toxins

MDPI

*Article*

# Incidence of Aflatoxins and Ochratoxin A in Wheat and Corn from Albania

**Dritan Topi [1,2,*], Janja Babič [1], Breda Jakovac-Strajn [1] and Gabrijela Tavčar-Kalcher [1]**

[1] Veterinary Faculty, Institute of Food Safety, Feed and Environment, University of Ljubljana, Gerbičeva 60, 1000 Ljubljana, Slovenia; janja.babic@vf.uni-lj.si (J.B.); breda.jakovacstrajn@vf.uni-lj.si (B.J.-S.); gabrijela.tavcar-kalcher@vf.uni-lj.si (G.T.-K.)

[2] Department of Chemistry, Faculty of Natural Sciences, University of Tirana, Boulevard Zogu 1, 25/1, 1000 Tirana, Albania

* Correspondence: dritan.topi@unitir.edu.al

**Abstract:** In this study, aflatoxins (AFs) and ochratoxin A (OTA) were analyzed in grains, specifically wheat and corn, from Albania. To summarize, 71 wheat and 45 corn samples from different growing areas were collected. The multi-toxin analytical procedure involved sample extraction and liquid chromatography–tandem mass spectrometry (LC–MS/MS). The incidence of AF was 18% in the analyzed wheat and 71% in the corn samples. The concentration of AFs was much higher in the corn samples than in the wheat samples. The maximum permitted levels for aflatoxin B1 (AFB1) and total AFs were not exceeded in the wheat samples, while they were exceeded in 36% of the corn samples. In the wheat samples, the AFB1 concentration varied between 0.2 and 0.4 µg $kg^{-1}$. However, the highest concentrations in the corn samples were 2057, 2944, and 3550 µg $kg^{-1}$. OTA was present in only three corn samples and one wheat sample. However, all contaminated samples exceeded the maximum permitted levels. This report reveals the presence of AFs and OTA in grain commodities, specifically wheat and corn, grown in Albania.

**Keywords:** aflatoxins; ochratoxin A; liquid chromatography–tandem mass spectrometry; corn; wheat; Albania

**Key Contribution:** This study provides information on the occurrence of aflatoxins and ochratoxin A in corn and wheat produced in Albania.



**Citation:** Topi, D.; Babič, J.; Jakovac-Strajn, B.; Tavčar-Kalcher, G. Incidence of Aflatoxins and Ochratoxin A in Wheat and Corn from Albania. *Toxins* **2023**, *15*, 567. https://doi.org/10.3390/toxins15090567

Received: 14 August 2023
Revised: 2 September 2023
Accepted: 7 September 2023
Published: 12 September 2023



## 1. Introduction

Mycotoxins are secondary metabolites of low molecular weight produced by filamentous fungi, or more specifically, molds [1–11]. They contaminate cereals and other foods such as nuts, spices, fruits, and their by-products [8,12,13]. Contamination of cereal crops with toxigenic fungi and, hence, mycotoxins can occur in the field and during harvesting, handling, storage, and processing [1,4,6,8–11,14–16]. Exposure to mycotoxins in food is associated with adverse effects on humans and animals, which can be both acute and chronic [7–9,11,16–21]. They are considered one of the most important diet risks, which is why the content in food is regulated in the European Union [22].

Aflatoxins (AFs) are chemically classified as polyketide-derived difuranocoumarins, produced mainly by two *Aspergillus* species. *A. flavus*, the most important producer, is ubiquitous, favoring the aerial parts of plants and producing B-type aflatoxins, while *A. parasiticus* is adapted to the soil environment with more limited distribution, capable of further expanding oxidative rings to produce G-type aflatoxins [4,23,24]. Contamination of cereals with AFs occurs mainly during storage [6,7], but can also occur in the field, before and during harvesting [4,14,15,17,25].

There are four main types of AFs: Aflatoxin B1 (AFB1), aflatoxin B2 (AFB2), aflatoxin G1 (AFG1), and aflatoxin G2 (AFG2). AFB1 is considered the most toxic aflatoxin and the most potent carcinogen, classified as Group 1 ("carcinogenic to humans") by the International Agency of Research on Cancer [26]. It is estimated that aflatoxin exposure may be responsible for 5–28% of all cases of hepatocellular carcinoma worldwide [27]. Chronic exposure to AFs in food commodities manifests immunosuppressive, genotoxic, mutagenic, teratogenic, hepatotoxic, and hepatocarcinogenic effects [4,6–8,17,21,24,25,28]. High levels of exposure are manifested as acute aflatoxicosis [4,6–8,17,21,24,25,28].

Ochratoxin A (OTA) is a natural contaminant in many foodstuffs, including cocoa beans, coffee beans, cassava flour, cereals, fish, peanuts, dried fruits, wine, and meat products such as salami and ham. It is produced by fungi of the genera *Aspergillus* and *Penicillium*. [4,29,30]. OTA possesses nephrotoxic, hepatotoxic, neurotoxic, teratogenic, and immunotoxic effects [30].

The presence of AFs and OTA in foodstuffs is regulated by Commission Regulation (EU) No. 2023/915 [22]. The maximum permitted levels of AFB1 and the sum of AFB1, AFB2, AFG1, and AFG2 in corn and rice intended for consumers or use as an ingredient in food are 5.0 and 10.0 µg $kg^{-1}$, respectively. In other cereals, they are 2.0 and 4.0 µg $kg^{-1}$, respectively. The maximum permitted level of OTA in unprocessed cereal grains is 5 µg $kg^{-1}$.

Favorable growth regions for aflatoxigenic fungi belong to tropical and subtropical regions [5,28,31]. Due to climate change, AF contamination of cereal crops has been documented in southern Europe in recent decades [16,32–39]. Further reports on contamination with Afs, as well as with OTA, in cereals and cereal products from European countries have been published by Binder et al. [40], Serrano et al. [41], Bryła et al. [42], Jakovac–Strajn et al. [43], Alkadri et al. [44], Kirinčič et al. [45], EFSA [24], Pleadin et al. [46,47], Gagiu et al. [38], and Marin et al. [4]. There are several surveys on the occurrence of AFs from other parts of the world reported by Gruber–Dorninger et al. [48], Rodrigues et al. [49], Aasa et al. [50], Ayeni et al. [51], Abdallah et al. [52], Dong et al., [53], Jiang et al. [54], and Sun et al. [55].

The analytical determination of mycotoxins In food and feed commodities is carried out by a number of quantitative and semi-quantitative techniques, enzyme-linked immunosorbent assay (ELISA) [32–34,38,46,47], gas chromatography–mass spectrometry [7,18], and liquid chromatography coupled with different detectors, including ultraviolet [32,40,45], fluorescence [32,33,40,52,56], and mass spectrometry [35,39,41,42,44,46,54]. Liquid chromatography with fluorescence detection (HPLC–FLD) is often employed for quantitatively identifying individual mycotoxins. Their simultaneous determination is possible using a technique that combines liquid chromatography and mass spectrometry [7,18,57].

This study aimed to obtain data on the occurrence of aFs and OTA in cereals from Albania. Sample collection was carried out from different regions of the country, considering production capacity and geography. The countrywide annual output of corn, wheat, barley, and rye is 380,000, 275,000, 7500, and 3000 tons, respectively, according to data from the FAO (Food and Agriculture Organization) [58]. Accordingly, mainly wheat and corn samples were taken, but also a limited number of samples of barley and rye. The analytical procedure involved a sample extraction and LC–MS/MS. This study is the first-ever report on the presence of Afs and OTA in plant commodities from Albania. The data will expand the information on the presence of mycotoxins in different cereals from southern Europe, a region of growing interest in relation to the impact of climate change.

## 2. Results and Discussion

### 2.1. Occurrence of aFs and OTA in Grains

The grain samples containing one or more individual AF or OTA at levels above the limit of quantification (LOQ) were considered positive. Overall, 40% of the grain samples tested were contaminated. However, there were large differences in contamination between corn and wheat and between harvesting years. The contamination rate for corn was 71%, while it was only 20% for wheat. In 2014, 90% of the corn samples were contaminated, while in 2015, only 29% were. The contamination rate for wheat was 40% in 2014, but no incidence was revealed in 2015. The incidence of positive samples, mean, median, and minimum and maximum concentrations of positive samples are shown in Tables 1 and 2.

**Table 1.** Occurrence of aflatoxins and OTA in corn from the 2014 and 2015 harvesting seasons.

| | AFB1 | AFB2 | AFG1 | AFG2 | Sum | OTA |
|---|---|---|---|---|---|---|
| 2014 | | | | | | |
| Analyzed samples | 31 | 31 | 31 | 31 | 31 | 31 |
| Positive samples | 23 | 15 | 15 | 25 | 28 | 2 |
| Incidence (%) | 74 | 48 | 48 | 81 | 90 | 6.0 |
| Mean value (μg $kg^{-1}$) | 464 | 93.9 | 158 | 16.4 | 531 | 260 |
| Median value (μg $kg^{-1}$) | 21.1 | 8.5 | 5.4 | 1.3 | 6.6 | 260 |
| Minimum value (μg $kg^{-1}$) | 0.3 | 0.2 | 0.2 | 0.2 | 0.2 | 187 |
| Maximum value (μg $kg^{-1}$) | 3550 | 539 | 978 | 144 | 4822 | 333 |
| 2015 | | | | | | |
| Analyzed samples | 14 | 14 | 14 | 14 | 14 | 14 |
| Positive samples | 3 | 0 | 0 | 1 | 4 | 1 |
| Incidence (%) | 21 | 0.0 | 0.0 | 7.1 | 29 | 7.1 |
| Mean value (μg $kg^{-1}$) | 55.7 | - | - | 0.2 | 41.8 | 488 |
| Median value (μg $kg^{-1}$) | 31.7 | - | - | 0.2 | 20.6 | 488 |
| Minimum value (μg $kg^{-1}$) | 9.4 | - | - | 0.2 | 0.2 | 488 |
| Maximum value (μg $kg^{-1}$) | 126 | - | - | - | 126 | 488 |
| 2014–2015 | | | | | | |
| Analyzed samples | 45 | 45 | 45 | 45 | 45 | 45 |
| Positive samples | 26 | 15 | 15 | 26 | 32 | 3 |
| Incidence (%) | 58 | 33 | 33 | 58 | 71 | 7.0 |
| Mean value (μg $kg^{-1}$) | 417 | 93.9 | 158 | 15.8 | 469 | 336 |
| Median value (μg $kg^{-1}$) | 22.2 | 8.5 | 5.4 | 1.3 | 8.5 | 333 |
| Minimum value (μg $kg^{-1}$) | 0.3 | 0.2 | 0.2 | 0.2 | 0.2 | 187 |
| Maximum value (μg $kg^{-1}$) | 3550 | 539 | 978 | 144 | 4822 | 488 |

**Table 2.** Aflatoxins and ochratoxin A occurrence in wheat from the 2014 and 2015 harvesting seasons.

| | AFB1 | AFB2 | AFG1 | AFG2 | Sum | OTA |
|---|---|---|---|---|---|---|
| 2014 | | | | | | |
| Analyzed samples | 35 | 35 | 35 | 35 | 35 | 35 |
| Positive samples | 2 | 0 | 0 | 11 | 13 | 1 |
| Incidence (%) | 6 | 0 | 0 | 31 | 37 | 3 |
| Mean value (μg $kg^{-1}$) | 0.3 | - | - | 0.3 | 0.3 | 611 |
| Median value (μg $kg^{-1}$) | 0.3 | - | - | 0.2 | 0.2 | 611 |
| Minimum value (μg $kg^{-1}$) | 0.2 | - | - | 0.2 | 0.2 | 611 |
| Maximum value (μg $kg^{-1}$) | 0.4 | - | - | 0.3 | 0.4 | 611 |
| 2015 | | | | | | |
| Analyzed samples | 36 | 36 | 36 | 36 | 36 | 36 |
| Positive samples | 0 | 0 | 0 | 0 | 0 | 0 |
| Incidence (%) | - | - | - | - | - | - |
| Mean value (μg $kg^{-1}$) | - | - | - | - | - | - |
| Median value (μg $kg^{-1}$) | - | - | - | - | - | - |
| Minimum value (μg $kg^{-1}$) | - | - | - | - | - | - |
| Maximum value (μg $kg^{-1}$) | - | - | - | - | - | - |
| 2014–2015 | | | | | | |
| Analyzed samples | 71 | 71 | 71 | 71 | 71 | 71 |
| Positive samples | 2 | 0 | 0 | 11 | 13 | 1 |
| Incidence (%) | 3 | 0 | 0 | 15 | 18 | 1.5 |
| Mean value (μg $kg^{-1}$) | 0.3 | - | - | 0.3 | 0.3 | 611 |
| Median value (μg $kg^{-1}$) | 0.3 | - | - | 0.2 | 0.2 | 611 |
| Minimum value (μg $kg^{-1}$) | 0.2 | - | - | 0.2 | 0.2 | 611 |
| Maximum value (μg $kg^{-1}$) | 0.4 | - | - | 0.3 | 0.4 | 611 |

As shown in Tables 1 and 2, the AF levels were much higher in the corn compared to the wheat samples. While the AFB1 concentration in the wheat samples ranged between 0.2 and 0.4 µg kg$^{-1}$, the concentrations in corn were as high as 2944 and 3550 µg kg$^{-1}$ (all in 2014). The maximum permitted levels for AFB1 and total aFs [22] were not exceeded in the wheat samples, but they were 4 in 16 corn samples (36%). Three of them were from the 2015 harvesting year, and the other 13 samples were from 2014. Thus, more than 40% of the corn samples in 2014 did not comply with the legal requirements [22], which can pose a significant health risk for consumers. Therefore, regular and systematic control of the presence of aFs in grain commodities and actions for their reduction are crucial.

Altogether, 34% of the contaminated corn samples contained only one AF (either AFB1 or AFG2), and 38% of the contaminated corn samples contained all four aFs. Meanwhile, 13% contained two and 16% contained three aFs. AFB1 and AFG2 were present in 81% of the contaminated samples, while AFB2 and AFG1 were present in 47% of the contaminated samples. All contaminated wheat samples contained only one AF (either AFG1 or AFG2). AFG2 was present in 31% and AFG1 in 6% of the wheat samples from 2014.

In total, only three corn samples (7%) and one wheat sample (1.5%) were contaminated with OTA. However, the concentrations were high. They exceeded the maximum permitted level in all contaminated samples. The differences between the years were smaller than in the case of aFs. In 2014, the incidence of OTA in corn was 6% and in 2015 it was 7%. The maximum concentrations were 333 and 488 µg kg$^{-1}$ in 2014 and 2015, respectively. OTA was not detected in the barley and rye samples, while in one sample of rye, AFB2 was present (0.61 µg kg$^{-1}$).

The data on the incidence of aFs and OTA in corn commodities according to the regions are presented in Table 3. The incidence is comparable in all five regions (71–100%). Additionally, the mean concentrations were comparable in all regions. However, there were significant differences in the maximum concentrations of AFB1 and the sum of aFs among the regions.

**Table 3.** Occurrence of aflatoxins and ochratoxin A in corn according to region from the harvesting year 2014.

| | AFB1 | AFB2 | AFG1 | AFG2 | Sum | OTA |
|---|---|---|---|---|---|---|
| Fieri | | | | | | |
| Analyzed samples | 4 | 4 | 4 | 4 | 4 | 4 |
| Positive samples | 4 | 3 | 4 | 4 | 4 | 0 |
| Incidence (%) | 100 | 75 | 100 | 100 | 100 | 0.0 |
| Mean value (µg kg$^{-1}$) | 315 | 39.6 | 5.08 | 2.50 | 352 | - |
| Median value (µg kg$^{-1}$) | 12.7 | 1.91 | 2.92 | 2.43 | 18.7 | - |
| Minimum value (µg kg$^{-1}$) | 2.82 | 0.20 | 0.22 | 0.82 | 5.65 | - |
| Maximum value (µg kg$^{-1}$) | 1232 | 117 | 14.3 | 4.34 | 1367 | - |
| Lushnja | | | | | | |
| Analyzed samples | 7 | 7 | 7 | 7 | 7 | 7 |
| Positive samples | 7 | 6 | 7 | 7 | 7 | 1 |
| Incidence (%) | 100 | 85.7 | 100 | 100 | 100 | 14 |
| Mean value (µg kg$^{-1}$) | 1235 | 173 | 332 | 54.7 | 1795 | 187 |
| Median value (µg kg$^{-1}$) | 36.2 | 155 | 7.22 | 3.08 | 46.7 | 187 |
| Minimum value (µg kg$^{-1}$) | 5.46 | 1.79 | 2.39 | 0.2 | 1.0 | 187 |
| Maximum value (µg kg$^{-1}$) | 3550 | 539 | 978 | 144 | 4822 | 187 |
| Kruja | | | | | | |
| Analyzed samples | 7 | 7 | 7 | 7 | 7 | 7 |
| Positive samples | 7 | 5 | 3 | 6 | 7 | 1 |
| Incidence (%) | 100 | 71 | 43 | 86 | 100 | 14 |
| Mean value (µg kg$^{-1}$) | 93.7 | 14.2 | 8.98 | 1.60 | 109 | 333 |
| Median value (µg kg$^{-1}$) | 10.6 | 10.0 | 0.32 | 1.01 | 12.0 | 333 |
| Minimum value (µg kg$^{-1}$) | 0.32 | 0.21 | 0.16 | 0.18 | 0.32 | 333 |
| Maximum value (µg kg$^{-1}$) | 344 | 47.5 | 26.5 | 3.47 | 393 | 333 |

**Table 3.** *Cont.*

| | AFB1 | AFB2 | AFG1 | AFG2 | Sum | OTA |
|---|---|---|---|---|---|---|
| Elbasan | | | | | | |
| Analyzed samples | 6 | 6 | 6 | 6 | 6 | 6 |
| Positive samples | 2 | 1 | 0 | 5 | 5 | 0 |
| Incidence (%) | 33 | 17 | 0.0 | 83 | 83 | 0.0 |
| Mean value (µg kg$^{-1}$) | 48.6 | 8.54 | - | 1.03 | 22.2 | - |
| Median value (µg kg$^{-1}$) | 48.6 | 8.54 | - | 1.17 | 1.58 | - |
| Minimum value (µg kg$^{-1}$) | 1.42 | 8.54 | - | 0.25 | 0.25 | - |
| Maximum value (µg kg$^{-1}$) | 95.8 | 8.54 | - | 1.58 | 106 | - |
| Korça | | | | | | |
| Analyzed samples | 7 | 7 | 7 | 7 | 7 | 7 |
| Positive samples | 3 | 0 | 1 | 3 | 5 | 0 |
| Incidence (%) | 43 | 0.0 | 14 | 43 | 71 | 0.0 |
| Mean value (µg kg$^{-1}$) | 1.29 | - | 0.24 | 0.80 | 1.30 | - |
| Median value (µg kg$^{-1}$) | 1.39 | - | 0.24 | 0.77 | 1.39 | - |
| Minimum value (µg kg$^{-1}$) | 0.85 | - | 0.24 | 0.56 | 0.56 | - |
| Maximum value (µg kg$^{-1}$) | 1.62 | - | 0.24 | 1.06 | 1.86 | - |

Samples from two regions, Fieri and Lushnja, located in the western plain in the Adriatic Sea, had higher maximum levels than samples from Kruja, Elbasan, and especially the eastern region of Korça. Concerning the maximum concentrations, the order of incidence according to the areas was Lushnja > Fieri > Kruja > Elbasan > Korça. Except for Korça, the other regions are located in the western plain, characterized by a Mediterranean climate of a hot summer and a humid winter, while the eastern areas of the country are characterized by a temperate continental climate. The highest AFB1 levels were found in a sample from Lushnja (3550 µg kg$^{-1}$) and a corn sample from the Fieri region (1232 µg kg$^{-1}$), which are the main producing regions of corn and dairy [59].

### 2.2. Comparison with the Incidence of aFs from Other Countries

The incidence of AFB1 and aFs in corn samples in our study (58% and 71%, respectively) is comparable to the results from Serbia [33,35,56] given in Table 4, while in other studies, a lower incidence was reported [4,38,39,43,45,47–49,52–54]. In Croatia, Slovenia, Romania, the EU, Egypt, China, the Middle East, and African countries, it was 0–45.4% [4,38,39,43,45,47–49,52–54]. In a global survey on the occurrence of mycotoxins in feed performed in 100 countries, an AFB1 incidence of 24% was reported [48], while Lee and Ryu [60] reported a 55% incidence for aFs based on global occurrence data reported from 2006 to 2016. The determined concentrations were much higher than in all other countries, with the exception of the study from Croatia [47] and the worldwide occurrence [60], where comparable maximum concentrations (2072 and 1642 µg kg$^{-1}$, respectively) were reported. However, as mentioned above, there was also a big difference between the 2014 and 2015 occurrences in Albania, where 90% of the corn samples were contaminated in 2014 and only 29% in 2015. The maximum concentrations were 4822 and 126 µg kg$^{-1}$, respectively.

**Table 4.** Occurrence of aflatoxins and ochratoxin A in corn samples from different studies.

| Country | Sampling Year | Analytical Method | LOD/LOQ (µg kg$^{-1}$) | Mycotoxin | No. of Samples | Positive Sample Rate (%) | Mean (µg kg$^{-1}$) | Median (µg kg$^{-1}$) | Max (µg kg$^{-1}$) | Reference |
|---|---|---|---|---|---|---|---|---|---|---|
| Albania | 2014–2015 | LC–MS/MS | 0.6/2.0 | AFB1 | 45 | 58 | 417 [a] | 22.2 [a] | 3550 | This study |
| | | | | aFs | 45 | 71 | 469 [a] | 8.5 [a] | 263 | |
| | | | | OTA | 45 | 7.0 | 336 [a] | 333 [a] | 488 | |
| Serbia | 2012 | LC–MS/MS | 0.33/1.0 | AFB1 | 40 | 60 | - | - | 70.3 | [33] |

**Table 4.** *Cont.*

| Country | Sampling Year | Analytical Method | LOD/LOQ (μg kg$^{-1}$) | Mycotoxin | No. of Samples | Positive Sample Rate (%) | Mean (μg kg$^{-1}$) | Median (μg kg$^{-1}$) | Max (μg kg$^{-1}$) | Reference |
|---|---|---|---|---|---|---|---|---|---|---|
| | | ELISA | 1.40/5.0 | | | | | | | |
| Serbia | 2015 | HPLC–FLD | 0.4/1.3 | AFB1 | 180 | 57.2 | 11.4 ± 14.5 | - | 88.8 | [56] |
| | | | - | aFs | 180 | 57.2 | 12.7 ± 17.3 | - | 91.4 | |
| Serbia | 2012 | LC–MS/MS | 0.25/- | AFB1 | 51 | 94 | 44 ± 49 | 26 | 205 | [35] |
| | 2013 | | | | 51 | 33 | 8 ± 11 | 5 | 48 | |
| | 2014 | | | | 51 | 0 | - | - | - | |
| | 2015 | | | | 51 | 90 | 8 ± 9 | 4 | 41 | |
| | 2012 | | 0.4/- | OTA | 51 | 25 | 53 ± 108 | 6 | 318 | |
| | 2013 | | | | 51 | 2 | - | - | - | |
| | 2014 | | | | 51 | 0 | - | - | - | |
| | 2015 | | | | 51 | 18 | 6 ± 8 | 5 | 27 | |
| Croatia | 2013 | ELISA | 1.0/1.7 | AFB1 | 972 | 31.4 | 38.46 | - | 2072 | [47] |
| Croatia | 2016 | LC–MS/MS | 0.3/1.0 | AFB1 | 61 | 0 | - | - | - | [39] |
| | 2017 | | | | 23 | 8.7 | 5.5 | - | 9.7 | |
| | 2016 | | 0.3/1.0 | OTA | 61 | 0 | - | - | - | |
| | 2017 | | | | 23 | 0 | - | - | - | |
| Slovenia | 2007–2008 | HPLC–FLD | 0.2/0.6 | AFB1 | 58 | 0 | - | - | - | [43] |
| | | | 10/30 | OTA | 58 | 1.7 | 30 | 30 | 30 | |
| Slovenia | 2008–2012 | LC–MS/MS | -/0.2 | AFB1 | 69 [b] | 0 | - | - | - | [45] |
| | | | -/0.8 | aFs | 69 [b] | 0 | - | - | - | |
| | | | -/1 | OTA | 69 [b] | 0 | - | - | - | |
| Romania | 2012–2015 | ELISA | - | aFs | 97 [c] | 45.4 | 3.85 ± 14.80 | <1.75 | 82.94 | [38] |
| | | | - | OTA | 97 [c] | 6.8 | 2.70 ± 0.43 | <2.50 | 6.72 | |
| EU | 2000–2006 | - | 0.1–0.2/- | AFB1 | 943 | 14 | 0.26 | 0.12 | 8 | [4] |
| | | | 0.2–0.4/- | aFs | 943 | 14 | 0.41 | 0.24 | 9 | |
| | | - | 0.01–0.5/- | OTA | 5180 [c] | 54 | 0.29 | - | 33.3 | |
| Egypt | 2014–2015 | LC–MS/MS | 0.72/2.4 | AFB1 | 79 | 16 | - | 4.81 | 197.5 | [52] |
| | | | 2.8/9.4 | OTA | 79 | 3 | - | <LOQ | 11 | |
| Middle East and African countries | 2009 | HPLC–FLD | 0.3/0.8 | aFs | 63 | 35 | 28 | 32 [a] | 343 | [49] |
| | | | 0.2/30.5 | OTA | 1 | 0 | - | - | - | |
| Shandong, China | 2014–2015 | UPLC–Q-TOF-MS | 0.05/0.1 | AFB1 | 520 | 18.08 | 7.62 | - | 573.13 | [53] |
| Shandong, China | 2016 | LC–MS/MS | 0.01/0.03 | AFB1 | 90 [b] | 32.2 | 0.22 | - | 8.04 | [54] |
| | | | | OTA | 90 [b] | 0 | - | - | - | |
| Global | 2008–2017 | HPLC | 0.2–2.7/- | AFB1 | 15,889 | 24 | - | 4 | 6105 | [48] |
| | | LC–MS/MS | 0.2–5/- | | | | | | | |
| | | ELISA | 1–3/- | | | | | | | |
| | | HPLC | 0.06–2/- | OTA | 6388 | 5 | - | 3 [a] | 889 | |
| | | LC–MS/MS | 0.2–100/- | | | | | | | |
| | | ELISA | 0.2–2/- | | | | | | | |
| Global | | - | - | aFs | | 55 [c] | | | 1642 | [60] |
| | | - | - | OTA | | 29 [c] | | | 1164 | |

[a] Only positive samples. [b] Corn and corn-based processed foods. [c] Cereals, processed cereals, and cereal-based food.

The incidence of AFB1 and aFs in the wheat samples in our study (3% and 18%, respectively) is comparable to the results from other countries, where an incidence of 0–19% was reported (Table 5), except in Romania, where the incidence was 45.4%. The maximum level of AFB1 and aFs in the wheat samples from Albania (0.4 μg kg$^{-1}$) was much lower than in other studies, where maximum AFB1 concentrations from 5.41 μg kg$^{-1}$ in a sample from Croatia [47] to 109 μg kg$^{-1}$ in a sample from EU [4] and 161 μg kg$^{-1}$ in a sample from a study on global occurrence of mycotoxins [48] were reported.

**Table 5.** Occurrence of aflatoxins and ochratoxin A in wheat samples from different studies.

| Country | Sampling Year | Analytical Method | LOD/LOQ (μg kg$^{-1}$) | Mycotoxin | No. of Samples | Positive Sample Rate (%) | Mean (μg kg$^{-1}$) | Median (μg kg$^{-1}$) | Max (μg kg$^{-1}$) | Reference |
|---|---|---|---|---|---|---|---|---|---|---|
| Albania | 2014–2015 | LC–MS/MS | 0.6/2.0 | AFB1 | 71 | 3 | 0.3 | 0.3 | 0.4 | This study |
| | | | | aFs | 71 | 18 | 0.3 | 0.2 | 0.4 | |
| | | | | OTA | 71 | 1.0 | 611 [a] | 611 [a] | 611 | |
| Serbia | 2012 | LC–MS/MS | 0.33/1.0 | AFB1 | 30 | 0 | - | - | - | [33] |
| | | ELISA | 1.40/5.0 | | | | | | | |
| Croatia | 2013 | ELISA | 1.0/1.7 | AFB1 | 201 | 7.5 | 1.65 | - | 5.41 | [47] |
| Croatia | 2016 | LC–MS/MS | 0.3/1.0 | AFB1 | 57 | 0 | - | - | - | [39] |
| | 2017 | | | | 47 | 2.1 | 16.2 | - | 16.2 | |
| | 2016 | | 0.3/1.0 | OTA | 57 | 0 | - | - | - | |

**Table 5.** *Cont.*

| Country | Sampling Year | Analytical Method | LOD/LOQ (µg kg⁻¹) | Mycotoxin | No. of Samples | Positive Sample Rate (%) | Mean (µg kg⁻¹) | Median (µg kg⁻¹) | Max (µg kg⁻¹) | Reference |
|---|---|---|---|---|---|---|---|---|---|---|
| | 2017 | | | | 47 | 4 | 153.7 | - | 614 | |
| Slovenia | 2007–2008 | HPLC–FLD | 0.2/0.6 | AFB1 | 20 | 5 | 0.2 | 0.2 | 0.2 | [43] |
| | | | 10/30 | OTA | 20 | 0 | - | - | - | |
| Slovenia | 2008–2012 | LC–MS/MS | -/0.2 | AFB1 | 80 [b] | 0 | - | - | - | [45] |
| | | | -/0.8 | aFs | 80 [b] | 0 | - | - | - | |
| | | | -/1 | OTA | 80 [b] | 2.5 | 3.9 ± 2.8 | - | 5.8 | |
| Romania | 2012–2015 | ELISA | - | aFs | 97 [c] | 45.4 | 3.85 ± 14.80 | <1.75 | 82.94 | [38] |
| | | | - | OTA | 97 [c] | 6.8 | 2.70 ± 0.43 | <2.50 | 6.72 | |
| Italy | 2009–2010 | LC–MS/MS | 0.2/1 | AFB1 | 46 | 0 | - | - | - | [44] |
| | | | 0.4/1 | OTA | 46 | 0 | - | - | - | |
| Mediterranean area | 2009–2010 | LC–MS/MS | -/0.25 | AFB1 | 65 | 15 | - | - | 66.7 | [41] |
| Poland | 2014 | UHPLC–HRMS | -/5 | aFs | 99 | 0 | - | - | - | [42] |
| | | | -/4 | OTA | 99 | 0 | - | - | - | |
| EU | 2000–2006 | - | 0.1–0.2/- | AFB1 | 3010 [d] | 7 | 0.35 | 0.20 | 109 | [4] |
| | | | 0.2–0.4/- | aFs | 3010 [d] | 7 | 0.51 | 0.40 | 117 | |
| | | | 0.01–0.5/- | OTA | 5180 [d] | 54 | 0.29 | - | 33.3 | |
| The Middle East and African countries | 2009 | HPLC–FLD | 0.3/0.8 | aFs | 32 [e] | 19 | 1 | 2 [a] | 7 | [49] |
| | | | 0.2/30.5 | OTA | 1 | 0 | - | - | - | |
| Global | 2008–2017 | HPLC<br>LC–MS/MS<br>ELISA | 0.2–2.7/-<br>0.2–5/-<br>1–3/- | AFB1 | 2210 | 10 | - | 1 | 161 | [48] |
| | | HPLC<br>LC–MS/MS<br>ELISA | 0.06–2/-<br>0.2–100/-<br>0.2–2/- | OTA | 1973 | 9 | - | 3 | 364 | |

[a] Positive samples. [b] Wheat and wheat-processed foods. [c] Cereal, processed cereal, and cereal-based food. [d] Cereals other than corn. [e] Wheat/wheat bran.

The low rate of OTA-positive corn and wheat samples is comparable to the incidence in other surveys, where it was <10% [38,39,43,45,48,49,52,54]. However, Kos et al. [35] reported an incidence of 25% and 18% in corn from Serbia in 2012 and 2015, respectively, while Marin et al. [4] reported 54% of cereal samples to be positive in the EU [4], and Lee and Ryu [60] reported a global incidence of 29% between 2006 and 2016. Nevertheless, the concentrations determined in our study are much higher than those reported in all other studies.

The occurrence of mycotoxins in cereals produced in Albania, especially aflatoxin contamination, is associated with other food commodities, such as dairy products, showing mycotoxin contamination [61]. The survey of mycotoxin contamination in the country is more and more relevant due to climate change and the country's accession to the EU.

## 3. Conclusions

In this study, the presence of AF and OTA was determined in 125 samples from two seasons, providing the first insight into their occurrence in Albanian grains by contributing to our knowledge of the mycotoxin contamination problem in southern Europe.

The incidence of four aflatoxins in the corn samples was higher than in wheat. When compared to publications of the same period from this region, it is comparable to the incidence reported in Serbia but higher than that reported in other countries. Additionally, the concentrations were significantly higher than elsewhere. The incidence of AFB1 in wheat was similar to the incidence in the region, but its concentrations were lower.

The incidence of OTA in corn and wheat was rather low, comparable to the incidence reported in other countries, except for in a few studies, where a higher incidence was reported.

However, the main concern is the high rate of results exceeding the maximum permitted levels, which can pose a significant health risk for consumers. As mentioned, regular and systematic control of the presence of aFs in grain commodities and actions for their reduction are important. The significant difference between the data from two harvesting years, 2014 and 2015, indicates that further surveys based on harvesting years need to be conducted to adequately characterize the occurrence of aFs in cereals in Albania.

## 4. Materials and Methods

### 4.1. Sample Collection

Wheat and corn grains were sampled during harvesting seasons from five main agriculture regions: Fieri, Lushnja, Korça, Elbasan, and Kruja. Grain sampling was conducted in June–July for two harvesting years, 2014 and 2015. Corn sampling was accomplished in October for the two harvesting years. The samples were taken from the field site from small farms and warehouses. At the time of sampling or during grain growth, the weather conditions were not recorded. The Commission Regulation (EC) No. 401/2006 [62] was followed in terms of the sampling procedure to ensure representative samples. During the two harvesting seasons, 2014 and 2015, 71 wheat samples and 45 corn samples were collected. As part of this study, 35 wheat and 31 corn samples were collected in 2014, and 36 wheat and 14 corn samples were collected in 2015. In 2015, consent to collect corn samples was obtained from only 14 farms. In addition, seven barley samples and two rye samples were collected in the Korça and Fieri regions (in 2014, five barley and two rye samples; in 2015, two barley samples).

### 4.2. Standards and Chemicals

A mixed aflatoxin standard solution in acetonitrile (AFB1, AFB2, AFG1, and AFG2) was purchased from Romer Labs (Tulln, Austria). Stock standard consisting of 2 μg $mL^{-1}$ (AFB1 and AFG1) and 0.5 μg $mL^{-1}$ (AFB2 and AFG2) and mixed working standard solutions were stored in amber glass vials at −20 °C. Acetonitrile, acetic acid, and methanol of HPLC grade from Sigma-Aldrich (Steinheim, Germany) and ammonium acetate of p.a. grade by Merck (Darmstadt, Germany) were purchased. The Milli-Q system (Millipore, Bedford, MA, USA) was employed for deionized water production.

### 4.3. Sample Preparation

For the simultaneous determination of the four aFs, a procedure described in detail by Topi et al. [63] was employed. The procedure consisted of mycotoxin extraction from the ground cereal samples and LC–MS/MS, according to Rasmussen et al. [64], Lattanzio et al. [65], and Schenzel et al. [66]. The grains were ground to a particle size of 1 mm using a Retsch ZM 100 laboratory mill (Haan, Germany). A 10 g sample size was shaken with 100 mL of a mixture of acetonitrile and deionized water (84 + 16) for 1 h using an IKA HS 501 digital linear shaker (IKA Labortechnik, Staufen, Germany). An aliquot (4 mL) of the filtered extract was evaporated to dryness using the Syncore Polyvap system (Buchi, Flawil, Switzerland). When the mycotoxin concentration was above the calibration range, the filtered extracts were diluted for further work. The dry residue was reconstituted in a 0.5 mL mixture of deionized $H_2O$ and MeOH (20 + 80). Finally, 10 μL of the solution was injected into the UPLC–MS/MS "Acquity UHPLC Class System" connected to a quadrupole mass spectrometer (Xevo TQ MS) equipped with an electrospray ionization interface (ESI) and MassLynx software for data acquisition and processing (Waters, Milford, MA, USA). The vials were stored at 10 °C in the autosampler. For matrix-matched calibration, an appropriate amount of standard solution was added to a 4 mL aliquot of the filtered extract and prepared along with the sample.

### 4.4. LC–MS/MS Operation

Chromatographic separation was performed on a Zorbax Eclipse Plus C18 Rapid Resolution HD column (2.1 × 100 mm, 1.8 μm; Agilent). Aflatoxin separation was performed using mixture A (deionized water) and B (methanol) in a 60:40 ratio isocratic condition. For OTA analysis, an elution described elsewhere [63] was used. The mobile phase flow rate was fixed at 0.3 mL $min^{-1}$, and the column temperature at 40 °C. MS/MS analysis was performed in MRM (multiple reaction monitoring) mode. The specific MS/MS parameters (retention time, ionization mode, and monitored transitions) associated with specific mycotoxins are shown in Table 6. The LOD and LOQ for the single aFs were 0.6 and 2.0 μg kg$^{-1}$, respectively, while for OTA they were 1.5 and 5.0 μg kg$^{-1}$, respectively.

**Table 6.** Ionization mode, retention times, and monitored aflatoxin and ochratoxin A transitions.

| Mycotoxin | Mode of Ionization | Retention Time (min) | Ion Precursor (*m*/*z*) | Quantifier Ion (*m*/*z*) | Qualifier Ion (*m*/*z*) |
|---|---|---|---|---|---|
| AFB1 | ESI+ | 5.25 | 313.2 | 213.2 | 241.1 |
| AFB2 | ESI+ | 4.75 | 215.2 | 243.2 | 259.1 |
| AFG1 | ESI+ | 2.92 | 329.1 | 214.8 | 199.9 |
| AFG2 | ESI+ | 5.33 | 331.1 | 189.0 | 285.1 |
| OTA | ESI+ | 11.10 | 404.2 | 221.0 | 239.0 |

**Author Contributions:** Conceptualization, D.T., G.T.-K. and B.J.-S.; methodology, G.T.-K.; validation, G.T.-K. and J.B.; formal analysis, D.T.; investigation, D.T.; resources, G.T.-K.; data curation, D.T.; writing—original draft preparation, D.T.; writing—review and editing, J.B., G.T.-K. and J.B.; visualization, G.T.-K.; supervision, G.T.-K.; project administration, G.T.-K.; funding acquisition, B.J.-S. All authors have read and agreed to the published version of the manuscript.

**Funding:** The study was supported by the Slovenian Research Agency grant P4-0092 and the Erasmus Mundus program of the European Union. The APC was funded by Slovenian Research Agency.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** The data presented in this study are available on request from the corresponding author.

**Acknowledgments:** The authors thank Karin Šrimpf, Irena Ardalič, and Katarina Pavšič-Vrtač for performing chemical analyses.

**Conflicts of Interest:** The authors declare no conflict of interest. The funders had no role in the study's design; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

## References

1. Hussein, H.S.; Brasel, J.M. Toxicity, metabolism, and impact of mycotoxins on the humans and animals. *Toxicology* **2001**, *167*, 101–134. [CrossRef] [PubMed]
2. Bräse, S.; Encinas, A.; Keck, J.; Nising, C.F. Chemistry and biology of mycotoxins and related fungal metabolites. *Chem. Rev.* **2009**, *109*, 3903–3990. [PubMed]
3. Bhat, R.; Rai, R.V.; Karim, A.A. Mycotoxins in food and fed: Present status and future concerns. *Compr. Rev. Food Sci. Food Saf.* **2010**, *9*, 57–81. [CrossRef]
4. Marin, S.; Ramos, A.J.; Cano-Sancho, G.; Sanchis, V. Mycotoxins: Occurrence, toxicology, and exposure assessment. *Food Chem. Toxicol.* **2013**, *60*, 218–237. [CrossRef]
5. Steinberg, P. A food toxicological contemplation of mycotoxins. *Ernaehrungs Umsch. Int.* **2013**, *60*, 146–151.
6. Neme, K.; Mohammed, A. Mycotoxin occurrence in grains and the role of postharvest management as a mitigation strategy. A review. *Food Control* **2017**, *78*, 412–425. [CrossRef]
7. Alshannaq, A.; Yu, J.-H. Occurrence, toxicity, and analysis of major mycotoxins in food. *Int. J. Environ. Res. Public Health* **2017**, *14*, 632. [CrossRef]
8. Agriopoulou, S.; Stamatelopoulou, E.; Varzakas, T. Advances in occurrence, importance, and mycotoxin control strategies: Prevention and detoxification in foods. *Foods* **2020**, *9*, 137. [CrossRef] [PubMed]
9. Arce-López, B.; Lizarraga, E.; Vettorazzi, A.; González-Peñas, E. Human biomonitoring of mycotoxins in blood, plasma, and serum in recent years: A review. *Toxins* **2020**, *12*, 147. [CrossRef]
10. Perrone, G.; Ferrara, M.; Medina, A.; Pascale, M.; Magan, N. Toxigenic fungi and mycotoxins in a climate change scenario: Ecology, genomics, distribution, prediction, and prevention of the risk. *Microorganisms* **2020**, *8*, 1496. [CrossRef]
11. Zhang, X.; Li, G.; Wu, D.; Liu, J.; Wu, Y. Recent advances on emerging nanomaterials for controlling the mycotoxin contamination: From detection to elimination. *Food Front.* **2020**, *1*, 360–381. [CrossRef]
12. Wu, F. Global impacts of aflatoxin in maize: Trade and human health. *World Mycotoxin J.* **2015**, *8*, 137–142. [CrossRef]

13. Ferrigo, D.; Raiola, A.; Causin, R. *Fusarium* toxins in cereals: Occurrence, legislation, factors promoting the appearance and their management. *Molecules* **2016**, *21*, 627. [CrossRef] [PubMed]
14. Pitt, J.I.; Taniwaki, M.H.; Cole, M.B. Mycotoxin production in major crops as influenced by growing, harvesting, storage, and processing, with emphasis on the achievement of Food Safety Objectives. *Food Control* **2013**, *32*, 205–215. [CrossRef]
15. Perrone, G.; Gallo, A. Aspergillus species and their associated mycotoxins. In *Mycotoxigenic Fungi. Methods and Protocols*; Moretti, A., Susca, A., Eds.; Methods in Molecular Biology; Humana Press: New York, NY, USA, 2017; Volume 1542, pp. 33–49.
16. Udovicki, B.; Audenaert, K.; De Saeger, S.; Rajkovic, A. Overview on the mycotoxins incidence in Serbia in the period 2004–2016. *Toxins* **2018**, *10*, 279. [CrossRef]
17. Bennett, J.W.; Klich, M. Mycotoxins. *Clin. Microbiol. Rev.* **2003**, *16*, 497–516. [CrossRef]
18. Pereira, V.L.; Fernandes, J.O.; Cunha, S.C. Mycotoxins in cereals and related foodstuffs: A review on occurrence and recent methods of analysis. *Trends Food Sci. Technol.* **2014**, *36*, 96–136. [CrossRef]
19. Eskola, M.; Kos, G.; Elliott, C.T.; Hajslova, J.; Mayar, S.; Krska, R. Worldwide contamination of food crops with mycotoxins: Validity of the widely cited 'FAO estimate' of 25. *Crit. Rev. Food Sci. Nutr.* **2020**, *60*, 2773–2789. [CrossRef]
20. Kępińska-Pacelik, J.; Biel, W. Alimentary risk of mycotoxins for humans and animals. *Toxins* **2021**, *13*, 822. [CrossRef]
21. Wu, F.; Groopman, J.D.; Pestka, J.J. Public health impacts of foodborne mycotoxins. *Annu. Rev. Food Sci. Technol.* **2014**, *5*, 351–372. [CrossRef]
22. European Commission. Commission Regulation (EU) No 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**, *L 119*, 103–157. Available online: https://eur-lex.europa.eu/eli/reg/2023/915/oj (accessed on 5 August 2023).
23. Filazi, A.; Sireli, U.T. Occurrences of aflatoxins in food. In *Aflatoxins-Recent Advances and Prospects*; Razzaghi-Abyaneh, M., Ed.; InTech: London, UK, 2013; pp. 143–170. [CrossRef]
24. EFSA Panel on Contaminants in the Food Chain. Scientific opinion—Risk assessment of aflatoxins in food. *EFSA J.* **2020**, *18*, 6040. [CrossRef]
25. Rushing, B.R.; Selim, M.I. Aflatoxin B1: A review on metabolism, toxicity, occurrence in food, occupational exposure, and detoxification methods. *Food Chem. Toxicol.* **2019**, *124*, 81–100. [CrossRef]
26. IARC (International Agency for Research on Cancer). Chemical Agents and Related Occupations. A Review of Human Carcinogens. IARC Monographs on the Evaluation of Carcinogenic Risks to Humans. 2012. Available online: http://monographs.iarc.fr/ENG/Monographs/vol100F/ (accessed on 31 July 2023).
27. Liu, Y.; Wu, F. Global burden of aflatoxin-induced hepatocellular carcinoma: A risk assessment. *Environ. Health Persp.* **2010**, *118*, 818–824. [CrossRef]
28. Ráduly, Z.; Szabó, L.; Madar, A.; Pócsi, I.; Csernoch, L. Toxicological and medical aspects of *Aspergillus*-derived mycotoxins entering the Feed and Food Chain. *Front. Microbiol.* **2020**, *10*, 2908. [CrossRef]
29. Zain, M.E. Impact of mycotoxins on humans and animals. *J. Saudi Chem. Soc.* **2011**, *15*, 129–144. [CrossRef]
30. Perrone, G.; Susca, A. Penicillium species and their associated mycotoxins. In *Mycotoxigenic Fungi. Methods and Protocols*; Moretti, A., Susca, A., Eds.; Methods in Molecular Biology; Humana Press: New York, NY, USA, 2017; Volume 1542, pp. 107–119.
31. Cotty, P.J.; Jaime-Garcia, R. Influences of climate on aflatoxin producing fungi and aflatoxin contamination. *Int. J. Food Microbiol.* **2007**, *119*, 109–115. [CrossRef]
32. Griessler, K.; Rodrigues, I.; Handl, J.; Hofstetter, U. The occurrence of mycotoxins in Southern Europe. *World Mycotoxin J.* **2010**, *3*, 301–309. [CrossRef]
33. Kos, J.; Škrinjar, M.; Mandić, A.I.; Mišan, A.Č.; Bursić, V.P.; Šarić, B.; Janić-Hajnal, E. Presence of aflatoxins in cereals from Serbia. *Food Feed Res.* **2014**, *41*, 31–38. [CrossRef]
34. Kos, J.; Janić Hajnal, E.; Šarić, B.; Jovanov, P.; Mandić, A.; Đuragić, O.; Kokić, B. Aflatoxins in maize harvested in the Republic of Serbia over the period 2012–2016. *Food Addit. Contam. B* **2018**, *11*, 246–255. [CrossRef]
35. Kos, J.; Janic Hajnal, E.; Malachova, A.; Steiner, D.; Stranska, M.; Krska, R.; Poschmaier, B.; Sulyok, M. Mycotoxins in maize harvested in the Republic of Serbia in the period 2012–2015. Part 1: Regulated mycotoxins and its derivatives. *Food Chem.* **2020**, *312*, 126034. [CrossRef]
36. de Rijk, T.C.; van Egmond, H.P.; van der Fels-Klerx, H.J.; Herbes, R.; de Nijs, M.; Samson, R.; Slate, A.B.; van der Spiegel, M. A study of the 2013 Western European issue of aflatoxin contamination of maize from the Balkan area. *World Mycotoxin J.* **2015**, *8*, 641–651. [CrossRef]
37. Battilani, P.; Toscano, P.; Van Der Fels-Klerx, H.J.; Moretti, A.; Leggieri, M.C.; Brera, C. Aflatoxin B1 contamination in maize in Europe increases due to climate change. *Sci. Rep.* **2016**, *6*, 24328. [CrossRef] [PubMed]
38. Gagiu, V.; Mateescu, E.; Armeanu, I.; Dobre, A.A.; Smeu, I.; Cucu, M.E.; Oprea, O.A.; Iorga, E.; Belc, N. Post-harvest contamination with mycotoxins in the context of the geographic and agroclimatic conditions in Romania. *Toxins* **2018**, *10*, 533. [CrossRef]
39. Kovač, M.; Bulaić, M.; Nevistić, A.; Rot, T.; Babić, J.; Panjičko, M.; Kovač, T.; Šarkanj, B. Regulated Mycotoxin Occurrence and Co-Occurrence in Croatian Cereals. *Toxins* **2022**, *14*, 112. [CrossRef] [PubMed]
40. Binder, E.M.; Tan, L.M.; Chin, L.J.; Handl, J.; Richard, J. Worldwide occurrence of mycotoxins in commodities, feeds, and feed ingredients. *Anim. Feed Sci. Techol.* **2007**, *137*, 265–282. [CrossRef]
41. Serrano, A.B.; Font, G.; Ruiz, M.J.; Ferrer, E. Co-occurrence and risk assessment of mycotoxins in food and diet from Mediterranean area. *Food Chem.* **2012**, *135*, 423–429. [CrossRef]

42. Bryła, M.; Waśkiewicz, A.; Podolask, G.; Szymczyk, K.; Jędrzejczak, R.; Damaziak, K.; Sułek, A. Occurrence of 26 mycotoxins in the grain of cereals cultivated in Poland. *Toxins* **2016**, *8*, 160. [CrossRef]
43. Jakovac-Strajn, B.; Pavšič-Vrtač, K.; Ujčič-Vrhovnik, I.; Vengušt, A.; Tavčar-Kalcher, G. Microbiological and mycotoxicological contamination in Slovenian primary grain production. *Toxicol. Environ. Chem.* **2010**, *92*, 1551–1563. [CrossRef]
44. Alkadri, D.; Rubert, J.; Prodi, A.; Pisi, A.; Manes, J.; Soler, C. Natural co-occurrence of mycotoxins in wheat grains from Italy and Syria. *Food Chem.* **2014**, *157*, 111–118. [CrossRef]
45. Kirinčič, S.; Škrjanc, B.; Kos, N.; Kozolc, B.; Pirnat, N.; Tavčar-Kalcher, G. Mycotoxins in cereals and cereal products in Slovenia—Official control of foods in the years 2008–2012. *Food Control* **2015**, *50*, 157–165. [CrossRef]
46. Pleadin, J.; Vulić, A.; Perši, N.; Škrivanko, M.; Capek, B.; Cvetnić, Ž. Aflatoxin B1 occurrence in maize sampled from Croatian farms and feed factories during 2013. *Food Control* **2014**, *40*, 286–291. [CrossRef]
47. Pleadin, J.; Vulić, A.; Perši, N.; Škrivanko, M.; Capek, B.; Cvetnić, Ž. Annual and regional variations of aflatoxin B1 levels seen in grains and feed coming from Croatian dairy farms over a 5-year period. *Food Control* **2015**, *47*, 221–225. [CrossRef]
48. Gruber-Dorninger, C.; Jenkins, T.; Schatzmayr, G. Global Mycotoxin Occurrence in Feed: A Ten-Year Survey. *Toxins* **2019**, *11*, 375. [CrossRef] [PubMed]
49. Rodrigues, I.; Handl, J.; Binder, E.M. Mycotoxin occurrence in commodities, feeds and feed ingredients sourced in the Middle East and Africa. *Food Addit. Contam. B* **2011**, *4*, 168–179. [CrossRef]
50. Aasa, A.O.; Fru, F.F.; Adelusi, O.A.; Oyeyinka, S.A.; Njobeh, P.B. A review of toxigenic fungi and mycotoxins in feeds and food commodities in West Africa. *World Mycotoxin J.* **2023**, *16*, 33–47. [CrossRef]
51. Ayeni, K.I.; Sulyok, M.; Krska, R.; Warth, B. Mycotoxins in complementary foods consumed by infants and young children within the first 18 months of life. *Food Control* **2023**, *144*, 109328. [CrossRef]
52. Abdallah, M.F.; Girgin, G.; Bydar, T.; Krska, R.; Sulyok, M. Occurrence of multiple mycotoxins and other fungal metabolites in animal feed and maizecorn samples from Egypt using LC-MS/MS. *J. Sci. Food Agric.* **2017**, *97*, 4419–4428. [CrossRef]
53. Dong, T.; Fan, L.; Liang, J.; Wang, L.; Yuan, X.; Wang, Y.; Zhao, S. Risk assessment of mycotoxins in stored maize: Case study of Shandong, China. *World Mycotoxin J.* **2020**, *13*, 313–320. [CrossRef]
54. Jiang, D.; Li, F.; Zheng, F.; Zhou, J.; Li, L.; Shen, F.; Chen, J.; Li, E. Occurrence and dietary exposure assessment of multiple mycotoxins in corn-based food products from Shandong, China. *Food Addit. Contam. B* **2019**, *12*, 10–17. [CrossRef]
55. Sun, L.; Li, R.; Tai, B.; Hussain, S.; Wang, G.; Liu, X.; Xing, F. Current status of major mycotoxins contamination in food and feed in Asia—A review. *Food Sci. Techol.* **2023**, *3*, 231–244. [CrossRef]
56. Janić Hajnal, E.; Kos, J.; Krulj, J.; Krstović, S.; Jajić, I.; Pezo, L.; Šarić, B.; Nedeljković, N. Aflatoxins contamination of maize in Serbia: The impact of weather conditions in 2015. *Food Addit. Contam. Part A* **2017**, *34*, 1999–2010. [CrossRef]
57. Soleimany, F.; Jinap, S.; Abas, F. Determination of mycotoxins in cereals by liquid chromatography tandem mass spectrometry. *Food Chem.* **2012**, *130*, 1055–1060. [CrossRef]
58. FAOSTAT. Food and Agriculture Data. Crops. Production. 2023. Available online: http://www.fao.org/faostat/en/#data/QC (accessed on 21 June 2023).
59. INSTAT. Institute of Statistics of Albania Area of Field Crop. 2023. Available online: http://www.instat.gov.al/al/temat/bujq%C3%ABsia-dhe-peshkimi/bujq%C3%ABsia/#tab2. (accessed on 21 June 2023).
60. Lee, H.J.; Ryu, D. Worldwide occurrence of mycotoxins in cereals and cereal-derived food products: Public health perspectives of their co-occurrence. *J. Agric. Food Chem.* **2017**, *65*, 7034–7051. [CrossRef]
61. Topi, D.; Spahiu, J.; Rexhepi, A.; Marku, N. Two-year survey of aflatoxin M1 in milk marketed in Albania and human exposure assessment. *Food Control* **2022**, *136*, 108831. [CrossRef]
62. European Commission. Commission Regulation (EC), No 401/2006 of February 23, 2006, laid down the sampling methods and analysis for the official control of mycotoxins levels in foodstuffs. *Off. J. Eur. Union* **2006**, *L 70*, 12–34.
63. Topi, D.; Babič, J.; Pavšič-Vrtač, K.; Tavčar-Kalcher, G.; Jakovac-Strajn, B. Incidence of *Fusarium* mycotoxins in wheat and maize from Albania. *Molecules* **2021**, *26*, 172. [CrossRef]
64. Rasmussen, R.R.; Storm, I.M.L.D.; Rasmussen, P.H.; Smedsgaard, J.; Nielsen, K.F. Multi-mycotoxin analysis of silage by LC-MS/MS. *Anal. Bioanal. Chem.* **2010**, *397*, 765–776. [CrossRef] [PubMed]
65. Lattanzio, V.M.T.; Della Gatta, S.; Suman, M.; Visconti, A. Development and in-house validation of a robust and sensitive solid-phase extraction liquid chromatography/tandem mass spectrometry method for the quantitative determination of aflatoxins B1, B2, G1, G2, ochratoxin A, deoxynivalenol, zearalenone, T-2, and HT-2 toxins in cereal-based foods. *Rapid Commun. Mass Spectrom.* **2011**, *25*, 1869–1880.
66. Schenzel, J.; Forrer, H.R.; Vogelgsang, S.; Bucheli, T.D. Development, validation and application of a multi-mycotoxin method for the analysis of whole wheat plants. *Mycotoxin Res.* **2012**, *28*, 135–147. [CrossRef]