Journal homepage: http://www.ifrj.upm.edu.my

International FOOD <u>RESEARCH</u> Journat

Changes in phytochemical compounds and antioxidant potential of fresh, frozen, and processed figs (*Ficus carica* L.)

*Petkova, N., Ivanov, I. and Denev, P.

Department of Organic Chemistry and Inorganic Chemistry, University of Food Technologies, 26 Maritza Blvd., Plovdiv, 4002, Bulgaria

Article history

<u>Abstract</u>

Received: 30 March, 2019 Received in revised form: 18 October, 2019 Accepted: 23 October, 2019

<u>Keywords</u>

Ficus carica L. Carotenoids Anthocyanins Polyphenols Sugars Antioxidant capacity The aim of the present work was to evaluate some phytochemical compounds in fresh, frozen, and processed yellow-coloured figs (*Ficus carica* L.). The moisture content, ash, titrable acidity, total chlorophylls, total carotenoids, total phenols, total flavonoids, total anthocyanins, as well as carbohydrates (fructose, glucose, and sucrose) were evaluated. In addition, the antioxidant capacities of fresh, frozen, and processed figs (jam) were determined by DPPH and FRAP assays. The highest values of bioactive compounds and antioxidant activity were found in fresh fruits, while in frozen fruits their levels decreased significantly. The preparation of fig jam was a better approach for preserving some bioactive compounds, especially carotenoids and phenolic compounds. After four months of storage at -18°C, it was observed that there were significant losses in the antioxidant potential, decrease in sucrose content, as well as total phenols, total flavonoids, and total anthocyanins values. In spite of the reduction in some valuable compounds, jam processing retained good amount of them during storage in comparison with freezing storage.

© All Rights Reserved

Introduction

Fig (*Ficus carica* L.) is a deciduous tree from the Moraceae family, native to southwest Asia, and typically grown in the Mediterranean region. This plant is one of the earliest cultivated trees. Fig fruits are an important crop worldwide for both dry and fresh consumptions (Çaliskan and Aytekin Polat, 2011; Tanwar *et al.*, 2014) with about one million tons being produced annually (Hossain *et al.*, 2010). Turkey, Greece, Egypt, Morocco, Italy, Spain, Brazil, and other countries were among the main fig producers due to the proper climatic conditions such as hot and dry summers, and mild winters (Soni *et al.*, 2014).

The colour of figs varies from dark purple to green (Solomon *et al.*, 2006). The consumption of figs has positive health effects due to the numerous nutraceutical compounds that may help prevent cardiovascular diseases and the growth of carcinoma cells (Allegra *et al.*, 2017). Fresh and dried figs, as well as its syrup possess laxative action (Morton, 1987). The consumption of figs is recommended

good for eyesight as well as for liver and spleen diseases (Gani *et al.*, 2018). Figs are used as an expectorant and diuretic. In addition, juice from figs mixed with honey can be used for haemorrhages (Soni *et al.*, 2014). Oily macerates prepared from dried figs were also used for consumption because of the antioxidative and antimicrobial properties (Debib *et al.*, 2018).

The dried and fresh figs are reported to be a good source of amino acids, carbohydrates, sugars, fibres, minerals (copper, manganese, magnesium, potassium, and calcium), vitamins, organic acids, and phenolic compounds (Veberic *et al.*, 2008; Slatnar *et al.*, 2011) similar to some edible mushrooms (Dospatliev, 2018). The phytochemicals of figs also includes arabinose, β -amyrins, β -carotenes, glycosides, β -sitosterols, xanthotoxol, alkaloids, flavonoids, coumarins, saponins, and terpenes (Gilani *et al.*, 2008; Jeong *et al.*, 2009). Fresh fig is an important source of polyphenols such as rutin (up to 28.7 mg/100 g fresh weight (fw), (+)-catechin, (-)-epicatechin, chlorogenic acid (up to 1.71 mg/100 g fw), gallic acid (up to 0.38 mg/100 g fw) and

syringic acid (up to 0.10 mg/100 g fw) (Veberic *et al.*, 2008). Moreover, hydroxycinnamic acids (3-O- and 5-O-caffeoylquinic acids and ferulic acid), flavonoid glycosides (quercetin-3-O-glucoside and quercetin-3-O-rutinoside) and furanocoumarins as psoralen and bergapten have also been found in figs (Oliveira *et al.*, 2009; Debib *et al.*, 2014). Because of these natural compounds, figs are an important constituent in the Mediterranean diet.

However, figs are usually commercially available after drying because fresh fruits are only available during the season. The fresh fruits have a short postharvest life (7 - 10 days). A combination of cooler conditions and a modified atmosphere packaging can extend storage for up to 2 - 4 weeks (Sozzi *et al.*, 2005). The popular approach for preserving figs is through jam processing (Rababah *et al.*, 2011; Tanwar *et al.*, 2014). In Bulgaria, fig jam with whole green and ripen fruits is a very popular type of preservation of these seasonal fruits.

However, studies that demonstrated the behaviour of total phenolics, antioxidant activity, and anthocyanins of fig jams during long-term storage are rather limited. The effect of fruit jam on antioxidant activity, total phenolic, and anthocyanin components has been evaluated by only a few researchers (Rababah *et al.*, 2011; Tanwar *et al.*, 2014).

Furthermore, the effect of jam processing and its storage on the nutraceutical contents of figs is of a great importance in terms of the nutritional value of the final product. For these reasons, the effects of storage conditions upon the pigments, sugars, and polyphenol compounds present interest for quality of preserved foods. The evaluation of the influence of food processing or cold storage on natural occurring antioxidants and nutritional composition of fig products is a significant factor to preserve or improve their biological activity.

Therefore, the aim of the present work was to evaluate the influence of cold storage and processing on the physicochemical properties and phytochemical compounds in figs.

Materials and methods

Standards and reagents

All standards and reagents used in the present work were of analytical grade and purchased from Sigma-Aldrich (Germany).

Plant materials

Fresh fig samples of cultivar "Croatian Yellow Giant" (yellow-green colour fruits) were collected

during 2015 in Plovdiv province located in the central part of Bulgaria. Figs were harvested at their fully mature stage (August) in three replicates. For the phytochemical analysis, harvested fig samples were washed, blended, frozen, and stored at -18°C before usage. Another one part of the samples was frozen for four months prior to analyses. The final part of figs were processed into fig jam.

Preparation of fig jam

The jam processing was conducted as follows: the fresh figs were sorted, washed, and used whole without cutting or blending. The jam formulation included 2 kg of figs, 800 g of sugar, 250 g of water, 2.5 g/kg of citric acid, and 4 g/kg of high methoxy pectin. The fruits were heated at 80°C to inactivate the enzymes. Then, sucrose which was dissolved previously in water was added. The mixture was boiled to a final concentration of soluble solid value around 650 g/kg (approximately 104 - 105°C of the final boiling points). Then, pectin was added under a manual agitation, and the mixture was heated for 10 min to allow proper pectin hydration. The jam with whole fruits was hot-packed at 85°C in 100 g glass jars, immediately sealed with a metal cover, and inverted for 5 min to sterilise the glass containers. The samples were stored at 25°C for four months.

Physico-chemical analyses

The moisture content of the figs and jam were analysed following the Association of Official Analytical Chemists' method 981.12 (AOAC, 2007). Samples were dried at 105 \pm 1°C to a constant weight. The ash content the figs and jam determined by muffle incineration at 550°C. The titratable acidity content of the figs and jam was analysed by titration with 0.1 N NaOH up to pH 8.1 using a pH meter 7110 WTW (Germany) and expressed as g citric acid/100 g fw (ISO, 1998). The pH of the figs and jam was determined using a pH meter 7110 WTW (Germany).

Total chlorophylls and carotenoids contents

Figs and jam were extracted with acetone (80%) for evaluation of chlorophyll a, chlorophyll b, total chlorophylls, and the total carotenoids in the extracts. The absorbance of the mixed extracts was recorded at three wavelengths of 645, 633 and 470 nm with a spectrophotometer Campsec (UK). The analysis was done in triplicate for each extracts. The total chlorophyll, chlorophyll a, and chlorophyll b concentrations (mg/mL) were calculated according to Lichtenthaler and Wellburn (1983) and re-expressed as $\mu g/g$ fw.

Extraction

Figs (2 g) were extracted with 70% (v/v) ethanol in solid to liquid ratio 1:20 (w/v). The extraction procedure was performed in an ultrasonic bath (VWR, Malaysia, 45 kHz and 30 W) for 15 min, at 45°C (Petkova *et al.*, 2014a). Each extract was filtered out using a Whatman No. 3 filter paper and allowed to set in the dark until the analysis of the phytochemical compounds.

Total soluble carbohydrate content

The total soluble carbohydrate content was estimated by phenol-sulphuric acid method (DuBois *et al.*, 1956). Fig extracts (0.1 mL) were mixed with 5% of phenol (1 mL), 5 mL of concentrated sulphuric acid and placed in a water bath at 30°C for 20 min. The absorbance was measured at 490 nm against a blank prepared with dH₂O. The amount of carbohydrates presented was determined from the calibration curve for glucose, and the results were calculated as g/100 g fw.

Reducing sugars content

The reducing sugars were analysed by the 4-hydroxybenzoic acid hydrazide (PAHBAH) method (Lever, 1972). PAHBAH reagent (0.75 mL) was added to 0.25 mL properly diluted fig extract and boiled for 5 min, then cooled in the ice bath for 5 min. The absorbance was measured at 410 nm against a blank prepared with dH₂O.

HPLC analysis of mono- and disaccharides

The chromatographic determination of sugars was performed on an HPLC instrument (Elite Chrome Hitachi) with a refractive index detector (RID) Chromaster 5450, column Shodex[®] Sugar SP0810 (300 mm × 8.0 mm i.d.) with Pb²⁺ and guard column Shodex SP-G (5 μ m, 6 × 50 mm) operating at 85°C. The mobile phase was dH₂O and run at a flow rate of 1.0 mL/min with an injection volume of 20 μ L (Petkova *et al.*, 2014b).

Total phenolic contents

Folin-Ciocalteu reagent was used for the evaluation of the total phenolic content (Stintzing *et al.*, 2005). The reagent was diluted five times (1 mL), and mixed with 0.2 mL of fig extract, and then 0.8 mL of 7.5% Na₂CO₃ was added to them. Following 20 min incubation at room temperature in the dark, the absorbance was measured at 765 nm against a blank. The results were expressed as mg equivalent of gallic acid (GAE) per g fresh weight (fw) (Ivanov *et al.*, 2014).

The total flavonoids content

Al(NO₃)₃ reagent was used for the total flavonoid content (Kivrak *et al.*, 2009). Briefly, 1 mL of properly diluted fig extract was mixed with 100 μ L of 10% Al(NO₃)₃, 100 μ L of 1 M potassium acetate and 3.8 mL of dH₂O. The mixture was incubated for 40 min at room temperature. The absorbance was measured at 415 nm against blank prepared without the addition of 10% Al(NO₃)₃. The results were presented as mg equivalents quercetin (QE) per g fresh weight (fw) according to the calibration curve.

Determination of total monomeric anthocyanins

The total anthocyanins content was determined by the pH differential method (Lee *et al.*, 2005). The absorbance of the sample was measured at 520 and 700 nm, and expressed as cyanidin-3-glycoside (Eq. 1). Data was reported as means of cyanidin-3glycoside per 100 g of fresh weight (fw).

$$TMA = \frac{A \times MW \times DF \times 1000 \text{ mg/L}}{\varepsilon \times 1} \qquad (Eq. 1)$$

where A = $(A_{520nm} - A_{700nm})$ pH 1.0 - $(A_{520nm} - A_{700nm})$ pH 4.5, MW (molecular weight) = 449.2 g/mol cyanidin-3-glycoside (cyd-3-glu), DF = dilution factor, 1000 = factor for conversion from g to mg, ε = molar coefficient of 26900 L × mol⁻¹ × cm⁻¹ for cyd-3-glu, and 1 = path length in cm.

The DPPH radical-scavenging ability

Seventy percent of ethanol fig extracts (0.15 mL) were mixed with 2.85 mL freshly prepared 0.1 mM DPPH in methanol. The mixture was incubated for 15 min at 37°C in the dark. The reduction of absorbance was measured at 517 nm in comparison to the blank containing methanol and the percentage inhibition was calculated. The results were expressed in 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) as mM Trolox[®] equivalents (TE) per g fresh weight (fw) (Ivanov *et al.*, 2014).

Ferric reducing antioxidant power (FRAP) assay

The assay was performed according to Benzie and Strain (1996) with slight modification. The FRAP reagent was prepared by mixing 10 parts of 0.3 M acetate buffer (pH 3.6), 1 part of 10 mM 2,4,6tripyridyl-s-triazine (TPTZ) in 40 mM HCl and 1 part of 20 mM FeCl₃.6H2O in dH₂O. FRAP reagent (3.0 mL) was added to 0.1 mL of the extract. The reaction time was 10 min at 37°C in the dark, and the absorbance was measured at 593 nm against a blank. The antioxidant activity was expressed as mM Trolox equivalents (TE) per g fresh weight (fw) (Ivanov *et al.*, 2014).

Statistical analysis

All analyses were performed in triplicates (n = 3). The data were presented as mean values \pm standard deviation (SD). Statistical analysis was performed using MSExcel 2010. A difference was considered statistically significant, when p < 0.05. Statistical significance at p < 0.05 was determined by ANOVA (one and two way) followed by Tukey's multiple comparison tests.

Results and discussion

Physico-chemical parameters

The moisture contents for fresh figs and fig jam were 81.1% and 63.8%, respectively (Table 1). This result is in agreement with Tanwar *et al.* (2014) for fig pulp which had 87.7% moisture content.

The highest pH values measured was for fresh figs (4.86), followed by frozen figs (4.25) and fig jam (4.00). These values are almost similar to those reported by Silva *et al.* (2009) for untreated and irradiated figs. Additionally, these values were higher than those reported for fruits (4.0 - 3.5) and jams (3.7 - 3.0), by Rababah *et al.* (2011) and Tanwar *et al.* (2014), respectively. The pH of fig jam was lower than fruits due to the addition of citric acid as preservative. Similar trends of decreasing pH were also reported in apricot and fig jams during five months of storage as reported by Rababah *et al.* (2011).

The titrable acidity (TA) for fresh figs and jam are in an accordance with the reported results for some fig cultivars from Algeria (Mahmoudi *et al.*, 2018) and processed fruits (Tanwar *et al.*, 2014).

Table 1. Moisture, ash, pH, and titrable acidity of fresh, frozen, and processed figs (*Ficus carica* L.)

Sample	Moisture (%)	Ash (%)	pН	TA (g citric acid/100 g)
Fresh fruits	$\begin{array}{c} 81.10 \pm \\ 0.20^{a} \end{array}$	$\begin{array}{c} 0.98 \pm \\ 0.15^{\text{ac}} \end{array}$	$\begin{array}{c} 4.86 \pm \\ 0.05^{\text{b}} \end{array}$	$\begin{array}{c} 0.19 \pm \\ 0.05^a \end{array}$
Frozen fruits	$\begin{array}{c} 83.50 \pm \\ 0.50^{\mathrm{b}} \end{array}$	$0.78 \pm 0.05^{\mathrm{b}}$	$\begin{array}{c} 4.25 \pm \\ 0.03 c \end{array}$	0.21 ± 0.03^{b}
Jam	$\begin{array}{c} 63.80 \pm \\ 0.10^{\circ} \end{array}$	$1.11 \pm 0.08^{\rm b}$	$\begin{array}{c} 4.00 \pm \\ 0.04^a \end{array}$	$\begin{array}{c} 0.26 \pm \\ 0.06^{\rm ac} \end{array}$

Data area means of triplicates $(n=3) \pm$ standard deviation (SD). Means in the same column with different letters are significantly different at p < 0.05 by Tukey's multiple comparison test.

Chlorophyll and carotenoid contents

In plants and vegetables, the pigmentation is mainly due to the presence of bioactive natural compounds such as anthocyanins, chlorophylls, or carotenoids. Figs possess wide diversity of colours, ranging from dark purple to green (Solomon *et al.*, 2006). The total chlorophylls and total carotenoids of fresh, frozen, and processed figs are summarised in Table 2.

In general, all fig samples contained more chlorophylls than the total carotenoids. The values for chlorophylls (a, b, and total) and total carotenoids obtained in the present work are higher in comparison with the reported values of these natural pigments in the fresh and irradiated green-coloured figs (Silva et al., 2009). The decrease in the total chlorophyll content in the present work was observed after freezing. The highest content of carotenoids in the whole fruits was found in fresh fruits with $3.5 \ \mu g/g$ fw, while in frozen fruits and jam, their contents decreased by approximately 50% to 1.7 and 1.9 μ g/g fw, respectively. These values are almost similar to those in peels of two fig cultivars 'Dottato' and 'Melanzana' of 4.73 and 4.04 µg/g, respectively (Allegra et al., 2017). Salunkhe et al. (1991) reported that carotenoids can lose its provitamin A activity through oxidation during processing. However, the values for the total carotenoid content obtained in the present work are higher than the results for β -carotene in the fig pulp and jams of 0.16 and 0.01 mg/100 g fw, respectively (Tanwar et al., 2014). Contrary to the observation of Tanwar et al. (2014) for 93% reduction of β -carotene in fig jam, the present work demonstrated that the reduction of total carotenoids in fig jam was no more than 45%. In the frozen figs, their losses were 50%. Therefore, almost half of the total carotenoid content, as potential antioxidant, could be preserved during the thermal processing and the cold storage (Table 2).

Table 2. Chlorophylls and carotenoids of 80% acetoneextracts of fresh, frozen, and processed figs

(<i>Ficus carica</i> L.) (μ g/g fw).				
Sample	Chlorophyll a	Chlorophyll b	Total chlorophylls	Total carotenoids
Fresh fruits	$\begin{array}{c} 3.64 \pm \\ 0.45^a \end{array}$	$2.31 \pm 0.25^{\text{b}}$	$\begin{array}{c} 5.95 \pm \\ 0.52^a \end{array}$	$\begin{array}{c} 3.52 \pm \\ 0.12^{ab} \end{array}$
Frozen fruits	$\begin{array}{c} 1.95 \pm \\ 0.15^{\text{b}} \end{array}$	$\begin{array}{c} 2.04 \pm \\ 0.32^{a} \end{array}$	$\begin{array}{c} 3.99 \pm \\ 0.45^{\circ} \end{array}$	$\begin{array}{c} 1.73 \pm \\ 0.15^a \end{array}$
Jam	1.68 ± 0.25°	$\begin{array}{c} 6.87 \pm \\ 0.22^{\texttt{b}} \end{array}$	$\begin{array}{c} 8.55 \pm \\ 0.30^{\text{b}} \end{array}$	1.94 ± 0.45°

Data area means of triplicates $(n = 3) \pm$ standard deviation (SD). Means in the same column with different letters are significantly different at p < 0.05 by Tukey's multiple comparison test.

Carbohydrate content

The carbohydrate content, especially sugar composition, influences the perceived fruit sweetness. Therefore, sugar content is an important quality characteristic of figs. The carbohydrate contents of fresh and processed figs are summarised in Table 3.

Table 3. Carbohydrate contents in fresh, frozen, and processed figs (*Ficus carica* L.) (g/100 g fw).

Sample	Total soluble carbohydrate	Reducing sugars	Sucrose	Glucose	Fructose
Fresh fruits	$23.5 \pm 2.3^{ m b}$	10.5 ± 3.3ª	1.4 ± 0.1 ^b	$\begin{array}{c} 5.7 \pm \\ 0.1^{\text{b}} \end{array}$	$\begin{array}{c} 5.8 \pm \\ 0.2^{\text{b}} \end{array}$
Frozen fruits	$\begin{array}{c} 19.2 \pm \\ 1.3^a \end{array}$	5.8± 1.3°	absent	$\begin{array}{c} 1.8 \pm \\ 0.3^{\rm a} \end{array}$	${1.8}_{-\pm}{0.2^{\rm b}}$
Jam	$\begin{array}{c} 65.9 \pm \\ 0.3^{\circ} \end{array}$	$\begin{array}{c} 10.7 \pm \\ 2.5^{\text{b}} \end{array}$	$\begin{array}{c} 60.4 \pm \\ 0.5^a \end{array}$	$\begin{array}{c} 4.3 \pm \\ 0.2^{\rm c} \end{array}$	4.1 ± 0.1°

Data area means of triplicates $(n = 3) \pm$ standard deviation (SD). Means in the same column with different letters are significantly different at p < 0.05 by Tukey's multiple comparison test.

The detected sugars in all investigated samples were mainly sucrose, glucose, and fructose (Table 3). In fresh figs, the values of glucose and fructose were higher than sucrose, 5.8 g/100 g fw and 1.4 g/100 g fw, respectively. In frozen figs, sucrose was not detected and the content of monosaccharides deceased three times in comparison with the fresh figs (Table 3). This could be due to the lower temperature of the storage. In fig jam, the values of glucose and fructose were comparable with those of fresh figs, but sucrose content was significantly higher, mainly due to its addition to the jam processing. The total carbohydrates in fresh figs reached 23.5 g/100 g fw, as almost half of them were reducing sugars (glucose and fructose). In frozen figs, the reducing sugars decreased by 50%, while the content of total carbohydrates was 19 g/100 g fw. In fig jam, the total carbohydrates reached 65.9 g/100 g fw, as the reducing sugars were comparable with their content in fresh figs. These values are comparable with the report on fresh figs from the different varieties from Algeria which demonstrated that the pulp was four times sweeter than the peel of 19.475 and 4.843 g/100 g fw, respectively (Mahmoudi *et al.*, 2018). The levels of fructose found in the present work were in the range of 5.8 to 1.8 g/100 g which is an important feature for fig quality. According to Çaliskan and Aytekin Polat (2011), the perception of sweetness in fig is due to the prevalence of fructose.

Phenolic content and antioxidant activity

The decrease in values of the total phenolic compounds and the antioxidant activity was observed after freezing and thermal processing of figs (Table 4). The total polyphenolic content showed significant differences depending on the type of storage of figs. The highest content of polyphenols was found in fresh figs, followed by fig jam (Table 4). The lowest levels were detected in the frozen figs as the polyphenols were two times lower in comparison of fresh figs of 14 mg GAE/100 g fw. The total phenols in the investigated fresh figs were lower than some reported values which ranged from from 553.27 to 1276.22 $\mu g/g$ (Allegra *et al.*, 2017); and some were higher than our results ranging from 1224.24 mg GAE/kg to 56.0 - 74.9 mg GAE/100 g fw (Solomon *et al.*, 2006; Pande and Akoh, 2010; Rababah et al., 2011; Slatnar et al., 2011). The reported total phenolic content in fresh fruits $(31.0 \pm 2.0 \text{ mg GAE}/100 \text{ g})$ in the present work were comparable to those of Çaliskan and Aytekin Polat (2011). Bucić-Kojić et al. (2011) measured similar total phenolic values in the figs as compared to purple figs.

In the present work, the losses of total phenolic content did not exceed 10% in jam; while following storage in the fridge, their losses reached 45%. This figure is small when compared to other reports. For example, the total phenolic compounds decreased by 25% and 52% in fig jam and fig nectar, respectively, when compared with the fig pulp (Tanwar *et al.*, 2014). Moreover, Rababah *et al.* (2011) reported that total phenolics ranged from 131.0 to 1224.2 mg GAE/kg and decreased significantly by 76.2% after fig jam processing, also after one month and three months, respectively, and no changes after three months was observed. In addition, the losses of polyphenols did not exceed 15% after drying (Vallejo *et al.*, 2012).

Table 4. Total polyphenolics, flavonoids, anthocyanins, and antioxidant activities of fresh, frozen, and processed figs (*Ficus carica* L.).

Sample 7	Total phenolic compounds (mg GAE/100 g)	Total flavonoids (mg QE/100 g)	TMA (mg cyd-3-	Antioxidant activity (mM TE/100 g)	
			glu/100 g)	DPPH	FRAP
Fresh fruits	$31.0\pm2.0^{\mathrm{a}}$	$11.4\pm1.2^{\rm b}$	$0.8\pm0.1^{\rm a}$	$21.3\pm1.2^{\rm b}$	$55.5\pm1.0^{\rm b}$
Frozen fruits	$14.0\pm0.9^{\circ}$	$3.6\pm0.9^{\circ}$	n.d.	n.d.	$27.8\pm4.3^{\rm a}$
Jam	$28.5\pm1.9^{\rm b}$	$6.6\pm1.0^{\rm a}$	$0.4\pm0.1^{\rm a}$	$13.2\pm2.1^{\rm a}$	$49.9 \pm 1.0^{\rm b}$

Data area means of triplicates $(n = 3) \pm$ standard deviation (SD). Means in the same column with different letters are significantly different at p < 0.05 by Tukey's multiple comparison test. n.d. = not detectable; TE = Trolox equivalent; GAE = gallic acid equivalent; QE = quercetin equivalent.

The total flavonoids in fresh figs were the highest (11.4 mg QE/100 g fw) while in frozen figs the lowest (3.6 mg QE/100 g fw) (Table 4). In fig jam, the total flavonoids were 6.6 mg QE/100 g fw, which showed 42% reduction after jam processing. The losses in the flavonoid content in the frozen figs were 68%. These values are higher than those reported by Tanwar *et al.* (2014), in which the total flavonoids were 4.4 and 0.4 mg QE/%, respectively.

It has been established that anthocyanins are mainly concentrated in the peels and constituted the main colouring compounds (Solomon et al., 2006). In the present work, the total anthocyanin was 0.78 mg cyd-3-glu/100 g fw in fresh figs. In fig jam, this decreased by 50%, while in frozen figs it was totally absent after four months. A similar decreasing trend in anthocyanin content in the fig jam (from 60.2%) to 79%) was reported by Rababah et al. (2011) and Tanwar et al. (2014). However, total monomeric anthocyanins remained in fig jam, and jam processing is a proper approach for processing fruits, better than frost treatment. Our data for the fresh figs were comparable to the report of Solomon et al. (2006) in which values for the yellow cultivars "Brunswick" was 0.7 mg of cyd-3-glu/100 g and lower than the values for fresh figs and fig jam, from 11.2 to 41.5 cyd-3-glu/kg (Rababah et al., 2011).

The antioxidant activity of the figs was evaluated by both DPPH and FRAP methods (Table 4). In general, the highest antioxidant activity was detected in fresh figs ($21.3 \pm 1.2 \text{ mM TE}/100 \text{ g fw}$ for DPPH assay and $55.5 \pm 1.0 \text{ mM TE}/100 \text{ g fw}$ for FRAP assay). The antioxidant potential of figs decreased significantly after freezing, while in jam, only a slight decrease was observed. Similar trends in the reduction of antioxidant potential during the five months of storage were reported for orange, apricot and fig jams (Rababah *et al.*, 2011). After four months, the antioxidant activity decreased significantly in jam by 38.9%.

The results showed positive linear correlations between the total antioxidant activities, total phenolic contents, total flavonoids, total monomeric anthocyanins, and total carotenoid content (Table 5). The coefficient of correlations (r) were 0.99 to 0.73 for FRAP and DPPH values, respectively. Therefore, it could be concluded that the total phenols and the total anthocyanins in fig samples provided the antioxidant activity. Similar observation for high correlation was demonstrated between either the total polyphenols or the total anthocyanins and antioxidant capacities (Solomon *et al.*, 2006). Based on the results obtained, the phenolic and flavonoid compounds have antioxidant, radical scavenging, and metal chelating properties. This however, is contrary to the results obtained by Bucić-Kojić *et al.* (2011) which demonstrated that the correlation coefficients (r) obtained between the antioxidant capacity of figs and corresponding TP (r = 0.191), TF (r = 0.451) and TPA (r = 0.343) were weak. Nevertheless, the findings obtained in the present work are in the good agreement with Mahmoudi *et al.* (2018) who demonstrated that antioxidant activity of fig peels and pulps were dependent on phenolic concentrations. The total carotenoids also brought about the antioxidant activity, but mainly by the radical scavenging activity.

Table 5. Correlation coefficient (*r*) between the total phenolic content, total flavonoids, total carotenoids, and the antioxidant activities (DPPH and FRAP)

the antioxidant activities (DITTT and FRAT).				
Correlation coefficient (r)	DPPH	FRAP		
Total phenolic content	0.97	0.99		
Total flavonoids	0.96	0.89		
TMA	0.99	0.95		
Total carotenoids	0.85	0.73		

Conclusion

The present work evaluated the polyphenol content and antioxidant potential of fresh figs and fig jams. Fresh figs demonstrated high levels of total phenolic compounds and sugar content. The continuous storage of figs destroyed monomeric anthocyanidins and lowered the antioxidant activity of the product. A homemade jam was demonstrated as the proper form for storage as it could keep the antioxidant potential of figs, thereby ensuring the quality of the stored product.

References

- Allegra, A., Alfeo, V., Gallotta, A. and Todaro, A. 2017. Nutraceutical content in 'Melanzana' and 'Dottato' fig fruit (*Ficus carica* L.). Acta Horticulturae 1173: 319-322.
- Association of Official Analytical Chemists (AOAC). 2007. Official methods of analysis of AOAC International. (18th ed). United States: AOAC.
- Benzie, I. F. and Strain, J. J. 1996. The ferric reducing ability of plasma (FRAP) as a measure of "antioxidant power": the FRAP assay. Analytical Biochemistry 239(1): 70-76.
- Bucić-Kojić, A., Planinić, M., Tomas, S., Jokić, S., Mujić, I., Bilić, M. and Velić, D. 2011. Effect of extraction conditions on the extractability of phenolic compounds from lyophilised fig fruits (*Ficus carica* L.). Polish Journal of Food and Nutritional Sciences 61(3): 195-199.

- Çaliskan, O. and Aytekin Polat, A. 2011. Phytochemical and antioxidant properties of selected fig (*Ficus carica* L.) accessions from the eastern Mediterranean region of Turkey. Scientia Horticulturae 128(4): 473-478.
- Debib, A., Tir-Touil, A., Mothana, R. A., Meddah, B. and Sonnet, P. 2014. Phenolic content, antioxidant and antimicrobial activities of two fruit varieties of Algerian *Ficus carica* L. Journal of Food Biochemistry 38(2): 207-215.
- Debib, A., Tir-Touil, M. A., Meddah, B., Hamaidi-Chergui, F., Menadi, S. and Alsayadi, M. S. 2018. Evaluation of antimicrobial and antioxidant activities of oily macerates of Algerian dried figs (*Ficus carica* L.). International Food Research Journal 25(1): 351-356.
- Dospatliev, L. 2018. Macro and micro elements, phospholipids and fatty acids in edible wild mushroom (*Cantharellus cibarius*) from Batak area (Rhodope mountains, Bulgaria). Comptes Rendus de l'Acade'mie Bulgare des Sciences 71(11): 1458-1465.
- DuBois, M., Gilles, K. A., Hamilton, J. K., Rebers, P. A. and Smith, F. 1956. Colorimetric method for determination of sugars and related substances. Analytical Chemistry 28(3): 350-356.
- Gani, G., Fatima, T., Qadri, T., Beenish, Jan, N. and Bashir, O. 2018. Phytochemistry and pharmacological activities of fig (*Ficus carica*): a review. International Journal of Research in Pharmacy and Pharmaceutical Sciences 3(2): 80-82.
- Gilani, A. H, Mehmood, M. H, Janbaz, K. H., Khan, A. U. and Saeed, S. A. 2008. Ethnopharmacological studies on antispasmodic and antiplatelet activities of *Ficus carica*. Journal of Ethnopharmacology 119(1): 1-5.
- Hossain, A. B. M. S., Salleh, A., Mekhled, M. A. and Al-Saif, A. M. 2010. Chlorophyll fluorescence intensity, photosynthetic yield and flowering in fig fruit trees as affected by phloem stress. Bulgarian Journal of Agricultural Science 16(5): 547-552.
- International Organization for Standardization (ISO). 1998. ISO 750:1998(E): fruit and vegetable products determination of titratable acidity. Geneva: ISO.
- Ivanov, I. G., Vrancheva, R. Z., Marchev, A. S., Petkova, N. T., Aneva, I. Y., Denev, P. P., ... and Pavlov, A. I. 2014. Antioxidant activities and phenolic compounds in Bulgarian *Fumaria* species. International Journal of Current Microbiology and Applied Sciences 3(2): 296-306.
- Jeong, M.-R., Kim, H.-Y. and Cha, J.-D. 2009. Antimicrobial activity of methanol extract from *Ficus carica* leaves against oral bacteria. Journal of Bacteriology and Virology 39(2): 97-102.
- Kivrak, I., Duru, M. E., Öztürk, M., Mercan, N., Harmandar, M. and Topçu, G. 2009. Antioxidant, anticholinesterase and antimicrobial constituents from the essential oil and ethanol extract of *Salvia potentillifolia*. Food Chemistry 116(2): 470-479.
- Lee, J., Durst, R. W. and Wrolstad R. E. 2005. Determination of total monomeric anthocyanin pigment content of fruit juices, beverages, natural colorants, and wines by the pH differential method: collaborative study. Journal of AOAC International 88(5): 1269-1278.

- Lever, M. 1972. A new reaction for colorimetric determination of carbohydrates. Analytical Biochemistry 47(1): 273-279.
- Lichtenthaler, H. K. and Wellburn, A. R. 1983. Determination of total carotenoids and chlorophylls a and b of leaf in different solvents. Biochemical Society Transactions 11(5): 591-592.
- Mahmoudi, S., Khali, M., Benkhaled, A., Boucetta, I., Dahmani, Y., Attallah, Z. and Belbraouet, S. 2018. Fresh figs (*Ficus carica* L.): pomological characteristics, nutritional value, and phytochemical properties. European Journal of Horticultural Science 83(2):104-113.
- Morton, J. F. 1987. Fig. In Morton, J. F. (ed). Fruits of Warm Climates. United States: Creative Resource Systems, Inc.
- Oliveira, A. P., Valentão, P., Pereira, J. A., Silva, B. M., Tavares, F. and Andrade, P. B. 2009. *Ficus carica* L.: metabolic and biological screening. Food and Chemical Toxicology 47(11): 2841-2846.
- Pande, G. and Akoh, C. C. 2010. Organic acids, antioxidant capacity, phenolic content, and lipid characterisation of Georgia-grown under-utilized fruit crops. Food Chemistry 120(4): 1067-1075.
- Petkova, N., Vrancheva, R., Denev, P., Ivanov, I. and Pavlov, A. 2014a. HPLC-RID method for determination of inulin and fructooligosacharides. Acta Scientifica Naturalis 1: 99-107.
- Petkova, N., Ivanov, I., Denev, P. and Pavlov, A. 2014b. Bioactive substance and free radical scavenging activities of flour from Jerusalem artichoke (*Helianthus tuberosus* L.) tubers - a comparative study. Turkish Journal of Agricultural and Natural Sciences (Special Issue 2): 1773-1778.
- Rababah, T. M., Al-Mahasneh, M. A., Kilani, I., Yang, W., Alhamad M. N., Ereifej, K. and Al-u'datt, M. 2011. Effect of jam processing and storage on total phenolics, antioxidant activity, and anthocyanins of different fruits. Journal of the Science of Food and Agriculture 91(6): 1096-1102.
- Salunkhe, D. K., Bolin, H. R. and Reddy, N. R. 1991. Freezing. In Salunkhe, D. K. (ed). Storage, Processing, and Nutritional Quality of Fruits and Vegetables (Volume 2), p. 41-47. United States: CRC Press.
- Silva, L. C. A. S., Harder, M. N. C., Arthur, P. B., Lima, R. B., Modolo, D. M. and Arthur, V. 2009. Physicalchemical characteristics of figs (*Ficus carica*) preready to submitted to ionizing radiation. In 2009 International Nuclear Atlantic Conference. Rio de Janeiro, Brazil.
- Slatnar, A., Klancar, U., Stampar, F. and Veberic, R. 2011. Effect of drying of figs (*Ficus carica* L.) on the contents of sugars, organic acids, and phenolic compounds. Journal of Agricultural and Food Chemistry 59(21): 11696-11702.
- Solomon, A., Golubowicz, S., Yablowicz, Z., Grossman, S., Bergman, M., Gottlieb, H. E., ... and Flaishman, M. A. 2006. Antioxidant activities and anthocyanin content of fresh fruits of common fig (*Ficus carica* L.). Journal of Agricultural and Food Chemistry 54(20): 7717-7723.

- Soni, N., Mehta, S., Satpathy, G. and Gupta, R. K. 2014. Estimation of nutritional, phytochemical, antioxidant and antibacterial activity of dried fig (*Ficus carica*). Journal of Pharmacognosy and Phytochemistry 3(2): 158-165.
- Sozzi, G. O, Abraján-Villaseñor, M. A., Trinchero, G. D. and Fraschina, A. A. 2005. Postharvest response of 'Brown Turkey' figs (*Ficus carica* L.) to the inhibition of ethylene perception. Journal of the Science of Food and Agriculture 85(14): 2503-2508.
- Stintzing, F. C., Nerbach, K. M., Mosshammer, M. R., Carle, R., Yi, W., Sellappan, S., ... and Felker, P. 2005. Color, betalain pattern, and antioxidant properties of cactus pear (*Opuntia* spp.) clones. Journal of Agricultural and Food Chemistry 53(2): 442-451.
- Tanwar, B., Andallu, B. and Modgil, R., 2014. Influence of processing on physicochemical, nutritional and phytochemical composition of *Ficus carica* L. (fig) products. Asian Journal of Dairy and Food Research 33(1): 37-43.
- Vallejo, F., Marín, J. G. and Tomás-Barberán, F. A. 2012. Phenolic compound content of fresh and dried figs (*Ficus carica* L.). Food Chemistry 130(3): 485-492.
- Veberic, R., Colaric, M. and Stampar, F. 2008. Phenolic acids and flavonoids of fig fruit (*Ficus carica* L.) in the northern Mediterranean region. Food Chemistry 106(1): 153-157.