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# Effect of pretreatments on mycotoxin profiles and levels in dried figs

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The aim of this explorative study was to investigate how effective drying preservation methods are in reducing mycotoxin content in figs. Dried autochthonous varieties of white and dark figs (Petrovača Bijela and Šaraguja, respectively) were analysed for mycotoxins using an LC-MS/MS "dilute and shoot" method capable of determining 295 fungal and bacterial secondary metabolites. Before drying in a cabinet dryer the figs were preserved with 0.5 % citric acid solution or 0.5 % ascorbic acid solution or 0.3 % L–cysteine solution or 0.2 % chestnut extract solution or 0.15 % Echinacea extract solution by immersion. We found nine metabolites: aflatoxin B1 ($AFB_1$), ochratoxin A, ochratoxin alpha, kojic acid, emodin, altenuene, alternariol methyl ether, brevianamide F, and tryptophol. The most efficient preserver was L-cysteine (15 % reduction), while ascorbic acid favoured mycotoxin production (158 % increase). However, all pretreatment solutions reduced $AFB_1$, which is a major fig contaminant.

KEY WORDS: *antimycotoxigenic effect; drying; LC-MS/MS; multi-mycotoxin analysis*

Mycotoxins are naturally occurring secondary metabolites of various moulds, some of which are the natural microbiota of figs and are unavoidable contaminants. However, through the implementation of good agricultural and manufacturing practices it is possible to minimise the contamination. According to Heperkan et al. (1), the dominant fungal flora in the Mediterranean dried figs are the *Aspergillus*, *Fusarium*, and *Penicillium* species and the related *Aspergillus* (2–5), *Fusarium* (6, 7), *Penicillium* (8, 9), and *Alternaria* mycotoxins (10). The last, *Alternaria alternate* has been reported in figs from Saudi Arabia and Montenegro. The Saudi study reported the presence of altenuene and alternariol (11), but the Montenegrin mycotoxin analysis found no mycotoxins (12). Other authors (13) reported high amounts of kojic acid (KA) in dried figs, as well as methoxysterigmatocystin, roquefortine A, fumagillin, fumigaclavine B, malformins (peptides), aspergillic acid, nigragillin, terrein, terrestric acid, and penicillic acid.

Mycotoxin production in figs begins on the tree and depends on a number of extrinsic and intrinsic factors, including stress and physical damage (1). Mycotoxin levels may increase during transportation, storage, and processing, if the conditions are favourable. However, contamination can be controlled with effective measures, starting with the orchards through transportation, storage, and processing like drying in a solar drier (1, 3). Drying is one of the oldest preservation methods that can also reduce and/or control mycotoxin contamination. Traditional sun drying is undesirable because figs are prone to contamination with fungal spores from the soil (14). An alternative to traditional drying are mechanical air-drying systems which provide controlled environment (15).

Drying is often combined with pretreatment with water and chemical solutions (14, 16, 17) to counter undesired effects and improve the quality of dried products. L-cysteine has proved its worth in preventing the oxidation (and related browning) of apples and potatoes (18), and other natural antioxidants, such as ascorbic acid, Echinacea (19), citric acid (16), and phenolic compounds extracted from chestnuts (20, 21) have also been successful antioxidants. Since oxidative stress is one of the prerequisites for mycotoxin biosynthesis (22), antioxidants such as these also exert antimycotoxigenic effects (23). However, knowledge about these antimycotoxigenic effects is still scarce (24). The aim of this study was therefore to determine the efficiency of pretreatment with the most common antioxidants in reducing mycotoxin content in dried autochthonous Croatian fig varieties.

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## MATERIALS AND METHODS

### *Chemicals and reagents*

HPLC-grade methanol and acetonitrile as well as MS-grade ammonium acetate and glacial acetic acid (p.a.) were purchased from Sigma-Aldrich (Vienna, Austria). Ultraclean water was obtained by purification of reverse osmosis water with a Purelab Ultra system (ELGA LabWater, Celle, Germany). Ascorbic acid, citric acid, and L-cystein were purchased from Sigma-Aldrich Chemie GmbH (Taufkirchen, Germany). The chestnut extract was produced from whole local chestnut variety Lovranski marun, and the Echinacea extract was produced from the flower of *Echinacea purpurea.* Both were obtained by adding 250 mL of 50 % ethanol to 50 g of the sample (sample-to-solvent ratio was 1:5) and applying ultrasonication at 50 Hz and 125 W for 30 min. The liquid extract was obtained by filtration through Whatman® Grade 4 filter paper (Whatman, Maidstone, United Kingdom) and removal of solvent by evaporation under vacuum at 40 °C.

### *Sampling*

Figs were obtained from a pilot orchard in Kazela, Istria, Croatia, harvested in August 2015. We used two most common autochthonous varieties of fresh white and dark figs, namely Petrovača Bijela and Šaraguja (Figure 1, Table 1). Two random batches (5 kg) per treatment were used and mixed before mycotoxin measurements. Control samples were pretreated with distilled water.

### *Drying*

Untreated (control) and pretreated whole figs were dried on 17 trays with 2.5 kg of figs on each in a cabinet dryer (2x4 kW heater power, 150 kg drying capacity, Rasadnik Skink Rovinj, Croatia). The dryer was equipped with temperature and airflow velocity controllers. Drying temperature was 60-70 °C.

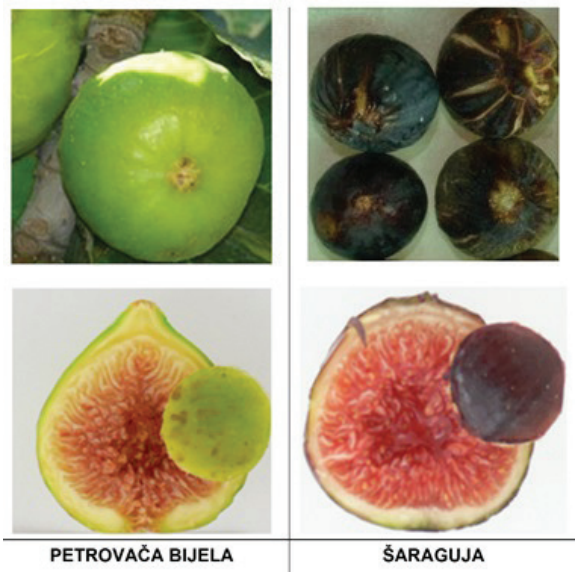


**Figure 1** *Autochthonous white (left) and dark (right) fig varieties in Istria used for testing the efficiency of pretreatment in reducing mycotoxin levels*

Before drying, the figs intended for pretreatment were immersed in 0.5 % citric acid solution or 0.5 % ascorbic acid solution or 0.3 % L-cysteine solution or 0.2 % chestnut extract solution or 0.15 % Echinacea extract solution for 5 min.

The measurements started as soon as the fig samples were placed onto the trays in the cabinet dryer. Sample weight loss was measured with a digital scale every 10 min, and drying stopped when the moisture content dropped to 25 % of the fresh fruit content. The samples were then stored in paper bags at room temperature for 30 days before mycotoxin analysis.

### *Mycotoxin analysis*

Standards for mycotoxin analysis were prepared as described elsewhere (24). Briefly, 34 stock solutions of analyte standards were mixed into one working solution, which was used for spiking and calibration.

We used an LC-MS/MS "dilute-and-shoot" method capable of determining 295 fungal and bacterial secondary metabolites in one run as described elsewhere (24).

Frozen, dried, and cut figs were transported to Austria, ground there with an Osterizer blender (Sunbeam Oster Household Products, Fort Lauderdale, FL, USA), and 5 g samples weighed and put in 50 mL tubes. Then 20 mL of extraction solvent (acetonitrile/water/acetic acid 79:20:1, v/v/v) was added and the content thoroughly mixed by hand. Followed a 90-minute extraction at room temperature with a GFL 3017 rotary shaker set at 180 rpm (GFL, Burgwedel, Germany). After extraction, the samples were allowed to precipitate, and 500 µL of the extract was diluted with 500 µL of dilution solvent (acetonitrile/water/acetic acid 20:79:1, v/v/v) in 1.5 mL vials. The vials were mixed by vortexing for 10 s, and 5 µL of the diluted sample extract was directly injected into a liquid chromatography – mass spectrometry (LC–MS/MS) system. The LC part consisted of an Agilent 1290 binary UHPLC pump (Agilent Technologies, Waldbronn, Germany) coupled with a Sciex QTRAP® 5500 triple quadruple MS/MS detector (Sciex, Foster City, CA, USA). The chromatographic conditions were strictly followed as described elsewhere (24). The sample with the lowest concentration of mycotoxins was used for the recovery experiment by spiking 250 mg of the sample with 100 µL of the working standard solution. Mycotoxin levels used for recovery were as follows: aflatoxin B1 ($AFB_1$) – 4.57 µg $kg^{-1}$; OTA – 26.1 µg $kg^{-1}$; ochratoxin α (OTα) – 3.30 µg $kg^{-1}$; alternariolmethylether (AME) – 14.4 µg $kg^{-1}$; emodin (EMO) – 7.21 µg $kg^{-1}$; altenuen (ATN) – 7.21 µg $kg^{-1}$; KA – 206 µg $kg^{-1}$; tryptophol (TryOH) – 26.1 µg $kg^{-1}$; and brevianamide F (BREF) – 5.23 µg $kg^{-1}$. The exact mycotoxin levels in the mastermix

**Table 1** *Characteristics of the investigated fig varieties*

| | Petrovača Bijela | Šaraguja |
|---|---|---|
| Fruit volume ($cm^3$) | 94 | 45 |
| Fruit weight (g) | 82 | 51 |
| Fruit width (mm) | 55 | 46 |
| Fruit length (mm) | 57 | 45 |
| Fruit skin thickness (mm) | 11 | 4 |
| Fruit skin firmness | Medium | Firm |
| Fruit skin cracks | Absent | Minute crack |
| Resistance to cracks | Resistant | Intermediate |
| Fruit skin ground colour | Green | Green |
| Overcolour – regular bands | Yellow, green | Purple |
| Pulp internal colour | Pink, amber | Red |
| Dry weight (%) | 25.5 | 26.5 |

*All values are the means of three measurements*

have been described elsewhere (24). The sample was left at room temperature in the dark overnight to allow for the solvent to evaporate and to ensure complete sample-spike equilibration of the mycotoxins in the matrix.

All results were corrected for the obtained recovery. Measurements were performed as described elsewhere (24) and quantified with the Analyst® 1.6.2 and Multiquant® 3.0.2. software (Sciex). The limits of detection (LOD) and quantification (LOQ) were determined as the signal-to-noise ratio of 3 and 10 in the tested matrices, respectively, while the linearity of the calibration curve was calculated automatically with the Multiquant® 3.0.2. software.

The accuracy of the method had been verified through regular participation in proficiency testing schemes organised by the Bureau Interprofessionnel d'Etudes Analytiques - BIPEA (Genneviliers, France). All six results submitted for fig samples exhibited a z-score between -2 and 2.

*Statistical analysis*

The data were checked for normality of distribution with the Shapiro-Wilk test and compared with nonparametric methods (Mann-Whitney U test for the comparison between two fig varieties and Kruskal-Wallis ANOVA for the comparison of pretreatments). For the analysis we used Statistica® 12.0 (Dell Inc., Round Rock, TX, USA) and Microsoft Excel® 2016 (Microsoft, Redmond, WA, USA). Differences <0.05 were considered significant.

## RESULTS AND DISCUSSION

Of the 295 bacterial and fungal metabolites detectable by the method, we found only nine mycotoxins above LOD (Table 2). Of these, TryOH, BREF, EMO, AME, ATN, and OTα were evidenced for the first time in figs.

Table 3 shows the effects of pretreatment compared to control samples. L-cysteine pretreatment achieved the best overall results, while all pretreatments proved to be effective against the most problematic mycotoxin in figs, $AFB_1$, with reductions ranging from 25 to 93 %.

$AFB_1$ levels were below the LOD in half of the samples. In the rest of them they were much lower than the EU limit of 6 μg $kg^{-1}$ (14). The dark Šaraguja variety had higher median and maximum values than the white variety, but the difference was not statistically significant (Table 2). The best reduction (to below LOD) was achieved with ascorbic acid (both levels were below LOD).

OTA was detected in only one sample (at level below the LOQ), unlike its main major metabolite OTα, whose levels were higher than those of $AFB_1$ in all but the white fig sample pretreated with citric acid. This finding indicates that OTA had been present in the figs at some point, but the phenylalanine part was cleaved off by other microflora in the fig (25). The dark Šaraguja variety had significantly higher OTα levels than the white Petrovača variety ($p=0.005$).

$AFB_1$ and OTA have often been reported in dried figs (1, 3, 23), and particularly high contamination was reported in dried figs from Turkey and Egypt (1). In contrast, all of our samples from Croatia were below MLs (26). This may be related to harsher climate that does not favour *Aspergillus* spp. growth. However, if the global temperature will continue to increase, aflatoxigenic Aspergilli will inevitably take a stronger hold in southeast Europe, including Croatia (27). OTA, on the other hand, is also produced by the *Penicillium* spp. that grow in colder climates, which calls for regular OTA monitoring in figs, regardless of their region of origin.

Even though this is the first evidence of AME in dried figs, its concentrations were very low or below LOD. The acute toxicity of AME is low, and little is known about its mutagenic and genotoxic properties (10). AME, ATN, alternariol (AOH), tentoxin (TTX), and tenuazonic acid (TeA) are produced by the *Alternaria* spp., whose occurrence is rare in dried figs (10). Again, the dark variety

**Table 2** *Mass fractions (μg kg$^{-1}$) of mycotoxins above LOD*

| Sample | Sort | AFB1 | OTA | OTα | AME | ATN | EMO | KA | TryOH | BREF |
|---|---|---|---|---|---|---|---|---|---|---|
| Control | white | 0.41 | <LOD | 2.00* | 0.30 | 12.0 | 0.64 | 449 | 21.2* | 8.02 |
| Control | dark | 0.65 | <LOD | 3.81 | 0.10* | 16.7 | <LOD | 5.575 | <LOD | 4.34 |
| L-cysteine | white | 0.12* | <LOD | 0.92* | <LOD | 9.20 | <LOD | 1.191 | <LOD | 5.27 |
| L-cysteine | dark | <LOD | <LOD | 5.71 | <LOD | 10.9 | <LOD | 4.636 | 17.4* | 5.65 |
| Ascorbic acid | white | <LOD | 0.5* | <LOD | 0.36 | 16.5 | 0.24* | 1.388 | 20.8* | 7.71 |
| Ascorbic acid | dark | <LOD | <LOD | 4.53 | 0.34 | 21.9 | 1.88 | 5.257 | 27.0* | 10.9 |
| Citric acid | white | 0.32 | <LOD | <LOD | <LOD | 30.0 | 0.26* | 1.148 | 30.7* | 8.38 |
| Citric acid | dark | <LOD | <LOD | 6.81 | <LOD | 30.8 | <LOD | 4.853 | <LOD | 5.09 |
| Chestnut extract | white | <LOD | <LOD | 2.54* | 0.11* | 12.1 | 0.71 | 1.414 | 19.0* | 7.99 |
| Chestnut extract | dark | 0.33 | <LOD | 6.58 | 0.23* | 24.8 | <LOD | 8.594 | 99.1 | 7.21 |
| Echinacea extract | white | <LOD | <LOD | 2.87 | <LOD | 6.80 | <LOD | 850 | 20.2* | 8.56 |
| Echinacea extract | dark | 0.44 | <LOD | 3.59 | 0.23* | 14.7 | <LOD | 5.416 | <LOD | 5.71 |
| Recovery | | 91 % | 96 % | 71 % | 89 % | 111 % | 95 % | 89 % | 79 % | 76 % |
| LOD | | 0.08 | 0.40 | 0.80 | 0.08 | 0.80 | 0.16 | 5.00 | 16.0 | 0.40 |
| LOQ | | 0.30 | 1.30 | 2.60 | 0.30 | 2.60 | 0.50 | 16.5 | 52.8 | 1.30 |
| $r^2$ | | 0.998 | 0.999 | 0.998 | 0.999 | 0.994 | 0.999 | 0.997 | 0.999 | 0.999 |

*All values are the means of three measurements. AFB1=aflatoxin B1; OTA=ochratoxin A; OTα=ochratoxin alpha; AME=alternariol methyl ether; ATN=altenuene; EMO=emodin; KA=kojic acid; TryOH=tryptophol; BREF=brevianamide F; LOD=limit of detection; LOQ=limit of quantification. *values below LOQ*

had higher concentrations of AME and ATN, but the difference was not statistically significant. L-cysteine and citric acid were the most effective against AME, and ATN concentrations were reduced by L-cysteine and Echinacea extract.

Kojic acid in our study was found in all samples above LOD, and its levels in pretreated figs were the highest compared to other mycotoxins. However, many studies claim unlikely or no adverse effects of KA, as it acts as an antioxidant and is widely used in food and cosmetic industry (5).

EMO was detected in several samples above LOD, most notably in the control white fig variety and in the dark variety pretreated with ascorbic acid. EMO was first confirmed in chestnuts (28, 29) and afterwards in many other matrices (30, 31) but never in dried figs. This anthraquinone polyphenol can be produced by various species (plant and fungal), but in most food it occurs as the *Aspergillus* metabolite (30). From the toxicological point of view, EMO has been documented in a wide range of actions, from cytotoxic to laxative and antitumour (32). The Echinacea extract lowered its levels below LOD in both varieties, but there was no significant difference between the treatments.

To the best of our knowledge, this is the first study to report TryOH in dried figs, but concerning the producing microbiota, this finding is not a surprise. TryOH is not strictly classified as a mycotoxin, as it has important functions in filamentous fungi and is one of the most prevalent fungal metabolites (30, 31, 33). In one study (34) it was also determined as one of the fermentation products in bread.

BREF was determined in all samples with concentrations above LOQ. This *Aspergillus* metabolite is quite common in a number of matrices (33), but it has not been reported in dried figs until now. Pretreatment was more effective in the white variety, whereas in the dark variety it only increased BREF levels. However, little is known about its toxicity.

## CONCLUSIONS

Our results suggest that L-cysteine is a potent antimycotoxigenic agent for fig treatment before drying, but the winner against aflatoxins is citric acid. L-cysteine potently reduced the *Alternaria*, EMO, and KA in the dark variety, while the Echinacea extract was the best against EMO. The dark fig variety Šaraguja was more prone to mycotoxin contamination, most likely because of thinner skin with many more cracks in it, which facilitates fungal infection.

Before these pretreatments are applied on the industrial scale, their efficiency should be tested on intentionally infected figs (by different mycotoxin-producing fungi) and on a larger scale to obtain relevant practical findings. Our study has just given a glimpse of potential issues with dry fig mycotoxin contamination and treatment possibilities.

**Table 3** *Increase or reduction of mycotoxin levels by fig variety and pretreatment agent*

| Sample | Fig variety | $AFB_1$ | OTA | OTα | AME | ATN | EMO | KA | TryOH | BREF |
|---|---|---|---|---|---|---|---|---|---|---|
| L-cysteine | white | 25 | 100 | 45 | 13 | 77 | 13 | 265 | 38 | 65 |
| L-cysteine | dark | 7 | 100 | 150 | 40 | 65 | 100 | 83 | 218 | 130 |
| Ascorbic acid | white | 10 | 250 | 20 | 100 | 138 | 33 | 309 | 98 | 96 |
| Ascorbic acid | dark | 7 | 100 | 118 | 300 | 131 | 2250 | 94 | 338 | 253 |
| Citric acid | white | 75 | 100 | 20 | 13 | 250 | 33 | 256 | 145 | 104 |
| Citric acid | dark | 7 | 100 | 179 | 40 | 184 | 100 | 87 | 100 | 116 |
| Chestnut extract | white | 10 | 100 | 125 | 33 | 101 | 117 | 315 | 90 | 99 |
| Chestnut extract | dark | 50 | 100 | 171 | 200 | 149 | 100 | 154 | 1239 | 167 |
| Echinacea extract | white | 10 | 100 | 140 | 13 | 57 | 13 | 189 | 95 | 106 |
| Echinacea extract | dark | 67 | 100 | 92 | 200 | 88 | 100 | 97 | 100 | 133 |

*The figures show the percentage of control levels, which are 100 %. Figures below 100 % show reduction and above 100 % increase. AFB1=aflatoxin B1; OTA=ochratoxin A; OTα=ochratoxin alpha; AME=alternariol methyl ether; ATN=altenuene; EMO=emodin; KA=kojic acid; TryOH=tryptophol; BREF=brevianamide F; all results are mean values of three measurements*

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**Utjecaj predtretmana sušenja na pojavnost i količinu mikotoksina u suhim smokvama**

Za određivanje pojavnosti mikotoksina u osušenim autohtonim sortama bijelih i tamnih smokava petrovača bijela i šaraguja korištena je metoda LC-MS / MS "razrijedi i mjeri", kojom je moguće analizirati 295 gljivičnih i bakterijskih sekundarnih metabolita. Smokve su sušene u pilot-pogonu uz korištenje različitih predtretmana za održavanje sušenog voća: uranjanje u 0,5 %-tnu otopinu limunske kiseline, 0,5 %-tnu otopinu askorbinske kiseline, 0,3 %-tnu otopinu L-cisteina, 0,2 %-tnu otopinu ekstrakta kestena i 0,15 %-tnu otopinu ekstrakta Echinaceae. Ovim se istraživanjem otkrilo devet različitih metabolita u koncentracijama iznad njihove granice detekcije: aflatoksin B1, okratoksin A, okratoxin α, kojična kiselina, emodin, altenuen, alternariolmetileter, brevianamid F i triptofol. Sveukupno, najbolji antimikotoksigenski učinak postignut je predtretmanom s L-cisteinom (prosječno smanjenje od 15 %), a tretman s askorbinskom kiselinom pokazao je najveći učinak na indukciju mikotoksina (povećanje od 158 %). Svi predtretmani smanjili su koncentracije aflatoksina, glavnog kontaminanta smokava.

KLJUČNE RIJEČI: *antimikotoksigeni učinak; LC-MS/MS; multimikotoksinska analiza; sušenje*