Fusarium toxin contents of maize and maize products purchased in the years 2000 and 2001 in Germany

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Abstract

Samples (n=106) of maize and maize products were analysed for 13 trichothecene toxins and zearalenone (ZON). All 14 toxins examined were detected, although with varying frequency. Co-occurrence of two or more toxins was observed in 96% of samples. The toxins of the scirpenol group scirpentriol, 15-monoacetoxyscirpenol and diacetoxyscirpenol were detected in 14, 27 and 3% of the samples analysed, the toxins of the T-2 group T-2 toxin, HT-2 toxin, T-2 triol und T-2 tetraol were found in 33, 66, 2 and 7%. Toxin content was higher in feeds than in foods (semolina and flour). In food samples, the German regulatory level for DON (500 µg/kg) was not exceeded, three samples of maize flour contained ZON above the regulatory level (50 µg/kg).

Keywords: *Fusarium* toxins, trichothecenes, scirpenolgroup, T-2 group, zearalenone, maize, food, feed

Introduction

Maize and maize products often contain *Fusarium* toxins (1). Data exist about incidences and levels of deoxynivalenol (DON), 3- and 15- acetylDON (3- and 15- ADON), nivalenol (NIV) and zearalenone (ZON) (2), other studies included the type-A trichothecenes T-2 und HT-2 toxin (HT-2, T-2) (3). So far little is known about the occurrence of a broad spectrum of *Fusarium* toxins including toxins of the scirpenolgroup and neosolaniol (NEO) in maize based food and feed. Therefore a total of 106 maize samples, including whole maize plant, silage, maize gluten, germ and other maize products (bran, oil meal, screenings), as well as of maize semolina and flour for food use were analysed for the type-A trichothecenes scirpentriol (SCIRP), 15-monoacetoxyscirpenol (MAS), diacetoxyscirpenol (DAS), HT-2, T-2, T-2 triol, T-2 tetraol und NEO, for the type-B trichothecenes DON, 15-ADON, 3-ADON, NIV, fusarenon-X (FUS-X), as well as for ZON.

Material and Methods

Samples, taken at random, were provided by firms and public authorities, purchased at food stores or collected by University Hohenheim staff during 2000 and 2001.

Samples were dried and milled if necessary, and stored at -20 °C prior to analysis. Trichothecene analysis was carried out as described in detail previously (4, 5). In brief, extraction was performed with a mixture of acetonitrile and water followed by liquid/liquid extraction with hexane. Clean up was by solid phase extraction using a florisil and a cation exchange cartridge. Derivatization was carried out with trifluoroaceticanhydride and measurement was by GC-MS using a Magnum Ion Trap system in the chemical ionisation mode with isobutane as reactant gas. Detection limits ranged between 3 and 19 µg per kg.

Determination of ZON was carried out as described elsewhere (5, 6). In brief, after extraction with a mixture of acetonitrile and water, sample clean up was carried out using an immunoaffinity column. Identification and quantification of ZON was carried out by HPLC with fluorescence detection, UV/ photodiodearray detection was used to contol toxin identity. The detection limit for ZEA was 1 µg/kg for fluorescence detection.

Results and Discussion

In only three out of a total of 106 samples, the concentration of each of the 14 *Fusarium* toxins under study was below the detection level, in one other sample only one toxin was found. All other samples contained at least two

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toxins, with 2, 3, 4, 5, 6, 7, 8, 9 10, 11 and 12 toxins occurring together in 10, 8, 12, 22, 18, 15, 9, 2, 4, 1 and 1 samples, respectively. Overall, each of the 14 toxins examined was detected in one or more samples. Based on the results of the present study, a multitoxin contamination of maize and maize based products must be taken into consideration, therefore regulations - in addition to the existing advisory levels for DON and ZON - might be desirable.

DON and ZON were detected with the highest incidence, while the highest toxin levels were found for DON and NIV (see Table 1). The incidence of 15-ADON was higher than that of 3-ADON. This is consistent with findings of Lew *et al.* (3) who analysed Austrian maize samples.

The toxins of the scirpenol group, namely SCIRP, MAS and DAS were detected in 14, 27 and 3% of the samples analysed. Toxins of the T-2 group, namely T-2, HT-2, T-2 triol und T-2 tetraol, were found in 33, 66, 2 and 7% of all 106 samples. Maximum levels for toxins of these two groups were found for SCIRP (815 µg/kg) and HT-2 (1307 µg/kg), respecttively. The explicetely higher toxicity of these trichothecenes compared to DON and NIV (7) must be taken into account.

The toxin concentrations in feeds were clearly higher that that found in foods (semolina and flour) (see Table 1).

Concerning food samples DON was detected in 86 and 87% of samples of semolina and maize flour respectively. ZON was found in 79% and 87% of semolina and corn flour. In an earlier study we found that in wheat flour the incidence of DON was at 98% and that of ZON was at 38% (8). It must be noted that the samples of wheat flour were collected in 1999, while the maize samples analysed in the present study were obtained in 2000 and 2001. A possible effect of the year of harvest must be taken into account. The median contamination level in DON-positive samples of maize flour and semolina was at 80 µg/kg and 117 µg/kg, respectively, while Schollenberger *et al.* (8) reported a corresponding value for wheat flour of 199 µg/kg. Median values for ZON found in this study were at 5 and 22 µg/kg in semolina and maize flour, respectively, whereas that of wheat flour was at 3 µg/kg.

The regulatory level of the German government (9) for DON of 500 µg/kg in grain and grain products for food use was not exceeded in any sample of foodstuff, the regulatory level of ZON of 50 µg/kg was exceeded in three samples of maize flour.

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Table 1. *Fusarium* toxins in maize and maize based products

Product	DON	15-ADON	3-ADON	NIV	FUS-X	SCIRP	MAS	DAS	T-2 tetraol	T-2 triol	HT-2	T-2	NEO	ZON
Kernels (n=24)														
% of pos. samples	100	100	46	83	17	8	21	4	0	0	71	25	0	96
Range of pos. samples (µg/kg)	14 - 4700	28 - 800	14 - 137	21 - 1388	16 - 94	50 - 97	5 - 51	21			5 - 41	6 - 8		2 - 310
Mean of pos. samples (µg/kg)	1101	175	43	406	55	74	37				18	7		42
Median of pos. samples (µg/kg)	880	134	22	219	56	74	43				15	7		21
Whole plant (n = 9)														
% of pos. samples	100	100	11	100	0	67	67	0	33	11	89	67	0	100
Range of pos. samples(µg/kg)	215 - 728	40 - 484	51	116 - 5910		32 - 815	9 - 76		20 - 703	68	7 - 1307	6 - 323		7 - 492
Mean of pos. samples (µg/kg)	540	136		1087		184	26		267		183	62		131
Median of pos. samples (µg/kg)	603	90		500		56	14		79		24	11		40
Silage (n = 18)														
% of pos. samples	100	94	0	89	0	11	44	6	6	0	89	0	0	100
Range of pos. samples (µg/kg)	323 - 3510	20 - 347		113 - 2750		64 - 110	14 - 51	64	79		5 - 47			2 - 1593
Mean of pos. samples (µg/kg)	1426	84		1049		87	32				21			116
Median of pos. samples (µg/kg)	1297	57		723		87	28				20			10
Maize gluten and gluten feed (n = 11)														
% of pos. samples	100	82	46	36	0	0	0	0	0	0	82	64	0	82
Range of pos. samples (µg/kg)	86 - 2455	112 - 565	14 - 78	82 - 268							17 - 90´	6 - 40		3 - 350
Mean of pos. samples (µg/kg)	1166	325	40	144							42	17		60
Median of pos. samples (µg/kg)	1200	378	37	112							33	8		15
Other feed components (n = 15)														
% of pos. samples	100	93	87	100	47	33	67	7	20	7	87	67	7	100
Range of pos. samples (µg/kg)	634 - 6682	165 - 1780	14 - 114	21 - 2050	29 - 494	12 - 38	5 - 39	21	11 - 56	8	5 - 99	7 - 70	9	57 - 1362
Mean of pos. samples (µg/kg)	1990	573	50	374	124	20	14		39		34	21		328
Median of pos. samples (µg/kg)	1500	441	38	115	47	12	11		50		19	14		151
Semolina (n = 14)														
% of pos. samples	86	64	14	7	0	0	0	0	0	0	21	14	0	79
Range of pos. samples (µg/kg)	15 - 229	15 - 45	15 - 17	36							5-26	4 -8		2 - 42
Mean of pos. samples (µg/kg)	93	24	16								12	6		12
Median of pos. samples (µg/kg)	80	22	16								6	6		5
Flour (n = 15)														
% of pos. samples	87	67	13	27	13	0	0	0	0	0	27	27	0	87
Range of pos. samples (µg/kg)	20 - 452	11 - 73	14 - 25	22 - 56	29						5	6 - 11		2 - 136
Mean of pos. samples (µg/kg)	160	40	20	39								7		40
Median of pos. samples (µg/kg)	117	35	20	39								6		22
Total samples (n = 106)														
% of pos. samples	96	87	32	65	12	14	27	3	7	2	66	33	1	93
Range of pos. samples (µg/kg)	14 - 6682	11 - 1780	14 - 137	21 - 5910	16 - 494	12 - 815	5 - 76	21 - 64	11 - 703	8 - 68	5 - 1307	4 - 323	9	2 - 1593
Mean of pos. samples (µg/kg)	1008	200	42	595	88	102	25	35	143	38	43	23		106
Median of pos. samples (µg/kg)	643	100	35	312	47	46	22	21	56	38	19	8		22