UDC 614.31: 543.51: 543.544.5.068.7: 543.64
DOI: 10.21668/health.risk/2024.4.06.eng

Read online

Research article

MYCOTOXINS CONTAMINATION OF GRAPE WINES MARKETED IN THE RUSSIAN FEDERATION

I.B. Sedova[1], Z.A. Chalyy[1], V.A. Tutelyan[1,2]

[1]Federal Research Centre of Nutrition and Biotechnology, 2/14 Ust'inskii pr., Moscow, 109240, Russian Federation
[2]Sechenov University, 8/2 Trubetskaya St., Moscow, 119991, Russian Federation

Grape wine is one of the most popular and widespread alcoholic beverages around the world. Contamination of grapes used in its preparation by mold fungi can lead to accumulation of mycotoxins that are harmful to human health. Due to their high toxicity and potential health risks, the aim of this study was to investigate the contamination of grape wines sold in Russia with a wide range of toxins and to assess the population health risk associated with their consumption.

The concentration of 27 mycotoxins in 36 samples of grape wines was determined by high-performance liquid chromatography coupled to tandem mass-spectrometric detection. The list of mycotoxins included regulated ones, their derivatives and structural analogs, as well as emergent mycotoxins (Alternaria toxins, citrinin, sterigmatocistin, cyclopiazonic and mycophenolic acids, moniliformin, enniatins, beauvericin).

The resulting data indicated contamination of grape wines consumed in Russia with mycotoxins. Thirty-one percent of the samples contained toxins at relatively low levels of contamination. Aflatoxin G2, altenuene, mycophenolic acid and sterigmatocystin were detected more often than other toxins; in some cases, zearalenone, tenuazonic acid and ochratoxin A. Red wine samples were the most contaminated with mycotoxins. Fumonisin B1, zearalenone, sterigmatocystin, altenuene and ochratoxin A were found only in imported wine samples. Several toxins were found simultaneously in four tested wine samples.

Despite a relatively low level of mycotoxin contamination of wine marketed in the Russian Federation, there is still a potential hazard of their chronic ingestion by humans. The calculated average intake of aflatoxins may reach 28 % of the reference value and the maximum intake can even exceed it. The calculated exposure of other mycotoxins detected in wine samples indicates an insignificant contribution of grape wine (up to 2.8 % of the reference value) to their intake by the Russian population.

Keywords: *mycotoxins, emergent mycotoxins, contamination, grape wine, ochratoxin A, aflatoxins, HPLC-MS/MS.*

Grape is one of the most popular and consumed berries in Russia. The fungal diseases caused by *Plasmopara viticola, Botrytis cinerea, Greeneria uvicola, Glomerella cingulata*, as well as *Alternaria, Aspergillus, Cladosporium, Penicillium, Rhizopus, Colletotrichum* and *Phomopsis* species reduce crop yield and grape quality [1]. Among the fungi that cause grape diseases are filamentous fungi of *Alternaria, Aspergillus* and *Penicillium*, capable of producing toxic secondary metabolites – mycotoxins (MT). There are reports on the contamination of alternariol (AOH), aflatoxins (AFL), alternariol monomethyl ether (AME), tenuazonic acid (TeA), fumonisins B1 and B2 (FB1 and FB2), patulin (PAT), penicillic acid (PA), cyclopiazonic acid (CPA), zearalenone (ZEN), citrinin (CIT) and ochratoxin A (OTA) in grape [2–5]. Their toxic effects are associated with nephrotoxicity, hepatotoxicity, cytotoxicity, teratogenicity and immunotoxicity [6]. Due to their high toxicity and possible health risk, numerous studies are being conducted for mycotoxins in raw materials and foodstuffs

OTA is the most studied of the MTs in grape products and may represent a real problem for the industry. The Codex Alimentarius

Irina B. Sedova – Candidate of Biological Sciences, Senior Researcher at the Laboratory of Enzymology of Nutrition (e-mail: isedova@ion.ru; tel.: +7 (495) 698-53-65; ORCID: https://orcid.org/0000-0002-6011-4515).

Zakhar A. Chalyy – Junior Researcher at the Laboratory of Enzymology of Nutrition (e-mail: brew@ion.ru; tel.: +7 (495) 698-53-65; ORCID: https://orcid.org/0000-0002-9371-8163).

Victor A. Tutelyan – Academician of the Russian Academy of Sciences, Professor, Academic Advisor (e-mail: tutelyan@ion.ru; tel.: +7 (495) 698-53-65; ORCID: https://orcid.org/0000-0002-4164-8992).

Commission estimates that wine is the second foodstuff, following cereals, with the highest contribution to OTA daily intake (up to 15 %) [7]. OTA contamination can occur during any stage of the winemaking process: from the colonization of mycotoxigenic fungi in grapes to the final steps in the wine packaging process. However, the main contamination of the finished product comes from the carryover–of mycotoxins from grapes [8]. In addition, the vinification process has a considerable influence on OTA content as higher concentrations are reported in red wines than in rose and white wines [9].

This toxin has been reported in wine over the last 20 years in various countries across the globe: in Russia (up to 0.64 μg/l) [10], USA (up to 8.6 μg/l) [11], Europe: Greece (up to 0.212 and 2.52 μg/l) [12, 13], Denmark (up to 0.6 μg/l) [14], Spain (up to 0.104 and 0.142 μg/l) [12, 15], Italy (up to 0.286, 1.56, 2.28 μg/l) [12, 16, 17], Portugal (up to 1.18 μg/l) [18], Serbia (up to 0.134 μg/l) [19], France (up to 0.088 μg/l) [12], Croatia (up to 0.061 and 0.24 μg/l) [2, 12], Argentina (up to 0.98 μg/l) [20], Brazil (up to 0.62 μg/l) [21], Israel (up to 0.065 μg/l) [12], China (up to 0.98 and 1.27 μg/l) [3, 22], Thailand (up to 1.72 μg/l) [23], Turkey (up to 0.101 μg/l [12], and South Africa (up to 0.455 μg/l) [12].

Currently, there are no national regulations in the Russian Federation on the content of MT in grape wine. At the same time, some countries have set the limits of OTA content in grape wine: in the EU countries[1] and South Korea – at the level of 2 ng/g and in Brazil – 10 ng/g [24].

To objectively assess contamination of grape wine with poorly studied toxins of micromycetes, 12 emergent MT species (EMTs) were included in the analysis. They can be produced both by *Fusarium* sp., *Aspergillus* sp., *Alternaria* sp., *Penicillium* sp. and by fungi from other genera.

In connection with the above, **the aim of this study** was to investigate frequency and levels of the contamination of grape wines sold in Russia with a wide range of toxins. Contents of 27 MTs were determined in the analyzed products including MTs regulated in foodstuffs of plant origin, their derivatives, structural analogs and EMTs.

Materials and methods. Grape wines were bought in retail outlets in Moscow and the Moscow region in 2020. Overall, we analyzed 36 samples that included 21 red wine samples, 12 white wine samples and 3 rose wine samples. The wine producers were from Russia, including the republics of Crimea and Dagestan, as well as Georgia, Chile, Italy, France, New Zealand, Argentina, Spain, Austria and Israel.

Sampling of food raw materials and food products for analysis was carried out in accordance with the GOST 33303-2015 Food Products. Sampling Methods for Identification of Mycotoxins[2].

Contents of the following MTs were identified in all analyzed samples: MTs produced by *Penicillium* and *Aspergillus* genera including OTA, AFL B1, B2, G1, G2, PAT, STC, CIT, PA, MPA by *Alternaria* genus including AOH, AME, ALT, TEN and TeA; by *Fusarium* genus including DON, FB1, FB2; such toxins as T-2, HT-2, ZEN, β-zearalenol (β-ZEL), nivalenol (NIV), moniliformin (MO), beauvericin (BEA), enniatins A and B (ENN A и B). Among the analyzed MTs, CIT, STC, MPA, MO, ENN A, ENN B, BEA, TEN, TeA, AOH, AME and ALT were emergent MTs (EMTs).

Preparation of wine samples. The study relied on using the methodology described in the [14] with minimal modifications. Five ± 0.02 grams of wine were taken from a representative sample with its volume being not less than 100 cm^3 and put into a 50 cm^3 centrifuge tube. Next, the sample was added with 5 grams of water and with 10 cm^3 of acetonitrile acidified with 1 %-solution of acetic acid and then mixed for 1 minute at 300 rpm. Next, the sam-

[1] European Commission. Commission Regulation (EU) 2023/915 of 25 April 2023 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006. *Off. J. Eur. Union*, 2023, no. L 119, pp. 103–157.

[2] GOST 33303-2015. Foodstuffs. Sampling methods for determination of mycotoxins: Interstate Standard. Moscow, Standartinform, 2016 (in Russian).

ple was added with 3 grams of anhydrous $MgSO_4$ and immediately mixed for 1 minute; 3 cm^3 of the acetonitrile extract aliquot (the upper layer) were put into another centrifuge tube, which contained 450 mg of anhydrous $MgSO_4$, and then mixed thoroughly mechanically for 1 minute. The extract was centrifuged at 4000 rpm under 10 °C for 4 minutes. The extract aliquot with its volume of 0.5 cm^3 was added with 0.5 cm^3 of methanol. The resulting solution was used for quantification. The samples were prepared for analysis in two replications.

Analysis of grape processing products for presence of MTs was carried out using the Waters Acquity H-plus HPLC system coupled with triple quadrupole mass spectrometric detector with the source heater (XEVO TQ-XS) controlled by the MassLynx V4.2 software (Waters Corporation, USA). The temperature in the autosampler was 4 °C. Analytes were separated on a column filled with silica gel with added octadecylsilane groups (Zorbax SB-C18, 150 × 4.6 mm, 3.5 μm, pore size is 80Å, Agilent). The column temperature was 30 °C. The eluent flow rate was 0.5 cm^3/min. The injection volume was 10 mm^3.

The gradient elution was applied to separate toxins: the phase A – water : methanol (95 : 5 vol. %); the phase B – methanol : water (95 : 5 vol. %); both phases were modified with 10 mM ammonia acetate. The gradient scheme under negative polarity was as follows: from 0 to 1 min at 0 % B; from 1 to 7 min, linear growth from 0 to 70 % B; from 7 to 15 min, linear growth from 70 to 100 % B; from 15 to 19 min, constantly at 100 % B; from 19 to 19.5 min, linear decline to 0 % B; from 19.5 to 22 min, the column equilibration at 0 % B. The gradient scheme under positive polarity was as follows: from 0 to 7 min, linear growth from 10 to 75 % B; from 7 to 17 min, linear growth from 75 to 100 % B; from 17 to 19 min, constantly at 100 % B; from 19 to 19.5 min, linear decline from 100 to 10 % B; from 19.5 to 24 min, the column equilibration at 10 % B. MS/MS detection was conducted in the positive electrospray mode and dynamic MRM mode under positive and negative ionization.

The source parameters were as follows: ion trap with electrospray ionization; capillary voltage was 0.5 kV; the cone voltage, 3 V; the source temperature, 500 ºC; the desolvation temperature, 500 ºC; the gas flow in the cone, 150 dm^3/h; the desolvation gas flow, 1000 dm^3/h; the gas flow for collision, 0.15 cm^3/min; the nebulizer pressure, 7 bars.

The standards solutions of MT mixes were made of dried standards (Sigma-Aldrich; Fermentek, Jerusalem, Israel). Stock solutions of AFL, STC, CIT, PA, PAT, group A and B trichothecenes, ZEN and its analogs, and OTA were prepared in acetonitrile; stock solutions of *Alternaria*, ENN A, ENN B, BEA, MPA, MO toxins, in methanol; FB1, FB2, in 50 / 50 (% vol.) acetonitrile / water mix, with a concentration of 100 or 500 μg/cm^3. Multistandards and calibration solutions were made from standard solutions. All solutions were stored at -18 °C.

Statistical data analysis. Data on MTs contents in the examined samples obtained by statistical data analysis were given as M - arithmetic mean of total samples and M_{cont} as arithmetic mean of all contaminated samples respectively; and 95 % percentile. When calculating the average contamination level in total samples, the MT content in samples with contamination below the limit of quantification for this method was taken as equal to zero.

To quantify MT content in grape wine, external calibrations on a ‘clean’ matrix were applied. The limits of quantification (LOQ) for MTs calculated per 10-σ criteria for the samples (in μg/kg) amounted to 0.25 for STC; OTA, 0.30; AFL B1, T-2, MPA, TEN, 0.40; AFL B2, G1, G2, HT-2, AME, ALT, 1.00; BEA, ENN A, ENN B, 1.50; AOH, PA, 2.00; CIT, 3.00; FB1, ZEN, 4.0; PAT, β-ZEL, 6.00, DON, NIV, 20.00; FB2, 25.0; MO, 30.00; TeA, 40.00.

Results and discussion. The study of MT contamination of grape wines showed the presence of 10 of 27 analyzed MTs in 11 (31 %) of the 36 samples studied (Table 1). Overall, relatively low frequency and levels of toxin’s contamination of these beverages were found

Table 1

Contents of mycotoxins in grape wine samples (n = 36)

Mycotoxin	The number of contaminated samples	Toxins content in contaminated samples, μg/kg		Toxins content in total samples, μg/kg	
		range	M_{cont}	M	90 %
AFL G2	4 (11 %)	1.01–3.05	1.74	0.19	0.50
ALT	3 (8 %)	1.35–2.98	1.90	0.16	0
MPA	3 (8 %)	3.54–5.51	4.37	0.36	0
TEN	2 (6 %)	0.65–0.84	1.74	0.04	0
FB1	2 (6 %)	26.40–27.04	26.72	0.19	0
STC	2 (6 %)	0.37–1.70	1.04	0.06	0
HT-2	1 (3 %)	1.18	1.18	0.03	0
ZEN	1 (3 %)	5.08	5.08	0.14	0
TeA	1 (3 %)	146.5	146.5	4.07	0
OTA	1 (3 %)	0.39	0.39	0.01	0

Four samples were contaminated with AFL G2 in amounts ranging from 1.01 to 3.05 μg/kg. ALT and MPA were detected a bit less frequently, in 8 % of the samples. TEN, FB1 and STC were found in two out of 36 analyzed samples in contents equal to 0.84, 27 and 1.70 μg/kg respectively. Such toxins as HT-2 (1.18 μg/kg), OTA (0.39 μg/kg), ZEN (5.08 μg/kg) and TeA (146.5 μg/kg) were found in single samples. OTA content did not exceed the values of hygienic regulations for these products.

The study [16] reported the presence of AFL in lower concentrations and OTA in grape wine samples. In this study, 10 to 33 % of the samples were contaminated with AFL B1, B2, G1, G2 in amount as low as 0.035, 0.016, 0.068 and 0.033 μg/m^3 respectively; approximately 97 % contained OTA, its levels varying between 0.021 and 1.56 μg/m^3.

Alternaria toxins were also reported in grape wine samples in [14, 17, 23, 25]. TeA and TEN were found in the study [25]; however, frequency of these MTs contamination was significantly higher than in our study. AOH and AME were found in more than a half of the analyzed samples as reported in [17, 25], and AME was found in one case [23]; however, ALT was not found in the wine samples analyzed in their studies.

There are data on the occurrence of fumonizins [23], HT-2 [17], ZEN and MPA [3, 14] in grape wine.

The analyzed wine samples were divided into three groups depending on the classification by wine color: 21 red wine samples, 12 white wine samples, and 3 rose wine samples. MTs were not found in any of three rose wine samples. Ten toxins were found in the analyzed red wine samples (Figure 1), whereas the white one samples contained only four MTs: FB1, MPA, STC и ZEN.

AFL G2 was the most frequent contaminant in the red wine samples and identified in 19 % of the cases. FB1 and AFL were found in 14 % of the cases; TEN and MPA, 10 %. The other toxins were found in single samples. Attention should be paid to the fact that OTA was found only in a red wine sample. Red wine production can involve a stage when a wine is kept for several days under high temperatures in aerobic conditions without filtration. This, in its turn, can contribute to toxin accumulation in it; in white wine production, after pressing the grape juice is filtered and fermented directly [10].

A range of MTs found in white wine was less substantial as compared with red wine. White wine was less frequently contaminated with FB1 than red wine, 8 % against 14 % respectively. In addition, MPA, STC and ZEN were found in the white wine samples with their frequency ranging between 4 and 10 %.

TeA and HT-2 were found only in Russian wines (Figure 2). MPA, AFL G2 and TEN

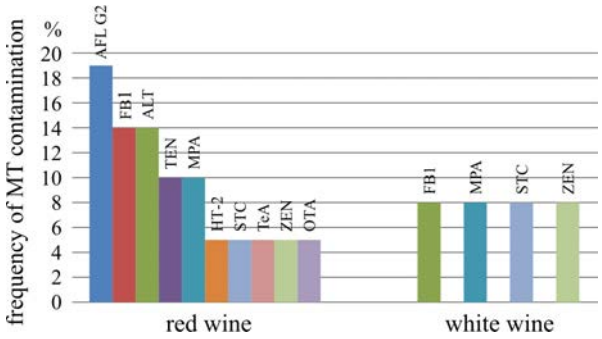

Figure 1. Frequency of MT contamination in red (n = 21) and white (n = 12)

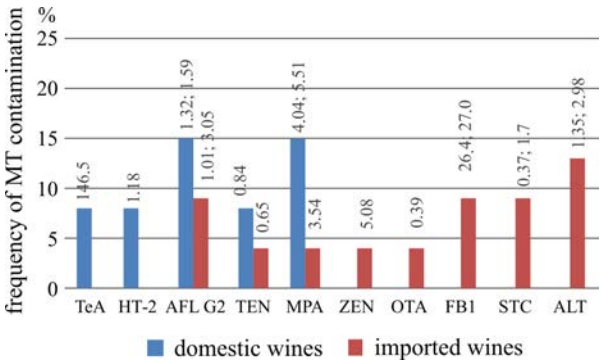

Figure 2. Frequency and levels of MT contamination in wine samples of different origin (MT content is given in μg/kg)

were found both in domestic and imported wines and frequency of their detection was several times higher in Russian wines. It is noteworthy that FB1, ZEN, STC, ALT and OTA were found only in imported wines.

It is noteworthy that three red wine samples and one white wine sample were simultaneously contaminated with several toxins. Table 2 provides the list of contaminated wines by variety, country of origin and detected MTs. STC and ZEN were found in an Italian dry white wine. AFL G2 and ALT were found in a Spanish dry red wine; four toxins (AFL G2, TEN, HT-2 and MPA) were found in a Russian red dry wine. A sweet red wine from Israel was contaminated with four toxins, OTA, STC, MPA and ALT. Two wine samples contained 2 MT (STC+ZEN and ALT+AFL G2); one sample contained 4 MT (ALT+MPA+OTA+STC); another one, 5 MT (TEN+AFL G2+HT-2+MPA+TeA).

Therefore, our study findings give evidence of OTA, AFL, FB1, STC occurrence in grape wines sold in Russia. It implies the establishment of appropriate control by producers and supervisory authorities, in particular, over the content of OTA.

According to the data provided by the Unified Interdepartmental Information Statistic System (UIISS), Federal Customs Service of the Russian Federation, Federal Center for Development of Agricultural Exports of the Ministry of Agriculture of the Russian Federation, Federal Service for Alcohol Market Regulation [26], wine consumption per capita

Table 2

Frequency and levels of MT contamination in wine samples depending on their origin

No.	Sugar contents	Country of origin	Toxin (concentration in μg/kg)
Red wine			
1	sweet	Israel	OTA (0.39) + STC (1.70) + MPA (3.54) + ALT (1.35)
2	sweet	Cyprus	FB1 (26.4)
3	dry	Georgia	TEN (0.65)
4	dry	Spain	AFL G2 (1.01) + ALT (2.98)
5	dry	Argentina	AFL G2 (3.05)
6	dry	Russia	AFL G2 (1.59) + TEN (0.84) + HT-2 (1.18) + MPA (5.51) + TeA (146.5)
7	dry	Chile	ALT (1.36)
8	semi-sweet	Russia	AFL G2 (1.32)
White wine			
9	dry	New Zealand	FB1 (27.04)
10	dry	Italy	STC (0.37) + ZEN (5.08)
11	semi-sweet	Russia	MPA (4.07)

varied between 6700 and 7400 cm^3/person a year in 2018–2020 in Russia. In 2020, it equaled 6700 cm^3. Recent data (2022) indicate that the highest amount of wine was consumed in the Nenets Autonomous Area, Karelia, Moscow region and Saint Petersburg (between 10,200 and 11,700 cm^3/person); the lowest amount, in North Caucasia (between 20 and 700 cm^3/person)[3].

Average weekly intake of the analyzed toxins for the Russian population varied between 0.07 ng/kg of body weight (0.0007 %) for TEN and 0.35 ng/kg of body weight (22.7–27.7 %) for AFL G2; for OTA, it was equal to 0.019 ng/kg of body weight (0.02 % of TWI). Maximum calculated intake of ZEN, FB1, OTA, HT-2, alternariatoxins and STC amounted to 0.27, 0.36, 0.64, 1.25, 2.6 and 2.82 % of the reference doses respectively. The highest probably MT intake was associated with consuming grape wine contaminated with AFL G2 at the level of 90 % (0.50 μg/kg) and at the maximum level (3.05 μg/kg). In this case, the total weekly intake could reach 0.91 ng/kg of body weight (between 9.6 and 31 % of the reference dose) and 5.6 ng/kg of body weight (58.8–190.5 % of the reference dose) respectively. The calculated AFL G2 intake can be 1.5–1.75 times higher than these doses for the population of the Nenets Autonomous Area, Karelia, Moscow region and Saint Petersburg.

The results of this study give evidence of insignificant intake of *Alternaria* toxins, STC, FB1, HT-2 and OTA and a higher AFL G2 intake associated with grape wine consumption. There is still a potential hazard of their chronic intake by humans.

Conclusion:

1. MT contamination was found in 31 % of the analyzed grape wine samples; predominantly, aflatoxin G2, altenuene, mycophenolic acid, fumonisin B1 and sterigmatocystin. Among regulated toxins, ochratoxin A, aflatoxin G2, fumonisin B1, zearalenone and sterigmatocystin were found in foreign wines and aflatoxin G2 – in domestic wines.

2. Estimated intake of mycotoxins in wine gives evidence of low relevant health risks (up to 2.8 % of their reference doses) for the Russian population. Aflatoxin G2 is the only exception as its average intake with wine can reach 28 %.

3. Grape wine as a source of mycotoxins does not pose any real health hazard for the country population. However, the detection of high levels of AFL G2 in grape wine samples implies the establishment of appropriate controls by producers and regulatory authorities on the content of this toxin and the EU regulated OTA.

Funding. The research was funded by the subsidy for the state assignment within the framework of the program of Fundamental Scientific Research (FGMF-2022-0003).

Competing interests. The authors declare no competing interests.

References

1. Welke J.E. Fungal and mycotoxin problems in grape juice and wine industries. *Current Opinion in Food Science*, 2019, vol. 29, pp. 7–13. DOI: 10.1016/j.cofs.2019.06.009

2. Čepo D.V., Pelajić M., Vrček I.V., Krivohlavek A., Žuntar I., Karoglan M. Differences in the levels of pesticides, metals, sulphites and ochratoxin A between organically and conventionally produced wines. *Food Chem.*, 2018, vol. 246, pp. 394–403. DOI: 10.1016/j.foodchem.2017.10.133

3. Zhang B., Chen X., Han S.-Y., Li M., Ma T.-Z., Sheng W.-J., Zhu X. Simultaneous Analysis of 20 Mycotoxins in Grapes and Wines from Hexi Corridor Region (China): Based on a QuEChERS-UHPLC-MS/MS Method. *Molecules*, 2018, vol. 23, no. 8, pp. 1926. DOI: 10.3390/molecules23081926

[3] Top-10 regionov Rossii po potrebleniyu vina [Top-10 Russian regions per wine consumption]. *OOO 'LUDING'*. Available at: https://luding.ru/news/top-10-regionov-rossii-po-potrebleniyu-vina (August 11, 2024) (in Russian).

4. Ostry V., Malir F., Cumova M., Kyrova V., Toman J., Grosse Y., Pospichalova M., Ruprich J. Investigation of patulin and citrinin in grape must and wine from grapes naturally contaminated by strains of *Penicillium expansum*. *Food Chem. Toxicol.*, 2018, vol. 118, pp. 805–811. DOI: 10.1016/j.fct.2018.06.022

5. Freire L., Braga P.A., Furtado M.M., Delafiori J., Dias-Audibert F.L., Pereira G.E., Reyes F.G., Catharino R.R., Sant'Ana A.S. From grape to wine: Fate of ochratoxin A during red, rose, and white winemaking process and the presence of ochratoxin derivatives in the final products. *Food Control*, 2020, vol. 113, no. 1, pp. 107167. DOI: 10.1016/j.foodcont.2020.107167

6. Omotayo O.P., Omotayo A.O., Mwanza M., Babalola O.O. Prevalence of mycotoxins and their consequences on human health. *Toxicol. Res.*, 2019, vol. 35, no. 1, pp. 1–7. DOI: 10.5487/TR.2019.35.1.001

7. JECFA. Evaluation of certain food additives and contaminants: sixty-eighth report of the Joint FAO/WHO Expert Committee on Food Additives. *WHO Technical Report Series*, 2007, no. 947. Available at: https://iris.who.int/bitstream/handle/10665/43870/9789241209472_eng.pdf?sequence=1 (April 01, 2024).

8. Ortiz-Villeda B., Lobos O., Aguilar-Zuniga K., Carrasco-Sánchez V. Ochratoxins in Wines: A Review of Their Occurrence in the Last Decade, Toxicity, and Exposure Risk in Humans. *Toxins (Basel)*, 2021, vol. 13, no. 7, pp. 478. DOI: 10.3390/toxins13070478

9. Lasram S., Mani A., Zaied C., Chebil S., Abid S., Bacha H., Mliki A., Ghorbel A. Evolution of ochratoxin A content during red and rose vinification. *J. Sci. Food Agric.*, 2008, vol. 88, no. 10, pp. 1696–1703. DOI: 10.1002/jsfa.3266

10. Aksionov I.V. Study of content of mikotoksin ohratoxin A in grape wines. *Plodovodstvo i vinogradarstvo Yuga Rossii*, 2018, no. 54 (06), pp. 124–129. DOI: 10.30679/2219-5335-2018-6-54-124-129 (in Russian).

11. De Jesus C.L., Bartley A., Welch A.Z., Berry J.P. High incidence and levels of ochratoxin A in wines sourced from the United States. *Toxins (Basel)*, 2018, vol. 10, no. 1, pp. 1. DOI: 10.3390/toxins10010001

12. Remiro R., Irigoyen A., González-Peñas E., Lizarraga E., López de Cerain A. Levels of ochratoxin in Mediterranean red wines. *Food Control*, 2013, vol. 32, no. 1, pp. 63–68. DOI: 10.1016/j.foodcont.2012.11.040

13. Sarigiannis Y., Kapolos J., Koliadima A., Tsegenidis T., Karaiskakis G. Ochratoxin A levels in Greek retail wines. *Food Control*, 2014, vol. 42, pp. 139–143. DOI: 10.1016/j.foodcont.2014.02.003

14. Pizzutti I.R., Kok A., Scholten J., Righi L.W., Cardoso C.D., Rohers G.N., da Silva R.C. Development, optimization and validation of a multimethod for the determination of 36 mycotoxins in Ines by liquid chromatography–tandem mass spectrometry. *Talanta*, 2014, vol. 129, pp. 352–363. DOI: 10.1016/j.talanta.2014.05.017

15. Remiro R., González-Peñas E., Lizarraga E., Lopez de Cerain A. Quantification of ochratoxin A and five analogs in Navarra red wines. *Food Control*, 2012, vol. 27, no. 1, pp. 139–145. DOI: 10.1016/j.foodcont.2012.03.006

16. Di Stefano V., Pitonzo R., Avellone G., Di Fiore A., Monte L., Ogorka A.Z.T. Determination of Aflatoxins and Ochratoxins in Sicilian Sweet Wines by High-Performance Liquid Chromatography with Fluorometric Detection and Immunoaffinity Cleanup. *Food Anal. Methods*, 2015, vol. 8, pp. 569–577. DOI: 10.1007/s12161-014-9934-3

17. Carballo D., Fernández-Franzón M., Ferrer E., Pallarés N., Berrada H. Dietary Exposure to Mycotoxins through Alcoholic and Non-Alcoholic Beverages in Valencia, Spain. *Toxins (Basel)*, 2021, vol. 13, no. 7, pp. 438. DOI: 10.3390/toxins13070438

18. Silva L.J.G., Rodrigues A.P., Pereira A.M.P.T., Lino C.M., Pena A. Ochratoxin A in the Portuguese Wine Market, Occurrence and Risk Assessment. *Food Addit. Contam. Part B Surveill.*, 2019, vol. 12, no. 2, pp. 145–149. DOI: 10.1080/19393210.2019.1595169

19. Torovic L., Lakatoš I., Majkic T., Beara I. Risk to public health related to the presence of ochratoxin A in wines from Fruška Gora. *LWT*, 2020, vol. 129, no. S3, pp. 109537. DOI: 10.1016/j.lwt.2020.109537

20. Mariño-Repizo L., Gargantini R., Manzano H., Raba J., Cerutti S. Assessment of ochratoxin A occurrence in Argentine red wines using a novel sensitive quechers-solid phase extraction approach prior to ultra high performance liquid chromatography-tandem mass spectrometry methodology. *J. Sci. Food Agric.*, 2017, vol. 97, no. 8, pp. 2487–2497. DOI: 10.1002/jsfa.8065

21. Terra M.F., Prado G., Pereira G.E., Ematne H.J., Batista L.R. Detection of ochratoxin A in tropical wine and grape juice from Brazil. *J. Sci. Food Agric.*, 2013, vol. 93, no. 4, pp. 890–894. DOI: 10.1002/jsfa.5817

22. Zhong Q.D., Li G.H., Wang D.B., Shao Y., Li J.G., Xiong Z.H., Wu Y.N. Exposure Assessment to Ochratoxin A in Chinese Wine. *J. Agric. Food Chem.*, 2014, vol. 62, no. 35, pp. 8908–8913. DOI: 10.1021/jf500713x

23. Puangkham S., Poapolathep A., Jermnak U., Imsilp K., Tanhan P., Chokejaroenrat C., Poapolathep S. Monitoring and health risk of mycotoxins in imported wines and beers consumed in Thailand. *World Mycotoxin J.*, 2017, vol. 10, no. 4, pp. 401–409. DOI: 10.3920/WMJ2017.2216

24. Lee H.J., Kim H.D., Ryu D. Practical Strategies to Reduce Ochratoxin A in Foods. *Toxins*, 2024, vol. 16, no. 1, pp. 58. DOI: 10.3390/ toxins16010058

25. Zwickel T., Klaffke H., Richards K., Rychlik M. Development of a high performance liquid chromatography tandem mass spectrometry based analysis for the simultaneous quantification of various *Alternaria* toxins in wine, vegetable juices and fruit juices. *J. Chromatogr. A*, 2016, vol. 1455, pp. 74–85. DOI: 10.1016/j.chroma.2016.04

26. Baimukhambetova L. Rossiiskoe vinodelie. Analiticheskoe issledovanie [Russian winemaking. Analytical study]. *NCR: National Credit Ratings*, 2021. Available at: https://ratings.ru/files/research/corps/NCR_Wine_July2021.pdf (May 12, 2024) (in Russian).

27. JECFA. Safety evaluation of certain food additives and contaminants: prepared by the Fifty-third meeting of the Joint FAO/WHO Expert Committee on Food Additives. *WHO Food Additives Series*, 2000, no. 44. Available at: https://inchem.org/documents/jecfa/jecmono/v44jec14.htm (April 01, 2024).

28. FAO JECFA Monographs 19 bis. Safety evaluation of certain contaminants in food: prepared by the eighty-third meeting of the Joint FAO/WHO Expert Committee on Food Additives. *WHO Food Additives Series*, 2018, no. 74. Available at: https://iris.who.int/bitstream/handle/10665/276868/9789241660747-eng.pdf?ua=1#page=423 (April 01, 2024).

29. JECFA. Summary and conclusions: Summary report of the ninety-third meeting of JECFA. Virtual meeting, 24, 25, 29, 30 March and 1 April 2022. Available at: https://cdn.who.int/media/docs/default-source/food-safety/jecfa/summary-and-conclusions/jecfa93-summary-and-conclusions-april2022.pdf?sfvrsn=33db6aca_3 (April 01, 2024).

30. JECFA. Safety evaluation of certain food additives and contaminants: prepared by the Sixty-eighth meeting of the Joint FAO/WHO Expert Committee on Food Additives. *WHO Food Additives Series*, 2008, no. 59, pp. 357–429. Available at: www.who.int/publications/i/item/9789241660594 (April 01, 2024).

31. EFSA Panel on Contaminants in the Food Chain (CONTAM), Schrenk D., Bignami M., Bodin L., Chipman J.K., del Mazo J., Grasl-Kraupp B., Hogstrand C. [et al.]. Risk assessment of aflatoxins in food. *EFSA J.*, 2020, vol. 18, no. 3, pp. e6040. DOI: 10.2903/j.efsa.2020.6040

32. European Food Safety Authority, Arcella D., Eskola M., Gomez Ruiz J.A. Dietary exposure assessment to *Alternaria* toxins in the European population. *EFSA Journal*, 2016, vol. 14, no. 12, pp. e04654. DOI: 10.2903/j.efsa.2016.465

33. Mujahid C., Savoy M-C., Baslé Q., Woo P.M., Ee E.C.Y., Mottier P., Bessaire T. Levels of *Alternaria* Toxins in Selected Food Commodities Including Green Coffee. *Toxins (Basel)*, 2020, vol. 12, no. 9, pp. 595. DOI: 10.3390/toxins12090595T

34. EFSA Panel on Contaminants in the Food Chain (CONTAM). Scientific Opinion on the risk for public and animal health related to the presence of sterigmatocystin in food and feed. *EFSA Journal*, 2013, vol. 11, no. 6, pp. 3254. DOI: 10.2903/j.efsa.2013.3254

Sedova I.B., Chalyy Z.A., Tutelyan V.A. Mycotoxins contamination of grape wines marketed in the Russian Federation. Health Risk Analysis, 2024, no. 4, pp. 63–71. DOI: 10.21668/health.risk/2024.4.06.eng

Received: 20.09.2024
Approved: 17.11.2024
Accepted for publication: 17.12.2024