). The main
234 neutral sugar was glucose from cellulose, present in higher concentration in the AIS of peel
235 tissue (222 and 135 mg/g AIS in peel and flesh respectively). The other major neutral sugars
236 of the AIS were arabinose (Ara), followed by galactose (Gal) and xylose (Xyl). Arabinose,
237 Xyl and Gal were identified as major neutral sugars in the AIS of 'Houraishi' figs cultivated
238 in Japan (Owino et al., 2004). In local figs, Ara ranged from 63 to 88 mg/g in the peel and
239 from 34 to 45 mg/g in the flesh. Gal ranged from 33 to 83 mg/g and from 25 to 38 mg/g of
240 AIS in peel and flesh respectively. Disparity between the two separate tissues was very
241 marked regarding concentrations of arabinose (almost twice as much in the peel as in the
242 flesh), less for galactose and xylose. Mannose was a minor AIS component, with twice as

243 high concentrations in the peel as in the flesh. The two yellowish 'BD' and 'KHD' cultivars
244 exhibited the greatest variations between the two compartments of the fruit (Table 3). There
245 were significant differences in neutral sugars composition between the five cultivars ($p < 0.01$).
246 Like AIS yields, neutral sugars compositions were little affected by caprification. This was
247 true for the five cultivars and in both tissues (Table 3). The most important variations were
248 recorded in rhamnose (Rha) concentrations and differences were highly significant between
249 cultivars and among both tissues ($p < 0.01$). Glucose, from cellulose, was unaffected by
250 caprification contrary to cultivar or compartment effects. Arabinose concentrations were
251 slightly higher in AIS of the peel of uncaprified figs. In contrast, caprified fruits had more Ara
252 and Gal in their fleshy receptacle. Cultivars 'KHD' and 'BHL' were the most influenced by
253 the lack of caprification considering the composition in cell wall polysaccharides.

254 Interaction between cultivar and compartment of the fruit was highly significant for cell wall
255 components and methylation degree of the pectin (Table 3). Cell wall composition is
256 extremely variable between cultivars and between both tissues. 'THG' was the richest fruit in
257 AIS (162 mg/g FW in the flesh) and exhibited the highest methylation degree of the pectin
258 (77% in the fleshy receptacle). Arabinose and cellulose, as well as pectin polymers, were
259 more concentrated in 'KHD' fruit.

260 The cultivar/caprification interaction (cv. \times trt.) was significant ($p < 0.01$) for Ara, Glc and
261 uronic acid contents of the AIS. Caprification effect varies from one cultivar to another and
262 variations affect essentially major neutral sugars and uronic acid contents of the cell wall.

263 Interaction between caprification and tissue type of the fruit (trt. \times part) was highly significant
264 for concentrations of Ara, Gal and Glc. The peel was more influenced by the lack of
265 caprification than the flesh considering these three monosaccharides.

266 The overall interaction between the three factors (cv. \times trt. \times part) was significant ($p < 0.01$) for
267 pectin polymers and almost all neutral sugars content. There was no change in AIS yield of

268 figs among the five cultivars and in both tissues whether the tree is caprifigged or not. Neutral
269 sugar concentrations change differently between cultivars and between tissue type of the fruit.

270

271 3.3. Phenolic compounds in figs and caprifigging effect

272 Composition and concentrations of the phenolics found in the peel and in the flesh of the five
273 fig varieties are shown in Table 4-A and 4-B respectively. Three major phenolic groups
274 (phenolic acids, quercetin glycosides and anthocyanins) with a total of six identified
275 individual compounds were quantified. The sum of phenolics determined by HPLC ranged
276 from 0.2 mg/kg (cv. THG) to 12.6 mg/kg FW (cv. BHL) in the peel. In the flesh, the total
277 polyphenols ranged from 0.7 mg/kg (cv. BHL) to 1.3 mg/kg FW (cv. KHD). These are overall
278 low phenolic contents (Harnly et al., 2006). Total polyphenol concentrations were higher in
279 the peel for the dark varieties BHL and ZD, and were lower in the peel than in the flesh for
280 the white varieties THG, BD and KHD. The three phenolic classes described above were
281 reported by Del Caro and Piga (2008) in 'Mattalona' (black) and 'SanPietro' (white) figs.
282 Concentrations of phenolic compounds are commonly higher in the peel as in the flesh, as
283 noted for example for apples by LeBourvellec et al. (2011).

284 Among the three major groups, anthocyanins were the predominant class in both peel (98%)
285 and flesh (99%) (Table 4-A and 4-B). Nevertheless, anthocyanins were absent in the peel of
286 white coloured fruit. Cyanidin-3-glucoside and cyanidin-3-rutinoside were the major
287 constituent of this class, and in all cultivars, cyanidin-3-rutinoside was predominant.
288 Anthocyanins were previously identified in figs grown in Spain (Duenas et al., 2008). Major
289 compounds described were: cyanidin-3,5-diglucoside, cyanidin-3-glucoside, cyanidin-3-
290 rutinoside and pelargonidin-3-rutinoside. In 'Cuello de Dama' figs (dark coloured fruit), the
291 peel was 7 times richer in anthocyanins than the flesh (97 mg/kg in the peel versus 14 mg/kg
292 in the flesh) (Duenas et al., 2008). 'Bursa' figs developed anthocyanins at the following

293 concentrations: 1 and 41 mg/kg FW in flesh and peel respectively (Solomon et al., 2006). In
294 cultivar 'Mission', the anthocyanins were represented for about 75% by cyanidin-3-rutinoside
295 and 11% by cyanidin-3-glucoside (Puech et al., 1975) and this is relatively in accordance with
296 our results. Anthocyanins are much more concentrated in many other fruits like plums,
297 blueberries or black grapes. Anthocyanins concentrations in plums ranged between 20 and
298 250 mg/kg FW (Shahidi and Naczk, 1995).

299 Flavonols were present exclusively in the peel for all five varieties. Three quercetin
300 glycosides were found and quantified in quercetin equivalent: quercetin-3-rutinoside >
301 quercetin-3-O-malonyl glucoside > quercetin-3-glucoside. The quercetin-3-O-malonyl
302 glucoside is described for the first time in fig fruit. Depending on the variety, total flavonols
303 ranged from 0.108 mg/kg (cv. ZD) to 0.277 mg/kg FW (cvs. BHL and BD). Quercetin-3-
304 rutinoside (rutin) was identified as major phenolic compound in Slovenian figs (up to 28.7
305 mg/100g FW) (Veberic et al., 2008). Flavonols are the most ubiquitous flavonoids in foods.
306 They are generally present at low concentrations of ~15 - 30 mg/kg FW. Flavonols
307 accumulate in the outer and aerial tissues because their biosynthesis is stimulated by light
308 (Price et al., 1995). This could explain the absence of such phenolics in the internal pulpy
309 flesh of figs.

310 For all varieties, hydroxycinnamic acids were a minor group which accounted for an average
311 of 0.2% of the total polyphenols as well as in the peel or in the flesh (Table 4-A and 4-B).
312 These levels increase to attain 5% in the peel of white coloured figs. The hydroxycinnamic
313 acid class was represented by 5'-caffeoylquinic acid. For all varieties higher concentrations
314 were found in the peel, up to 0.021 mg/kg FW, than in the flesh (up to 0.004 mg/kg FW).
315 Chlorogenic acid was also identified in figs original from southern Slovenia in low amounts
316 (Veberic et al., 2008). Hydroxycinnamic acids are found in all parts of the fruit in general,
317 although the highest concentrations are seen in the outer part of the receptacle. Concentrations

318 generally decrease during the course of ripening, but total quantities increase as the fruit
319 increases in size (Manach et al., 2004). The concentrations of hydroxycinnamic acid in figs
320 were very low and incomparable to quantities defined in plums, blueberries or apples. Plums
321 are very rich fruit in hydroxycinnamic acids (1150 mg/kg FW) (Shahidi and Naczki, 1995).
322 Blueberries can reach concentrations of 2200 mg/kg FW of chlorogenic acid. In 'Gala' apples,
323 caffeoylquinic acid reached 400 mg/kg DW in peel and 2600 mg/kg DW in flesh (Renard et
324 al., 2007).

325 Caprification had significant influence on polyphenols composition in all cultivars (Table 4).
326 However, the most outstanding effect was noted in dark-coloured fruits. Differences were
327 visible especially for concentrations of cyanidin-3-rutinoside. Non-caprifigged figs, with their
328 small fruit, were however more concentrated anthocyanins and concentrations were higher in
329 the external part of the two cultivars 'BHL' and 'ZD'. Flavonoid concentrations increased
330 twice in non-caprifigged 'ZD' fruit. The effect of caprification was clearer on the peel tissue.
331 Small changes touched internal part of the fruit. Phenolic compound levels were almost
332 similar in white coloured figs and were therefore slightly affected by caprification in the flesh
333 as in the peel. Janick and Moore (1975) reported that the type of caprifig may have a
334 significant effect on the colour of both the fruit skin and its interior edible flesh. Most
335 phenolic compounds, especially anthocyanins, cinnamic acid and flavonoids are concentrated
336 in the peel of figs (Solomon et al., 2006; Del Caro and Piga, 2008). This remains true as the
337 fruit develop without caprification. Skin colour and external visual appearance of Tunisian
338 figs exhibited no change with deficient caprification; however, a good pollination is generally
339 a precursor of pronounced pulp colour.

340 Anthocyanins remain the major contributors to colour of the purple figs. Non caprifigged fruits
341 were smaller but had a pronounced coloured skin which wrapped all surface of the receptacle.
342 Polyphenols concentrations increased noticeably in the outer part related to fresh weight.

343 However, examining the concentrations per fruit between caprified and non caprified
344 syconium we noticed that caprified figs were richer in anthocyanins. In cultivar ‘Bouhouli’,
345 the concentrations per fruit were respectively: 0.193 mg and 0.145 mg in the peel. In the flesh,
346 anthocyanins amounts were two times higher in caprified figs: 0.066 mg versus 0.035 mg.
347 This suggests that biosynthesis of phenolic compounds occurs later after pollination and could
348 be under effect of caprifification. Higher concentrations recorded in non-caprified figs could be
349 also explained by physiological and environmental conditions surrounding the fruit in the tree
350 when ripening occurs. Anthocyanins content of ripe fruit is affected by environmental
351 conditions and is enhanced by low rates of nitrogen fertilization ([Faragher, 1983](#); [Arakawa et](#)
352 [al., 1985](#)). In apples, anthocyanin accumulation is favoured by low temperatures, especially at
353 night. Flavonoids, before their positive effects on human health, are first recognized for their
354 physiological functions in plants as they increase the postharvest resistance of fruits to
355 pathogens ([Lattanzio, 2003](#)). Flavonoids synthesis is affected by light intensity and
356 wavelength, with fruits exposed to full sunlight containing more flavonoids than those in the
357 shade ([Awad et al., 2001](#)). Moreover it can be modified by temperature, humidity and
358 phytohormones, as demonstrated in citrus ([Arcas et al., 2000](#)).

359 The effect of caprifification on anthocyanins biosynthesis could be indirectly interpreted.
360 Anthocyanin pigments are assembled like all other flavonoids from two different streams of
361 chemical raw material in the cell. The first involves the shikimate pathway to produce the
362 amino acid phenylalanine. The second produces three molecules of malonyl-CoA, a C3 unit
363 from a C2 unit (acetyl-CoA) ([Sullivan, 1998](#)). More than five enzymes are thus required to
364 synthesize these pigments, each working in concert. Even a minor disruption in any of the
365 mechanisms of these enzymes by genetic or environmental factors would halt anthocyanin
366 production. Thus, the lack of pollination during fruit development could be offended in such
367 dis-functioning. While the biological burden of producing anthocyanins is relatively high,

368 plants benefit significantly from environmental adaptation, disease tolerance, and pest
369 tolerance provided by anthocaynins (Nakajima et al., 2001).

370 Multivariate analysis (variety, caprification and tissue type) with interactions effect was
371 performed based on fig's phenolic composition. The highest interaction effect was observed
372 between cultivar and compartment of the fruit (Table 4-C). Phenolic distribution between peel
373 and flesh varied from one cultivar to another. Dark skinned figs exhibited the highest
374 fluctuations and peel was richer than the flesh. Cyan-3-glucoside, cyan-3-rutinoside and
375 isoquercitrin were the most compound varying between the two distinct tissues. The two first
376 compounds were highly concentrated in the peel of dark coloured fruit ($p < 0.01$). Isoquercitrin
377 was present only in the peel tissue and concentrations are most important in cultivar 'BHL' (p
378 < 0.05). Levels of Isoquercitrin reached 24 $\mu\text{g}/\text{kg}$ FW in the peel of 'BHL' fruits; whereas,
379 concentrations were fewer in the peel of 'ZD' fruit (6 $\mu\text{g}/\text{kg}$ FW).

380 Caprification affected differently phenolic composition between cultivars. Cultivar \times
381 caprification interaction was significant as regards cyan-3-glucoside and cyan-3-rutinoside
382 concentrations. Cultivars 'BHL' and 'ZD' (dark skinned figs) were the most influenced by
383 lack of caprification traduced by an increase in anthocyanin levels when the fig reaches
384 maturity. Relationship between caprification and compartment of the fruit and interaction
385 effect on phenolic composition of figs showed that cyan-3-rutinoside is the only compound
386 affected by interaction of the two components (Table 4-C).

387 Interaction between the three components (cultivar \times treatment \times compartment) also exerted a
388 positive effect on cyan-3-glucoside concentrations and at minor level on cyan-3-rutinoside
389 amounts. Levels of two anthocyanins changed depending on cultivar and between separate
390 tissues of the fruit under effect of caprification. 'Zidi' fruit exhibits further the variation on
391 cyan-3-glucoside between peel and flesh under the effect of caprification.

392 Eventually, the first appreciation of caprification effect on quality of figs was observed in
393 fruit size and fresh weight. Caprifried figs were larger, fleshy and more prone to splitting, and
394 had a darker interior pulp colour. Caprification, consequently pollination, improved
395 pomological aspects of quality in figs like for many other common fruit species. Contrary to
396 physical aspects of quality, there is no commanding effect on biochemical traits related to cell
397 wall polysaccharides composition. Caprification could not be implied in chemical alterations
398 affecting cell wall of figs when ripe. Yet, it is important to retain that caprification relatively
399 improves textural properties of the fig receptacle by increasing methylation degree of the
400 pectin.

401 Anthocyanins, the main phenolics described in Tunisian figs, are relatively associated with
402 dark skinned fruit. There is a large discrepancy in levels of polyphenols between dark and
403 white coloured figs and between peel and flesh of the same fruit. Caprification obviously
404 altered concentrations of phenolic compounds. Anthocyanin concentrations were ultimately
405 affected and phenolic compounds concentrations increased in non-caprifried figs inversely
406 proportional to the size of the fruit. Non-caprifried retained syconia pursuit development on
407 the tree and produce remarkable small fruits when reaching maturity. These ripe receptacles
408 develop a pronounced pigmentation showing high levels of phenolic compounds which is
409 more visible in dark coloured fruit cultivars.

410

411 **4. Conclusions**

412 Caprification differently affected cell wall and phenolic contents in ripe figs. Contrary to cell
413 wall concentrations, which remain roughly proportional to fruit fresh weight, polyphenols
414 biosynthesis occurs before the last stage of fruit expansion. Anthocyanins, the major phenolic
415 compounds described in figs, concentrate more in the peel. Otherwise, *peel weight: total fruit*
416 *weight* ratio, higher in non caprifried figs, could explain the larger amount of polyphenols

417 determined in non caprifiged figs. Nevertheless, it is important to state that concentration per
418 fruit increases with caprifigication. The lack of caprifigication results on a loss in phenolics'
419 content per fruit due to decline of polyphenols biosynthesis in the last stage before ripening.
420 From a physiological point of view, caprifigication, otherwise pollination, may indirectly act as
421 a precursor of several hormonal activities. Hormones like ethylene, responsible of
422 physiological and biochemical changes in fruit development and maturation may be enhanced
423 by pollination. Structural components and secondary metabolites following two different
424 biosynthesis pathways play a commanding role in final quality of figs which remains
425 dependent on a good pollination unfolding ensured by a good practice of caprifigication during
426 receptivity of female fig syconium.

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471 **References**

- 472 **Arakawa, O., Hori, Y., Ogata, R.,** 1985. Relative effectiveness and interaction of ultraviolet-B, red
473 and blue light in anthocyanin synthesis of apple fruit. *Physiol. Plant.* 64, 323-327.
- 474 **Arcas, M.C., Botia, J.M., Ortuno, A.M.,** 2000. UV irradiation alters the levels of flavonoids
475 involved in the defence mechanism of *Citrus aurantium* fruits against *Penicillium digitatum*. *Eur. J.*
476 *Plant Path.* 106, 617-622.
- 477 **Awad, M.A., Wagenmakers, P.S., De Jager, A.,** 2001. Effects of light on flavonoid and chlorogenic
478 acid levels in the skin of 'Jonagold' apples. *Scientia Hort.* 88, 289-298.
- 479 **Baraket, G., Chatti, K., Saddoud, O., Mars, M., Marrakchi, M., Trifi, M., Salhi-Hannachi, A.,**
480 2009. Genetic analysis of Tunisian fig (*Ficus carica* L.) cultivars using amplified fragment length
481 polymorphism (AFLP) markers. *Scientia Hort.* 120, 487-492.
- 482 **Beck, N.G., Lord, E.M.,** 1988. Breeding System in *Ficus carica*, the Common Fig II Pollination
483 Events. *Am. J. Bot.* 75, 1913-1922.
- 484 **Blumenkrantz, N., Asboe-Hansen, G.,** 1973. New method for quantitative determination of uronic
485 acids. *Anal. Biochem.* 54, 484-489.
- 486 **Çaliskan, O., Polat, A.,** 2011. Phytochemical and antioxidant properties of selected fig (*Ficus carica*
487 L.) accessions from the eastern Mediterranean region of Turkey. *Scientia Hort.* 128, 473-478.
- 488 **Condit, L.J.,** 1947. The fig. *Chronica Botanica Co., Walthams, Mass. USA.* 222 pp.
- 489 **Crisosto, C.H., Bremer, V., Ferguson, L., Crisosto, G.M.,** 2010. Evaluating quality attributes of
490 four fresh figs (*Ficus carica* L.) cultivars harvested at two maturity stages. *HortScience* 45, 707-710.
- 491 **Del Caro, A., Piga, A.,** 2008. Polyphenol composition of peel and pulp of two Italian fresh fig fruits
492 cultivars (*Ficus carica* L.). *Eur. Food Res. Technol.* 226, 715-719.
- 493 **Duenas, M., Perez-Alonso, J.J., Santos-Buelga, C., Escribano-Bailon, T.,** 2008. Anthocyanin
494 composition in fig (*Ficus carica* L.). *J. Food Comp. Anal.* 21, 107-115.
- 495 **Englyst, H., Wiggins, H.S., Cummings, J.H.,** 1982. Determination of the nonstarch polysaccharides
496 in plant foods by gas-liquid chromatography of constituent sugars as alditol acetates. *Analyst* 107,
497 307-318.

- 498 **Faragher, J.D.**, 1983. Temperature regulation of anthocyanin accumulation in apple skin. *J. Exp. Bot.*
499 34, 1291-1298.
- 500 **Gaaliche, B., Trad, M., Mars, M.**, 2011. Effect of pollination intensity, frequency and pollen source
501 on fig (*Ficus carica* L.) productivity and fruit quality. *Scientia Hort.* 130, 737-742.
- 502 **Gross, K.C., Sams, C.E.**, 1984. Changes in cell wall neutral sugar composition during fruit ripening:
503 a species survey. *Phytochemistry* 23, 2457-2461.
- 504 **Guyot, S., Marnet, N., Sanoner, P., Drilleau, J-F.**, 2001. Direct thiolysis on crude apple materials
505 for high-performance liquid chromatography characterization and quantification of polyphenols in
506 cider apple tissues and juices. *Meth. Enzymol.* 335, 57-70.
- 507 **Harnly, J.M., Doherty, R.F., Beecher, G.R., Holden, J.M., Haytowitz, D.B., Bhagwat, S.**, 2006.
508 Flavonoid content of U.S. fruits, vegetables, and nuts. *J. Agric. Food Chem.* 54, 9966-9977.
- 509 **IPGRI-CIHEAM**, 2003. Descriptors for fig. International Plant Genetic Resources Institute, Rome,
510 Italy and International Centre for Advanced Mediterranean Agronomic Studies, Paris, France. ISBN
511 92-9043-598-4.
- 512 **Janick, J., Moore, N.J.**, 1975. *Advances in fruit breeding*, Purdue University Press, West Lafayette,
513 Indiana, USA. p. 621.
- 514 **Kislev, E.M., Hartmann, A., Bar-yosef, O.**, 2006. Early domesticated fig in the Jordan Valley.
515 *Science* 312, 1372-1374.
- 516 **Lattanzio, V.**, 2003. Bioactive polyphenols: Their role in quality and storability of fruit and
517 vegetables. *J. Appl. Bot.* 77, 128-146.
- 518 **LeBourvellec, C., Bouzerzour, K., Ginies, C., Regis, S., Plé, Y., Renard, M.G.C.C.**, 2011. Phenolic
519 and polysaccharidic composition of applesauce is close to that of apple flesh. *J. Food Comp. Anal.* 24,
520 537-547.
- 521 **Manach, C., Scalbert, A., Morand, C., Rémésy, C., Jiménez, L.**, 2004. Polyphenols: food sources
522 and bioavailability. *Am. J. Clin. Nutr.* 79, 727-747.
- 523 **Marlett, J.A., Vollendorf, N.W.**, 1994. Dietary fiber content and composition of different forms of
524 fruit. *Food Chem.* 51, 39-44.

- 525 **Mars, M., Chebli, T., Marrakchi, M.**, 1998. Multivariate analysis of fig (*Ficus carica* L.) germplasm
 526 in southern Tunisia. *Acta Hort.* 480, 75-81.
- 527 **Mars, M., Gaaliche, B., Ouerfelli, I., Chouat, S.**, 2009. Systèmes de production et ressources
 528 génétiques du figuier (*Ficus carica* L.) à Djebba et Kesra, deux villages de montagne au nord ouest de
 529 la Tunisie. *Rev. Régions Arides* 22, 33-45.
- 530 **Michailides, T.J., Morgan, D.P., Felts, D., Doster, M.A.**, 2008. Control of decay in caprifigs and
 531 calimyrna figs with fungicides. *Acta Hort.* 798, 269-275.
- 532 **Mohamed, M.S., Mrak, E.M.**, 1942. The Fig. Chemistry and food value. In: *The Fig*. Chronica
 533 Botanica Co., Walthams, Mass. USA. 222 pp.
- 534 **Nakajima, J., Tanaka, Y., Yamazaki, M., Saito, K.**, 2001. Reaction mechanism from
 535 leucoanthocyanidin to anthocyanidin 3-glucoside, a key reaction for colouring in anthocyanin
 536 biosynthesis. *J. Biol. Chem.* 276(28), 25797-803.
- 537 **Oukabli, A., Mamouni, A., Laghezali, M., Ater, M., Roger, J.P., Khadari, B.**, 2003. Local caprifig
 538 tree characterization and analysis of interest for pollination. *Acta Hort.* 605, 61-64.
- 539 **Owino, W.O., Nakano, R., Kubo, Y., Inaba, A.**, 2004. Alterations in cell wall polysaccharides
 540 during ripening in distinct anatomical tissue regions of the fig (*Ficus carica* L.) fruit. *Posthar. Biol.*
 541 *Technol.* 32, 67-77.
- 542 **Price, S.F., Breen, P.J., Valladao, M., Watson, B.T.**, 1995. Cluster sun exposure and quercetin in
 543 Pinot noir grapes and wine. *Am. J. Oenol. Viti.* 46, 187-194.
- 544 **Puech, A.A., Rebeiz, C.A., Catlin, P.B., Crane, J.C.**, 1975. Characterisation of anthocyanins in fig
 545 (*Ficus carica* L.) fruits. *J. Food Sci.* 40, 775-779.
- 546 **Rahemi, M., Jafari, M.**, 2008. Effect of Caprifig type on quantity and quality of Estahban dried fig
 547 *Ficus carica* cv. Sabz. *Acta Hort.* 798, 249-252.
- 548 **Renard, C.M.G.C.**, 2005. Variability in cell wall preparations: Quantification and comparison of
 549 common methods. *Carbohydr. Pol.* 60, 515-522.
- 550 **Renard, M.G.C.C., Dupont, N., Guillermin, P.**, 2007. Concentrations and characteristics of
 551 procyanidins and other phenolics in apples during fruit growth. *Phytochemistry* 68, 1128-1138.

- 552 **Renard, C.M.G.C., Ginies, C.,** 2009. Comparison of the cell wall composition for flesh and skin for
553 five different plums. Food chem. 114, 1042-1049.
- 554 **Saeman, J.F., Moore, W.E., Mitchell, R.L., Millett, M.A.,** 1954. Techniques for the determination
555 of flesh constituents by quantitative paper chromatography. Tappi, J. Tech. Assoc. Pulp Paper Ind. 37,
556 336-343.
- 557 **Shahidi, F., Naczk, M.,** 1995. Food phenolics, sources, chemistry, effects, applications. Lancaster,
558 PA: Technomic Publishing Co. Inc.
- 559 **Solomon, A., Golubowicz, S., Yablowicz, Z., Grossman, S., Bergman, M., Gottlieb, H.E., Altman,**
560 **A., Kerem, Z., Fleishman, M.A.,** 2006. Antioxidant activities and anthocyanin content of fresh fruits
561 of common fig (*Ficus carica* L.). J. Agric. Food Chem. 54, 7717-7723.
- 562 **Sullivan, J.,** 1998. Anthocyanin. International Carnivorous Plant Society. ICPS Newsletter.
563 September, 1998.
- 564 **Trad, M., Ginies, C., Gaaliche, B., Renard, C.M.G.C., Mars, M.,** 2012. Does pollination affect
565 aroma development in ripened fig [*Ficus carica* L.] fruit? Scientia Hort. 134, 93-99.
- 566 **Trad, M., LeBourvellec, C., Gaaliche, B., Renard, M.G.C.C., Mars, M.,** 2013. Nutritional
567 compounds in figs from southern Mediterranean region. Int. J. Food Prop. DOI:
568 10.1080/10942912.2011.642447.
- 569 **Veberic, R., Colaric, M., Stampar, F.,** 2008. Phenolic acids and flavonoids of fig fruit (*Ficus carica*
570 L.) in the northern Mediterranean region. Food Chem. 106, 153-157.
- 571 **Vinson, J.A.,** 1999. The functional food properties of figs. American Association of Cereal Chemists,
572 Inc. Cereal Foods World 44, 82-87.

Tables

Table 1. General description of the five Tunisian figs *Ficus carica*

Cultivar	Label	Botanical type	Origin	Shape ^a	Skin colour ^a	Pulp colour ^a
Bouhouli	BHL	San Pedro	Thibar	Oblate	Purple green	Amber
Zidi	ZD	Smyrna	Thibar	Oblong	Purple black	Red
Thgagli	THG	Smyrna	Thibar	Oblate	Yellow green	Amber
Bidhi	BD	Smyrna	Bekalta	Round	Yellow green	Pink
Khedri	KHD	Smyrna	Bekalta	Round	Yellow green	Pink

^a descriptors for figs (IPGRI and CIHEAM, 2003)

Table 2. Caprification effects on morphological aspects of the five Tunisian figs (means are displayed \pm SD, N=3)

		Fruit size (mm)	Fruit weight (g)	Flesh thickness (mm)	Skin part (%)	Dry weight (%)	Firmness (kg/cm ²)
<i>Pollinated</i>	BHL	64 \pm 2.0	112 \pm 4.0	19 \pm 1.5	14	19.7 \pm 2.5	0.34 \pm 0.02
	ZD	55 \pm 4.6	82 \pm 11.5	23 \pm 1.3	18	22.7 \pm 0.5	0.32 \pm 0.04
	THG	59 \pm 1.6	83 \pm 4.4	16 \pm 1.3	13	19.0 \pm 0.0	0.45 \pm 0.04
	BD	54 \pm 1.0	67 \pm 3.1	16 \pm 0.6	15	20.0 \pm 1.0	0.51 \pm 0.06
	KHD	54 \pm 1.6	63 \pm 3.1	17 \pm 1.1	12	20.3 \pm 0.5	0.51 \pm 0.11
	<i>Means</i>	57 \pm 4.5	81 \pm 18.5	18 \pm 3.0	14.4	20.3 \pm 1.7	0.43 \pm 0.05
<i>Non Pollinated</i>	BHL	49 \pm 1.8	65 \pm 8.9	14 \pm 1.2	16	17.7 \pm 0.5	0.38 \pm 0.02
	ZD	42 \pm 2.5	48 \pm 5.0	15 \pm 0.8	19	22.0 \pm 3.5	0.38 \pm 0.04
	THG	47 \pm 0.4	49 \pm 5.8	13 \pm 2.0	14	18.7 \pm 1.1	0.49 \pm 0.08
	BD	48 \pm 2.0	51 \pm 0.8	13 \pm 0.6	14	ND	0.57 \pm 0.06
	KHD	47 \pm 1.6	44 \pm 1.5	12 \pm 0.6	13	ND	0.68 \pm 0.09
	<i>Means</i>	47 \pm 3.2	51 \pm 8.9	13 \pm 1.4	15.2	19.4 \pm 2.7	0.50 \pm 0.06
Trt. <i>F</i> -value		179.16**	206.66**	131.20**	0.08	4.02*	6.09*

ND: not determined.

*: significant at $p < 0.05$, **: significant at $p < 0.01$. SD: Standard deviations

BHL: Bouhouli, ZD: Zidi, THG: Thgagli, BD: Bidhi, KHD: Khedri. Trt: treatment (caprification).

Table 3. AIS yields (mg.g⁻¹ FW), neutral sugars content (mg.g⁻¹ AIS) and degree of methylation (%) of the AIS isolated from peel and flesh of five Tunisian figs depending on caprification.

	Cv.	Yields	Rha	Fuc	Ara	Xyl	Man	Gal	Glc	Cellulose	AUA	MeOH(DM)	TS	
Peel	<i>Pollinated</i>	BHL	51	13	4	66	29	18	34	13	174	284	38 (73)	621
		ZD	55	13	5	63	39	21	35	12	202	339	32 (52)	717
		THG	59	11	5	63	33	21	33	16	196	339	37 (60)	699
		BD	49	13	6	84	46	20	75	19	263	258	29 (62)	766
		KHD	65	16	6	88	50	26	83	7	273	262	34 (72)	803
	<i>Means</i>	56	13	5	73	39	21	52	13	222	296	34 (64)	721	
	<i>Non-pollinated</i>	BHL	46	11	4	87	36	22	38	10	190	338	36 (58)	726
		ZD	52	14	4	62	38	19	34	13	187	331	31 (51)	688
		THG	50	13	4	67	32	21	32	11	194	346	33 (52)	708
		BD	49	15	7	86	51	28	90	6	291	254	32 (69)	822
KHD		54	12	6	77	48	25	74	5	262	269	31 (63)	772	
<i>Means</i>	50	13	5	76	41	23	54	9	225	308	33 (59)	743		
Flesh	<i>Pollinated</i>	BHL	110	9	3	45	32	12	32	10	145	245	30 (67)	522
		ZD	122	9	3	37	33	12	29	11	158	246	29 (65)	528
		THG	162	9	3	34	28	14	25	6	154	257	36 (77)	523
		BD	113	10	3	45	26	12	38	2	130	313	35 (62)	577
		KHD	148	6	2	34	17	8	30	2	88	364	36 (65)	549
	<i>Means</i>	131	9	3	39	27	12	31	6	135	285	33 (70)	540	
	<i>Non-pollinated</i>	BHL	130	9	3	37	37	12	25	10	145	251	29 (63)	518
		ZD	114	9	3	39	31	14	31	9	171	310	32 (57)	608
		THG	153	8	4	35	25	12	23	3	124	334	34 (56)	565
		BD	116	8	3	35	19	10	33	2	102	327	35 (58)	535
KHD		118	7	2	29	17	8	27	5	90	310	34 (60)	492	
<i>Means</i>	126	8	3	35	26	11	28	6	126	306	33 (59)	544		

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Table 3. (Continued). Significance levels of differences and interaction effects between variables

	Yields	Rha	Fuc	Ara	Xyl	Man	Gal	Glc	Cellulose	AUA	MeOH(DM)	TS
SD	35.7	1.1	0.5	4.2	2.9	2.1	3.1	2.6	13.5	13.5	3.2	77.1
Cv. <i>F</i> -value	6.81**	4.45**	12.07**	19.33**	7.58**	1.85	220.63**	32.67**	11.00**	8.50**	5.70**(4.07**)	11.28**
Trt. <i>F</i> -value	3.02	3.35	0.28	0.23	0.001	1.80	0.46	27.95**	1.09	12.49**	3.04 (14.16**)	3.26
Part. <i>F</i> -value	501**	325**	359**	1146**	329**	570**	777**	140**	798**	1.29	0.45(0.03)	614.05**
Cv. × Trt. <i>F</i> -value	1.86	2.7*	0.40	5.08**	3.50*	2.96*	3.72*	7.29**	1.15	5.62**	2.62(3.12*)	4.33**
Cv. × Part <i>F</i> -value	4.84**	8.12**	23.92**	19.00**	72.52**	20.00**	146.62**	19.61**	92.11**	39.36**	13.36**(7.6**)	11.28**
Trt. × Part <i>F</i> -value	0.14	0.02	0.26	8.68**	3.98	5.62*	8.17**	16.52**	2.61	0.80	0.92(0.09)	1.77
Cv. × Trt. × Part <i>F</i> -value	1.20	9.16**	2.92*	7.75**	2.29	7.64**	8.47**	8.47**	5.04**	8.39**	1.31(3.65*)	7.41**

BHL: ‘Bouhouli’; ZD: ‘Zidi’; THG: ‘Thgagli’; BD: ‘Bidhi’; KHD: ‘Khedri’.

Rha: rhamnose, Fuc: fucose, Ara: arabinose, Xyl: xylose, Man: mannose, Gal: galactose, Glc: glucose.

AUA: anhydro-uronic acid, MeOH: methanol, DM: degree of methylation

SD: standard deviation of the means. Trt.: treatment (caprification).

Table 4. Phenolic compounds changes (mg/kg FW), **A.** in the peel, **B.** in the flesh of five Tunisian figs with caprification.
C. Significance levels of differences and interactions effect between variables**A.** Phenolics in the peel

		Caffeoylquinic acid	Querc-3-rutinoside	Querc-3-glucoside	Querc-3-O-mal-glucoside ^a	Cyan-3-glucoside	Cyan-3-rutinoside	Total	
<i>Dark figs</i>	BHL	0.017 ±0.006	0.22 ±0.12	0.024 ±0.013	0.033 ±0.014	0.44 ±0.24	11.91 ±6.33	12.64	
	ZD	0.009 ±0.002	0.083 ±0.032	0.006 ±0.001	0.019 ±0.006	0.42 ±0.04	10.44 ±2.28	10.98	
	Means	0.01	0.15	0.02	0.03	0.43	11.18	11.81	
Pollinated	<i>White figs</i>	THG	0.004 ±0.003	0.18 ±0.04	0.010 ±0.002	0.019 ±0.003	0	0	0.21
		BD	0.021 ±0.011	0.24 ±0.13	0.010 ±0.005	0.027 ±0.015	0	0	0.30
		KHD	0.015 ±0.011	0.21 ±0.12	0.010 ±0.006	0.025 ±0.013	0	0	0.26
		Means	0.013	0.21	0.01	0.024	0	0	0.26
Non Pollinated	<i>Dark figs</i>	BHL	0.015 ±0.003	0.17 ±0.02	0.017 ±0.003	0.029 ±0.003	0.47 ±0.03	13.52 ±1.08	14.22
		ZD	0.018 ±0.004	0.16 ±0.03	0.011 ±0.002	0.033 ±0.005	0.76 ±0.12	18.50 ±2.10	19.64
		Means	0.017	0.17	0.014	0.031	0.62	16.01	16.93
		<i>White figs</i>	THG	0.009 ±0.001	0.168 ±0.009	0.010 ±0.001	0.019 ±0.001	0	0
BD	0.015 ±0.007		0.13 ±0.05	0.006 ±0.001	0.019 ±0.007	0	0	0.17	
KHD	0.018 ±0.009		0.17 ±0.02	0.007 ±0.001	0.022 ±0.004	0	0	0.22	
Means	0.014		0.156	0.008	0.02	0	0	0.20	

Values are means ± SD (N=3).

BHL: Bouhouli, ZD: Zidi, THG: Thgagli, BD: Bidhi, KHD: Khedri.

Querc-3-rutinoside : quercetin-3-rutinoside. Cyan-3-rutinoside : cyanidin-3-rutinoside.

^a quercetin-3-O-malonyl glucoside was identified by HPLC-MS.

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B. Phenolics in the flesh

		Caffeoylquinic acid	Querc-3-rutinoside	Querc-3-glucoside	Querc-3-O-mal-glucoside	Cyan-3-glucoside	Cyan-3-rutinoside	Total	
Pollinated	<i>Dark figs</i>	BHL	0.002 ±0.001	0	0	0	0.055 ±0.010	0.63 ±0.13	0.69
		ZD	0	0	0	0	0.055 ±0.010	1.01 ±0.09	1.07
		Means	0.001	0	0	0	0.055	0.82	0.88
	<i>White figs</i>	THG	0	0	0	0	0.062 ±0.023	0.99 ±0.12	1.05
		BD	0.003 ±0.00	0	0	0	0.12 ±0.04	0.73 ±0.14	0.85
		KHD	0.004 ±0.001	0	0	0	0.25 ±0.03	1.02 ±0.20	1.27
Means		0.002	0	0	0	0.144	0.913	1.06	
Non Pollinated	<i>Dark figs</i>	BHL	0.003 ±0.001	0	0	0	0.062 ±0.009	0.58 ±0.09	0.65
		ZD	0	0	0	0	0.041 ±0.008	0.75 ±0.15	0.79
		Means	0.002	0	0	0	0.05	0.67	0.72
	<i>White figs</i>	THG	0	0	0	0	0.048 ±0.002	0.82 ±0.06	0.87
		BD	0.007 ±0.001	0	0	0	0.25 ±0.07	0.89 ±0.07	1.15
		KHD	0.006 ±0.001	0	0	0	0.173 ±0.004	0.89 ±0.17	1.07
Means		0.004	0	0	0	0.157	0.87	1.03	

C. Significance levels of differences

	Caffeoylquinic acid	Querc-3-rutinoside	Querc-3-glucoside	Querc-3-O-mal-glucoside	Cyan-3-glucoside	Cyan-3-rutinoside	Total
Cv. <i>F</i> -value	5.07**	0.82 NS	4.72**	1.23 NS	36.83**	56.60**	53.46**
Trt. <i>F</i> -value	1.41NS	0.80 NS	0.56 NS	0.002 NS	5.29*	4.60*	4.35*
Part. <i>F</i> -value	71.47**	144.37**	114.15**	205.45**	29.81**	112.91**	114.79**
Cv. × Trt. <i>F</i> -value	0.52 NS	1.08 NS	0.87 NS	1.13 NS	3.78*	2.95*	2.95*
Cv. × Part <i>F</i> -value	0.99 NS	0.82 NS	4.72*	1.23 NS	75.66**	59.18**	57.44**
Trt. × Part <i>F</i> -value	0.04 NS	0.80 NS	0.56 NS	0.002 NS	3.57 NS	5.34*	4.95*
Cv. × Trt. × Part <i>F</i> -value	1.38 NS	1.08 NS	0.87 NS	1.13 NS	4.85**	3.28*	3.31*

NS, non significant; * significant at $p < 0.05$, ** significant at $p < 0.01$.

Cv.: cultivar, Trt.: treatment (caprification), Part: compartment of the fruit (peel or flesh).

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