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

3

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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.

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46  
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## 51 Abbreviations

52 BHL<sup>1</sup>

53 ZD<sup>2</sup>

54 THG<sup>3</sup>

55 BD<sup>4</sup>

56 KHD<sup>5</sup>

57 AIS<sup>6</sup>

58 HPLC-DAD<sup>7</sup>

59 MHDP<sup>8</sup>

60 GC-MS<sup>9</sup>

61 GC-FID<sup>10</sup>

62 PTFE<sup>11</sup>

63 SPSS<sup>12</sup>

64 GLM<sup>13</sup>

65 DM<sup>14</sup>

66 PG<sup>15</sup>

67 AUA<sup>16</sup>

68

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<sup>1</sup> Bouhouli

<sup>2</sup> Zidi

<sup>3</sup> Thgagli

<sup>4</sup> Bidhi

<sup>5</sup> Khedri

<sup>6</sup> Alcohol insoluble solids

<sup>7</sup> High performance liquid chromatography - diode array detector

<sup>8</sup> *m*-hydroxydiphenyl

<sup>9</sup> Gas chromatography - mass spectrometry

<sup>10</sup> Gas chromatography - flame ionisation detector

<sup>11</sup> Polytetrafluoroethylene

<sup>12</sup> Statistical Package for the Social Sciences

<sup>13</sup> General linear model

<sup>14</sup> Degree of methylation

<sup>15</sup> Degree of methylation

<sup>16</sup> 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 Naczki, 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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Tables

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

Cultivar	Label	Botanical type	Origin	Shape <sup>a</sup>	Skin colour <sup>a</sup>	Pulp colour <sup>a</sup>
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

<sup>a</sup> 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 <sup>2</sup> )
<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<sup>-1</sup> FW), neutral sugars content (mg.g<sup>-1</sup> 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		

Comment citer ce document :

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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 <sup>a</sup>	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	
	<b>Means</b>	0.01	0.15	0.02	0.03	0.43	11.18	11.81	
<b>Pollinated</b>	<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
		<b>Means</b>	0.013	0.21	0.01	0.024	0	0	0.26
<b>Non Pollinated</b>	<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
		<b>Means</b>	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	
<b>Means</b>	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.

<sup>a</sup> quercetin-3-O-malonyl glucoside was identified by HPLC-MS.

Comment citer ce document :

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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	
		<b>Means</b>	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	
		<b>Means</b>	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
<b>Means</b>			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	
		<b>Means</b>	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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