Caprification modifies polyphenols but not cell wall concentrations in ripe figs

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Abstract

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

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

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

1. Introduction

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

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

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

2. Materials and methods

2.1. Fruit sampling and preparation

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

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

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

2.2. Cell wall isolation and analysis

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

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

2.3. Polyphenols in figs

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

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

2.4. Statistics

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

3. Results and discussion

3.1. Morphological aspects of figs and caprification effect

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

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

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

3.2. Changes in cell wall of figs

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

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

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

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

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

The overall interaction between the three factors (cv. $\times$ trt. $\times$ part) was significant ($p < 0.01$) for 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

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

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

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

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

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

Anthocyanins remain the major contributors to colour of the purple figs. Non caprifiged fruits were smaller but had a pronounced coloured skin which wrapped all surface of the receptacle. 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 caprification. 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 caprification 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,

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

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

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

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

determined in non caprifiged figs. Nevertheless, it is important to state that concentration per fruit increases with caprification. The lack of caprification results on a loss in phenolics' content per fruit due to decline of polyphenols biosynthesis in the last stage before ripening. From a physiological point of view, caprification, otherwise pollination, may indirectly act as a precursor of several hormonal activities. Hormones like ethylene, responsible of physiological and biochemical changes in fruit development and maturation may be enhanced by pollination. Structural components and secondary metabolites following two different biosynthesis pathways play a commanding role in final quality of figs which remains dependent on a good pollination unfolding ensured by a good practice of caprification during 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 ^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	Pollinated	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
		Means	56	13	5	73	39	21	52	13	222	296	34 (64)	721
	Non-pollinated	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
		Means	50	13	5	76	41	23	54	9	225	308	33 (59)	743
Flesh	Pollinated	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
		Means	131	9	3	39	27	12	31	6	135	285	33 (70)	540
	Non-pollinated	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
		Means	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	0.21
		BD	0.021 ±0.011	0.24 ±0.13	0.010 ±0.005	0.027 ±0.015	0	0.30
		KHD	0.015 ±0.011	0.21 ±0.12	0.010 ±0.006	0.025 ±0.013	0	0.26
		Means	0.013	0.21	0.01	0.024	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
		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
		Means	0.017	0.17	0.014	0.031	0.62	16.01
	<i>White figs</i>	THG	0.009 ±0.001	0.168 ±0.009	0.010 ±0.001	0.019 ±0.001	0	0.21
		BD	0.015 ±0.007	0.13 ±0.05	0.006 ±0.001	0.019 ±0.007	0	0.17
		KHD	0.018 ±0.009	0.17 ±0.02	0.007 ±0.001	0.022 ±0.004	0	0.22
		Means	0.014	0.156	0.008	0.02	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	0.055 ±0.010	0.63 ±0.13	0.69
		ZD	0	0	0	0.055 ±0.010	1.01 ±0.09	1.07
		Means	0.001	0	0	0.055	0.82	0.88
	<i>White figs</i>	THG	0	0	0	0.062 ±0.023	0.99 ±0.12	1.05
		BD	0.003 ±0.00	0	0	0.12 ±0.04	0.73 ±0.14	0.85
		KHD	0.004 ±0.001	0	0	0.25 ±0.03	1.02 ±0.20	1.27
		Means	0.002	0	0	0.144	0.913	1.06
	<i>Non Pollinated</i>	<i>Dark figs</i>	BHL	0.003 ±0.001	0	0.062 ±0.009	0.58 ±0.09	0.65
			ZD	0	0	0.041 ±0.008	0.75 ±0.15	0.79
			Means	0.002	0	0.05	0.67	0.72
		<i>White figs</i>	THG	0	0	0.048 ±0.002	0.82 ±0.06	0.87
			BD	0.007 ±0.001	0	0.25 ±0.07	0.89 ±0.07	1.15
		KHD	0.006 ±0.001	0	0	0.173 ±0.004	0.89 ±0.17	1.07
		Means	0.004	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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