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Mycotoxin Contamination of Bush Mango, Cashew Nuts, Okra, Sesame and Sorghum Marketed in Nasarawa State, Nigeria (英文原文)

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Abstract: Food crops (bush mango (*n*=12), roasted cashew nut (*n*=12), dried okra (*n*=12), sesame (*n*=35) and sorghum (*n*=36)) sold in markets in Nasarawa state, Nigeria, were analyzed using a LC-MS/MS mycotoxin method. The hepatocarcinogenic aflatoxin B_1 was detected in 42%, 25% and 19% of bush mango, dried okra and sorghum samples at mean concentrations of 19.2 μg/kg, 8.27 μg/kg and 4.75 μg/kg, respectively, while fumonisin B_1 contaminated 9% of the sesame (mean: 12.5 μg/kg) and 47% of the sorghum (mean: 461 μg/kg) samples. At least 19% of the sorghum samples were co- contaminated with aflatoxin B_1 and fumonisin B_1. The nephrotoxic ochratoxin A was detected in bush mango, sorghum and, for the first time to the best of our knowledge, in dried okra. These vended food crops in the local markets are therefore prone to mycotoxin contamination, which may pose a health threat to consumers, and require intentional mitigation efforts.

Key words: Aflatoxin B_1; cereals; nuts; oil seeds; food safety; mycotoxins; public health; vegetables

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1. INTRODUCTION

The Food and Agricultural Organization of the United Nations reports that the average dietary energy supply worldwide increased from 2 753 kcal/cap/day in 2005 to 2 904 kcal/cap/day in 2016[1], suggesting a significant increase in food availability globally. Despite the upsurge in food availability, a good number of people in sub-Saharan Africa (SSA) still suffer from malnutrition, whose underlying factors are complex but include mycotoxin contamination of food crops and poverty[2-3]. Although these factors are not restricted to a particular region, the burden of the mycotoxin menace is higher in SSA countries such as Nigeria where pre- and post-harvest practices for crop handling and management are poorly developed and climatic conditions encourage frequent mycotoxin contamination of foods[4–11]. Depending on the quantity ingested through contaminated foods, continuous daily exposure to mycotoxins could lead to an array of deleterious effects in humans. These effects could range from acute (e.g., nausea, vomiting) to chronic (e.g., cancers) or even deaths [2,12].

In Nigeria, food crops such as bush mango (*Irvingia gabonensis*, locally known as *dika* nut or *ogbono*), cashew (*Anacardium occidentale* L.), okra (*Abelmoschus esculentus* (L.) Moench), sorghum (*Sorghum bicolor* L.) and sesame (*Sesamum indicum* L.) are staples consumed in most households and are available in various markets. The dry large seeds of bush mango extracted from the fruit (a drupe) and the whole fruits of fresh okra (a capsule) or its dried form ground into powder are made into thick slimy *ogbono* and *okro* soups, respectively[9,13–15]. Sorghum is a grain while sesame is a small oil seed, both widely cultivated in different parts of Nigeria. In Nigeria, sorghum is primarily processed into traditional foods such as *masa*, *ogi*, *tuwo* and *waina*[4,16], whereas sesame is often preferred for its oils and also serves as spice in meals [16–18]. Both sorghum and sesame also serve as raw ingredients for traditionally processed beverages (e.g., *kunu*)[19-20]. The roasted nut of cashew is commonly consumed as ready-to-eat snack. Taken together, these food crops contribute substantially to the dietary intake of households in Nigeria, especially in the northern region to which Nasarawa state belongs. Thus, it is pertinent to assess the mycotoxicological safety of the foods, in order to safeguard consumer health.

Previous studies have reported on the mycotoxin contamination of sorghum and sesame[4,16,18,21], bush mango[9] and cashew nuts[22] in different parts of Nigeria. However, there is sparse data on the mycotoxin profiles of these foods vended in markets in Nasarawa state. Moreover, high levels of mycotoxin cocktails have been quantified in maize[6,23], plate ready household foods[24] and human urine[25] from Nasarawa state. Consequently, Nasarawa state could be a hotspot for mycotoxin exposure; hence, there is a need to assess diverse food sources for their contribution to mycotoxin exposure in the state. In addition, it is important to monitor foods vended in open markets and commonly consumed as staples for mycotoxin contamination with a view to providing data to inform viable interventions. This study therefore aimed to elucidate the mycotoxin contamination profiles of bush mango, cashew nut, dry okra, sorghum and sesame marketed in Nasarawa state.

2. MATERIALS AND METHODS

2.1 Sample collection

A total of 107 food crops consisting of bush mango (n=12), roasted cashew nut (n=12), dried okra (n=12), sesame (n=35) and sorghum (n=36) were purchased from major markets in Nasarawa state, Nigeria. The major markets were selected to represent the three Agricultural zones (southern zone (Lafia area), northern zone (Akwanga area) and western zone (Keffi area)) of the state. In each market, vendors of the food crops were randomly selected; for every five vendors, one was selected. From each randomly selected vendor one sample of a food crop was collected. Each sesame and sorghum sample weighed 1 kg and consisted of 3~4 subsamples drawn from various portions of the vendor's storage bins for the food, while samples of the other foods weighed approximately 300 g and comprised of four randomly selected pre-packaged sub-samples of the foods. Food samples collected were not previously stored beyond 14 days in vendors' trading stores. All food samples were immediately comminuted and stored at –20 ℃ prior to multi-mycotoxin analysis at the Institute for Bioanalytics and Agro-Metabolomics, IFA–Tulln, Austria.

2.2 Multi-mycotoxin analysis of food samples

The LC-MS/MS method described by Sulyok et al.[26] was employed to quantify the presence of more than 500 microbial metabolites including mycotoxins in the food samples.

2.2.1 Chemicals

Methanol (LC gradient grade) and glacial acetic

acid (p.a) were purchased from Merck (Darmstadt, Germany), acetonitrile (LC gradient grade) from VWR (Leuven, Belgium), and ammonium acetate (MS grade) from Sigma-Aldrich (Vienna, Austria). Mycotoxin standards were obtained from various research groups or purchased from various commercial sources as specified by Abia et al[27]. Water was purified successively by reverse osmosis with an Elga Purelab ultra-analytic system from Veolia Water (Bucks, UK).

2.2.2 Extraction of metabolites and determination of apparent recoveries

For each food sample, 5 g was extracted with 20 mL of acetonitrile/water/acetic acid 79∶20∶1, (v/v/v) in a 50 mL polypropylene tube (Sarstedt, Nümbrecht, Germany). For determination of apparent recoveries, 0.25 g samples of food were spiked with 100 μL of a multi-component stock solution, left overnight to establish equilibrium between sample and spike, and subsequently homogenized with 1 mL of acetonitrile/water/acetic acid 79∶20∶1, v/v/v. Food and spiked samples were extracted for 90 min on a GFL 3017 rotary shaker (GFL, Burgwedel, Germany), an aliquot of the extract was diluted 1:1 (v/v) with dilution solvent (acetonitrile/water/acetic acid 20∶79∶1, v/v/v) and injected into the LC-MS/MS instrument [28].

2.2.3 LC-MS/MS parameters

LC-MS/MS screening of the microbial metabolites was performed with a QTrap 5500 LCMS/MS System (Applied Biosystem, Foster City, CA, USA) equipped with TurboIonSpray electrospray ionisation (ESI) source and a 1290 Series HPLC System (Agilent, Waldbronn, Germany). Chromatographic separation was performed at 25 ℃ on a Gemini® C18-column, 150×4.6 mm i.d., 5 μm particle size, equipped with a C18 4×3 mm i.d. security guard cartridge (Phenomenex, Torrance, CA, USA). The chromatographic method, chromatographic and mass spectrometric parameters are as described by Sulyok et al[26]. ESI-MS/MS was performed in the time-scheduled multiple reaction monitoring (MRM) mode both in positive and negative polarities in two separate chromatographic runs per sample by scanning two fragmentation reactions per analyte. The MRM detection window of each analyte was set to its expected retention time ±20 and ±26 s in the positive and the negative modes, respectively.

Confirmation of positive analyte identification was obtained by the acquisition of two MRMs per analyte (with the exception of moniliformin, which exhibited only one fragment ion). This yielded 4.0 identification points according to European Commission decision 2002/657 [29]. In addition, the LC retention time and the intensity ratio of the two MRM transitions agreed with the related values of an authentic standard within 0.1 min and 30% respectively.

2.2.4 Quantification of mycotoxins

Quantification was based on linear, 1/x weighed calibration using serial dilutions of an external multi-component stock solution. Concentrations were corrected for apparent recoveries. The accuracy of the method was previously verified by participation in inter-laboratory comparison studies[26,30] including a regular scheme organized by BIPEA (Gennevilliers, France). Limits of detection and limits of quantification were determined based on the standard deviation of samples spiked at low concentration levels following the EURACHEM guide[31].

2.3 Statistical analysis

SPSS 17.0 for Windows (SPSS Inc., Chicago, IL, USA) was used to analyze mycotoxin occurrence data. Simple descriptive statistics were used for data relating to distribution of mycotoxins/metabolites in the analyzed foods. Mycotoxin concentrations in food crops were transformed using the equation $y = \mathrm{Log}_{10}$ (1+ μg/kg of mycotoxin) to create a normal distribution for comparison of toxin levels in the foods. The error level was set at 5% for all analyses performed.

3. RESULTS AND DISCUSSION

3.1 LC-MS/MS method performance and overview of microbial metabolites

The method performance characteristics for 80 fungal and bacterial metabolites in the foods from Nasarawa state are presented in Table 1. The ranges of apparent recoveries of the metabolites were 38.9~167, 23.9~602, 9.3~243, 9.2~225 and 61.0~140 for bush mango, cashew nut, dried okra, sesame and sorghum, respectively. Large deviations from 100% apparent recoveries have largely been attributed to matrix effects, whereas recoveries of the extraction step have been found to comply to performance criteria (e.g., 70%~120%) for the majority of the analytes[26]. Limits of detection ranged from 0.008~240 μg/kg. Overall, of all the mycotoxins quantified in the 107 food samples,

deoxynivalenol (incidence: 65%; range: 8.7~256 μg/kg; mean: 63.6 μg/kg) was the most frequently occurring mycotoxin while total fumonisins had the highest contamination levels (incidence: 22%; range: 3.7~2 938 μg/kg; mean: 477 μg/kg (Figure 1).

Table 1 Method performance characteristics for metabolites in commonly consumed food crops marketed in Nasarawa state, Nigeria

Metabolites	LOD[a]	LOQ[b]	Recovery (%)[c]				
			Bush mango	Cashew nut	Dried okra	White sorghum	Sesame
3-Nitropropionic acid	0.8	2.4	56.3	82.9	30.8	*nd*	*nd*
Aflatoxin B_1	0.24	0.72	38.9	96.1	31.4	64.3	50.4
Aflatoxin G_1	0.32	0.96	76.6	96.0	30.5	*nd*	*nd*
Aflatoxin M_1	0.4	1.2	73.3	102.7	41.8	*nd*	*nd*
Agroclavine	0.12	0.36	100.0	105.4	9.3	*nd*	*nd*
Alternariol	0.4	1.2	100.0	92.8	24.6	77.9	52.5
Alternariolmethylether	0.16	0.48	100.0	102.5	33.3	88.9	80.9
Altenuene	0.8	2.4	*nd*	*nd*	*nd*	100.0	100.0
Altenusin	240	–	*nd*	*nd*	*nd*	*nd*	224.6
Altersetin	1.1	3.6	100.0	186.1	243	*nd*	*nd*
Altertoxin-I	0.4	–	*nd*	*nd*	*nd*	70.4	53.7
Andrastin A	0.25	0.75	113	69.1	84.9	92.8	95.4
Ascochlorin	0.07	0.24	69.9	88.1	99.9	*nd*	*nd*
Aspercolorin	0.8	2.4	100.0	127.7	31.0	*nd*	*nd*
Aspergillicin derivative	0.5	1.5	100.0	120.6	66.9	*nd*	*nd*
Aspergillimide	0.03	0.09	100.0	50.3	7	*nd*	*nd*
Asperglaucide	0.08	0.24	100.0	107.2	103.0	*nd*	*nd*
Asperphenamate	0.04	0.12	100.0	113.8	60.4	*nd*	*nd*
Averantin	0.04	0.12	50.8	49.1	88.3	*nd*	*nd*
Averufin	0.04	0.12	50	51.6	89.2	98.4	27.4
Beauvericin	0.008	0.024	52	92.8	100.0	83.4	89.7
Bikaverin	40	–	*nd*	*nd*	*nd*	120.0	65
Brevianamid F	0.1	0.3	103.2	140.0	17.3	*nd*	*nd*
Chloramphenicol	0.03	0.09	100.0	107.0	57.3	*nd*	*nd*
Citreorosein	0.64	1.92	100.0	100.1	45.9	*nd*	*nd*
Curvularin	0.4	1.2	135	89.5	73.9	80.2	60.4
cyclo(L-Pro-L-Tyr)	0.8	2.4	83.7	51.3	11.2	*nd*	*nd*
cyclo(L-Pro-L-Val)	0.64	1.92	100.0	100.0	100.0	*nd*	*nd*
Cyclopiazonic acid	6	20	159.7	103.8	100.0	*nd*	*nd*
Cytochalasin J	2.4	8	82.9	109.4	59.9	*nd*	*nd*
Deoxynivalenol	1.6	4.8	*nd*	*nd*	*nd*	79.9	61.7
Dihydroxymellein	0.5	1.7	100.0	102.8	43.4	*nd*	*nd*
Emodin	0.056	0.168	100.0	93.5	71.0	80.8	61.5
Equisetin	0.24	0.72	167.1	189.7	144.4	140.1	73.8
Fallacinol	0.1	0.3	100.0	92.2	52.8	*nd*	*nd*
Fellutanine A	1	3.3	100.0	137.3	34.3	*nd*	*nd*
Festuclavin	0.8	2.4	*nd*	*nd*	*nd*	82.3	69.4
Fumonisin B_1	8	24	*nd*	*nd*	*nd*	81.2	88.3
Fumonisin B_2	2.4	7.2	*nd*	*nd*	*nd*	90.5	90.2
Fumonisin B_3	1.6	4.8	*nd*	*nd*	*nd*	92.8	94.4
Fumonisin B_4	1.6	4.8	*nd*	*nd*	*nd*	100.0	90.2

Continued 1

Metabolites	LOD[a]	LOQ[b]	Recovery (%)[c]				
			Bush mango	Cashew nut	Dried okra	White sorghum	Sesame
Fusarinolic acid	40	120	*nd*	*nd*	*nd*	100.0	63.6
Hydrolysed fumonisin B_1	0.16	0.48	*nd*	*nd*	*nd*	92.3	111.3
Integracin A	0.04	0.13	155.5	602	193.1	*nd*	*nd*
Integracin B	0.09	0.3	58.7	64.8	121	*nd*	*nd*
Iso-Rhodoptilometrin	0.64	1.92	100.0	105.1	66.1	*nd*	*nd*
Kojic acid	16	48	100.0	73.9	16.0	121.1	43.9
Macrosporin	0.04	0.12	100.0	96.5	68.7	83.2	54.4
Malformin A	0.2	0.6	*nd*	*nd*	*nd*	61.0	93.0
Malformin C	0.4	1.2	77.1	114.7	57.3	61.0	100.8
Methylsulochrin	0.04	0.12	100.0	92.1	64.1	*nd*	*nd*
Monactin	0.05	0.18	100.0	106.3	84.1	*nd*	*nd*
Moniliformin	1.6	4.8	100.0	76.0	39.7	88.0	83.7
Monocerin	0.4	1.2	100.0	99.9	63.8	*nd*	*nd*
Mycophenolic acid	0.2	0.67	106	97.3	84.7	*nd*	*nd*
N-Benzoyl-Phenylalanine	0.064	0.192	100.0	105.2	72.9	*nd*	*nd*
Neoechinulin A	0.8	2.4	100.0	99.7	35.7	*nd*	*nd*
Nidurufin	0.16	0.48	108.4	99.1	48.8	78.2	61.6
Nivalenol	0.75	2.5	44.1	97.0	71.2	*nd*	*nd*
Nonactin	0.05	0.18	100.0	115.7	88.3	*nd*	*nd*
Norsolorinic acid	0.8	2.4	100.0	23.9	88.2	*nd*	*nd*
Ochratoxin A	0.4	1.2	99.3	100.5	70.1	85.1	71.3
Ochratoxin B	0.4	1.2	*nd*	*nd*	*nd*	86.5	96.8
O-Methylsterigmatocystin	0.12	0.36	43.6	105.9	54.1	73.0	68.5
Oxaline	0.4	1.2	100.0	86.1	45.5	*nd*	*nd*
Pestalotin	0.4	1.2	87.3	108.0	82.3	*nd*	*nd*
Questiomycin A	2	6	95.2	102.9	89.9	*nd*	*nd*
Quinolactacin A	0.013	0.042	114.6	96.2	30.6	*nd*	*nd*
Rugulusovin	0.2	0.6	114.1	100.0	34.6	*nd*	*nd*
seco-Sterigmatocystin	0.15	0.45	100.0	141.0	82.0	*nd*	*nd*
Skyrin	0.15	0.45	100.0	88.8	91.7	*nd*	*nd*
Sterigmatocystin	0.1	0.3	98.9	91.9	57.5	80.1	34.2
Sydowinin A	2.9	9.6	100.0	123.7	71.8	*nd*	*nd*
Terphenyllin	1.6	–	*nd*	*nd*	*nd*	74.8	9.2
Tryptophol	8	24	116.0	129.7	13.9	64.3	45.8
Versicolorin A	0.24	0.72	91.2	90.5	72.2	*nd*	*nd*
Versicolorin C	0.24	0.72	100.0	90.9	87.4	83.1	66.7
WIN-64821	0.32	1.1	100.0	110.9	81.2	*nd*	*nd*
Xanthotoxin	0.05	0.17	100.0	97.6	93.1	*nd*	*nd*
Zearalenone	0.4	1.2	*nd*	*nd*	*nd*	73.4	47.1

[a]Limit of detection: expressed as μg/kg sample; S/N = 3∶1. [b]Limit of quantification: expressed as μg/kg sample. [c]Recovery from spiking food samples (*n*=5).

3.2 Mycotoxins in sorghum and sesame

Occurrence data for mycotoxins and other fungal metabolites quantified in sorghum and sesame are presented in Table 2. A total of 14 mycotoxins including aflatoxin B_1 (AFB_1), fumonisin (FB_1), deoxynivalenol (DON), ochratoxin A (OTA) and zearalenone ZEN were detected in the sorghum samples, while eight mycotoxins (including

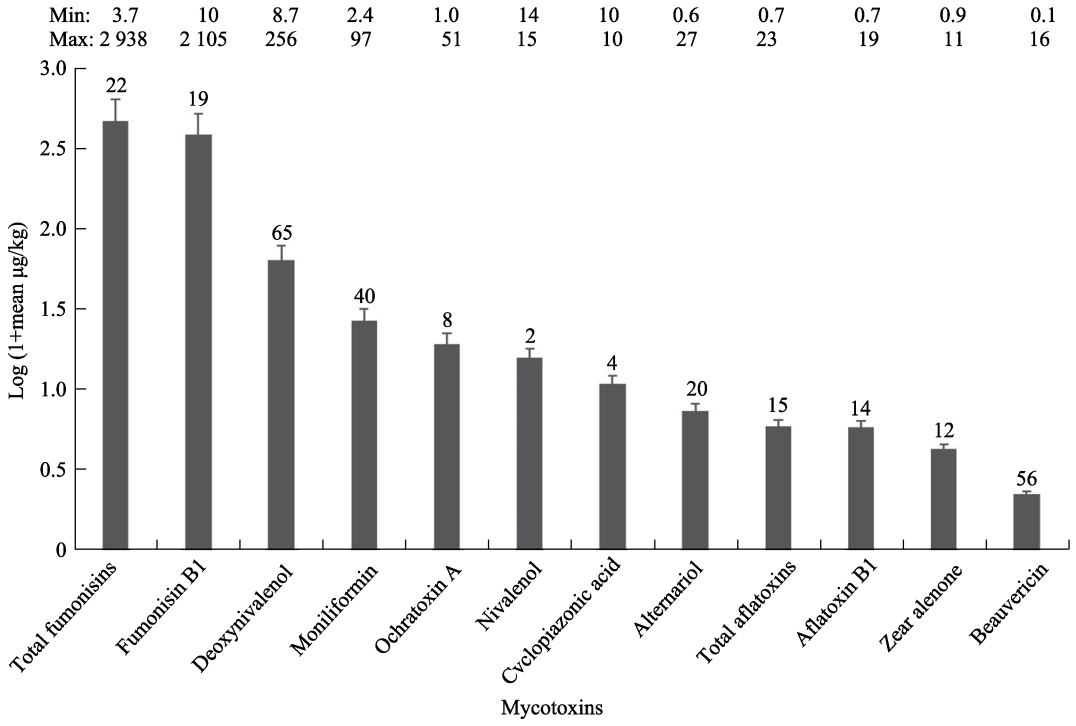

Fig.1 Mycotoxin levels in 107 commonly consumed food crop samples marketed in Nasarawa state, Nigeria. Values on error bars indicate percentage food samples contaminated by mycotoxin. Minimum and maximum mycotoxin concentrations (μg/kg) of mycotoxins are given at the top of the graph

DON, FB_1 and ZEN) were detected in the sesame. AFB_1 and OTA were not detected in any of the sesame samples. Our findings of AFB_1, OTA and ZEN in sorghum samples in the present study agree with previous reports from Nigeria, Ethiopia and Tunisia where similar mycotoxin profile were reported in sorghum[4,32-34]. Aflatoxin B_1 contaminated 19% of the sorghum samples (max: 4.75 μg/kg; mean: 2.04 μg/kg). This is lower than the 54% (max: 1 164 μg/kg; mean: 199.5 μg/kg) and 59.4% (max: 31.7 μg/kg; mean: 1.71 μg/kg) prevalence reported by Makun et al.[4] and Lahouar et al.[33] from Nigeria and Tunisia. Obvious reasons for this could be the quality and number of sorghum samples analyzed. Makun et al.[4] analyzed 168 visibly mouldy sorghum, which may have contributed to the higher prevalence reported, while Lahouar et al.[33] analyzed more samples (*n*=64). Our result was, however, higher than the prevalence of 12.9% (max: 62.5 μg/kg; mean: 29.5 μg/kg) reported by Chala et al.[32] for 70 sorghum in Ethiopia. Our observation of non-detects for AFB_1 in the sesame samples agrees with previous report from Nigeria wherein no aflatoxin was found in 17 sesame vended in Plateau state[18]. It is however in contrast with previous reports from Iran and Egypt, where AFB_1 was reported in 18.1% (mean: 1.62 μg/kg) and more than 60% (mean: 66.74 μg/kg) of sesame samples [35-36].

Table 2 Occurrence of mycotoxins and other microbial metabolites in sesame and sorghum marketed in Nasarawa state, Nigeria

Mycotoxins	Sesame (*n*=35)					Sorghum (*n*=36)				
	N(%)[a]	Min	Max	Mean	SD	N(%)[a]	Min	Max	Mean	SD
Aflatoxin B_1	0(0)	<LOD	<LOD	<LOD	–	7(19)	0.72	4.75	2.04	1.39
Alternariol (AOH)	19(54)	1.01	27.4	7.07	8.08	1(3)	0.65	0.65	0.65	–
AOHmethylether	25(71)	0.25	79.9	8.44	16.2	3(8)	0.25	0.51	0.34	0.10
Beauvericin	23(66)	0.10	5.29	0.75	1.17	26(72)	0.13	16.3	1.03	3.12
Deoxynivalenol	35(100)	37.1	256	114	56.5	35(97)	8.70	19.5	13.4	2.44
Fumonisin B_1 (FB_1)	3(9)	10.2	15.4	12.5	2.66	17(47)	11.9	2105	461	773
Fumonisin B_2	1(3)	6.30	6.30	6.30	–	14(39)	3.72	493	132	192
Fumonisin B_3	0(0)	<LOD	<LOD	<LOD	–	7(19)	1.89	264	137	117
Fumonisin B_4	0(0)	<LOD	<LOD	<LOD	–	6(17)	16.9	76.5	45.7	27.0

Continued 2

Mycotoxins	Sesame (*n*=35)					Sorghum (*n*=36)				
	N(%)[a]	Min	Max	Mean	SD	N(%)[a]	Min	Max	Mean	SD
Hydrolysed FB_1	0(0)	<LOD	<LOD	<LOD	–	6(17)	3.95	20.5	12.0	8.19
Moniliformin	4(11)	3.75	51.3	25.3	22.5	35(97)	7.88	97.1	29.1	19.0
Ochratoxin A	0(0)	<LOD	<LOD	<LOD	–	6(16)	0.96	42.9	16.5	18.6
Ochratoxin B	0(0)	<LOD	<LOD	<LOD	–	5(14)	0.82	5.89	3.00	2.47
Zearalenone	10(29)	0.93	4.33	1.82	1.05	3(8)	6.95	10.6	8.25	2.02
Fungal metabolites										
Altenuene	11(31)	1.12	25.5	6.92	8.48	0(0)	<LOD	<LOD	<LOD	–
Altenusin	4(11)	1 114	2 540	1 721	697	0(0)	<LOD	<LOD	<LOD	–
Altertoxin-I	14(40)	2.43	14.6	5.44	4.1	0(0)	<LOD	<LOD	<LOD	–
Andrastin A	5(14)	1.51	27.3	8.67	7.4	6(17)	2.38	33.9	15.2	10.1
Averufin	0(0)	<LOD	<LOD	<LOD	–	21(58)	0.21	1.59	0.98	0.50
Bikaverin	4(11)	103	134	115	13.2	29(81)	34.3	314	79.9	75.5
Curvularin	3(9)	1.03	1.03	1.03	0.00	18(50)	1.03	13.6	5.60	4.21
Emodin	26(74)	0.66	3.32	1.26	0.79	35(97)	0.56	5.71	2.40	1.49
Equisetin	8(23)	2.20	91.6	17.2	30.5	12(33)	1.60	208	50.4	65.3
Festuclavin	5(14)	1.15	52.90	17.6	21.2	1(3)	1.61	1.61	1.61	–
Fusarinolic acid	0(0)	<LOD	<LOD	<LOD	–	8(22)	44.3	111	70.0	27.2
Kojic acid	14(40)	554	1193	719	165	35(97)	199	34 223	6 749	10 253
Macrosporin	11(31)	1.49	11.67	3.01	2.93	35(97)	3.47	48.2	13.4	10.5
Malformin A	0(0)	<LOD	<LOD	<LOD	–	3(8)	29.3	50.5	39.3	10.7
Malformin C	0(0)	<LOD	<LOD	<LOD	–	3(8)	6.36	12.0	9.11	2.82
Nidurufin	6(17)	0.29	2.41	1.19	0.89	7(19)	0.15	2.07	0.97	0.78
O-methylSTER	0(0)	<LOD	<LOD	<LOD	–	3(8)	0.40	0.50	0.44	0.05
Sterigmatocystin (STER)	0(0)	<LOD	<LOD	<LOD	–	24(67)	0.30	19.2	4.51	5.52
Terphenyllin	0(0)	<LOD	<LOD	<LOD	–	8(22)	3.45	69.9	30.8	24.8
Tryptophol	35(100)	187	446	287	64.7	27(75)	66.1	164	97.8	28.7
Versicolorin C	1(3)	0.25	0.25	0.25	–	26(72)	0.25	0.84	0.37	0.16

[a]Number (percentage) of positive samples.

Fumonisins detected in sorghum and sesame include fumonisin B_1, B_2, B_3 and B_4. Fumonisin B_1 contaminated more samples of sorghum (incidence: 47%; max: 2 105 μg/kg; mean: 461 μg/kg) compared to the sesame (incidence: 9%; max: 15.4 μg/kg; mean: 12.5 μg/kg). The maximum and mean levels of total fumonisins (sum of FB_1, FB_2 & FB_3) detected in sorghum samples analyzed in the present study were up to 2 862 μg/kg and 730 μg/kg, respectively. These values are higher than the levels of total fumonisins (sum of FB_1, FB_2 & FB_3) previously reported in sorghum from Ethiopia (max: 40.99 μg/kg; mean: 22.22 μg/kg) and four agro-ecological zones of Nigeria (max: 179 μg/kg; mean: 150 μg/kg) [32,37], but lower than the levels found in 20 sorghum samples from four microclimatic zones of Niger state, Nigeria (max: 8 400 μg/kg; mean: 6 198 μg/kg)[34]. The levels of FB_1 + FB_2 in sesame analyzed in the present study (max: 21.7 μg/kg; mean: 18.8 μg/kg) are, however, relatively similar to the levels (max: 37.9 μg/kg; mean: 25.9 μg/kg) previously reported in 24 sesame samples from Abuja (northern Nigeria)[38]. Deoxynivalenol was the most frequently occurring mycotoxin in both sesame (incidence: 100%; max: 256 μg/kg; mean: 114 μg/kg) and sorghum (incidence: 97%; max: 19.5 μg/kg; mean: 13.4 μg/kg). Our data is higher than those reported by Fapohunda et al.[38] for DON (incidence: 58%; max: 171 μg/kg; mean: 78.3 μg/kg) in sesame. Ochratoxin A (max: 42.9 μg/kg; mean: 16.5 μg/kg) contaminated 16% of the sorghum samples; an incidence lower than the 94% (max: 29.5 μg/kg; mean: 8.28 μg/kg) and 75% (max: 5.60 μg/kg; mean: 2.44 μg/kg) previously reported

by Makun et al.[21] and Onyedum et al.[34], respectively. Conversely, none of our sesame samples were contaminated with OTA; this is in contrast to the observation of Makun et al.[21], who reported 100% OTA prevalence in 19 sesame from Niger state (northern Nigeria). About 29% of the sesame and 8% of the sorghum samples contained ZEN at the respective max (mean) concentrations: 4.33 (1.82) μg kg^{-1} and 10.6 (8.25) μg kg^{-1}. However, higher prevalence of 100% (max: 1.25 μg/kg; mean: 0.806 μg/kg) and 32.9% (max: 374 μg/kg; mean: 43.8 μg/kg) were reported in sesame and sorghum from Nigeria and Ethiopia, respectively[32,39]. The disparities in our data and previous reports on both crops in literature may be attributed to numerous factors including variations in sample sizes analyzed in the various studies, performances of analytical instrument at the time of the various analyses, geographical influences, agricultural (pre- and post-harvest) practices and diversity of moulds that contaminated the food crops. Of the 21 other fungal metabolites quantified in sorghum and sesame, emodin/kojic acid/macrosporin and tryptophol were the most occurring in 97% and 100% samples, respectively.

3.3 Mycotoxins in bush mango, cashew nut and okra

The distribution (occurrence and levels) of mycotoxins and other microbial metabolites in bush mango, cashew nut and dried okra are shown in Table 3. Of the 10 mycotoxins detected in these foods, six (AFB_1, AFG_1, AFM_1, alternariolmethylether, cyclopiazonic acid and OTA) were detected in bush mango, two (AOH and BEAU) in cashew nut and seven (all except AFG_1, AFM_1 and AOH) in the dried okra. The concentrations of AFB_1 (max: 19.2 μg/kg) and AFM_1 (max: 1.41 μg/kg) quantified in bush mango samples in the present study were 37 and 13 times lower than the maximum levels of AFB_1 (713.2 μg/kg) and AFM_1 (25.5 μg/kg previously reported in 40 bush mango samples from local markets in Lagos (southern Nigeria)[9]. The variations in these data are mainly attributed to post-harvest storage duration and conditions; samples in the present study were not stored beyond 14 days with the vendors whereas samples analyzed by Ezekiel et al.[9] were stored for 15~90 days with the maximum contamination levels as reported found in discoloured samples with longer storage duration.

Table 3 Mycotoxins and other microbial metabolites in cashew nut, bush mango and dried okra marketed in Nasarawa state, Nigeria

Mycotoxins	Cashew nut (*n*=12)					Bush mango (*n*=12)					Dried okra (*n*=12)				
	N(%)[a]	Min	Max	Mean	SD	N(%)[a]	Min	Max	Mean	SD	N(%)[a]	Min	Max	Mean	SD
Aflatoxin B_1	0(0)	<LOD	<LOD	<LOD	<LOD	5(42)	1.93	19.2	8.16	7.83	3(25)	4.53	8.27	6.24	1.89
Aflatoxin G_1	0(0)	<LOD	<LOD	<LOD	<LOD	3(25)	0.50	3.30	2.15	1.47	0(0)	<LOD	<LOD	<LOD	<LOD
Aflatoxin M_1	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	1.41	1.41	1.41	–	0(0)	<LOD	<LOD	<LOD	<LOD
Alternariol (AOH)	1(8)	0.60	0.60	0.60	–	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD
AOHmethylether	0(0)	<LOD	<LOD	<LOD	<LOD	5(42)	0.05	0.32	0.15	0.11	1(8)	0.24	0.24	0.24	–
Beauvericin	1(8)	0.09	0.09	0.09	–	0(0)	<LOD	<LOD	<LOD	<LOD	10(83)	0.17	15.5	3.19	5.21
Cyclopiazonic acid	0(0)	<LOD	<LOD	<LOD	<LOD	2(17)	10.0	10.0	10.0	0.00	2(17)	10.0	10.0	10.0	0.00
Moniliformin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	4(33)	2.40	5.31	3.13	1.45
Nivalenol	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	2(17)	14.6	15.3	15.0	0.49
Ochratoxin A	0(0)	<LOD	<LOD	<LOD	<LOD	2(17)	13.2	51.4	32.3	27.1	1(8)	3.39	3.39	3.39	–
Fungal metabolites															
3-Nitropropionic acid	8(67)	1.20	4.44	2.51	1.47	10(83)	1.20	32.3	6.79	9.42	12(100)	8.21	1570	249	508
Agroclavine	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	2(17)	68.3	99.0	83.6	21.7
Altersetin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	1.80	1.80	1.80	–
Andrastin A	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	0.38	0.38	0.38	–	1(8)	0.38	0.38	0.38	–
Ascochlorin	0(0)	<LOD	<LOD	<LOD	<LOD	6(50)	0.39	0.95	0.66	0.22	9(75)	0.29	3.30	1.04	1.01
Aspercolorin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	4(33)	1.20	2.93	2.00	0.93
Aspergillicin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	2(17)	12.4	13.2	12.8	0.58
Aspergillimide	0(0)	<LOD	<LOD	<LOD	<LOD	6(50)	0.69	1.73	1.12	0.47	1(8)	4.76	4.76	4.76	–
Asperglaucide	12(100)	4.67	49.2	16.6	13.9	12(100)	1170	6235	2477	1453	12(100)	7.27	98.3	39.5	32.6
Asperphenamate	12(100)	4.02	637	201	239	12(100)	348	832	477	151	12(100)	8.96	1795	453	785
Averantin	0(0)	<LOD	<LOD	<LOD	<LOD	8(67)	0.29	10.7	2.66	3.49	1(8)	2.34	2.34	2.34	–

Continued 3

Mycotoxins	Cashew nut (*n*=12)					Bush mango (*n*=12)					Dried okra (*n*=12)				
	N(%)[a]	Min	Max	Mean	SD	N(%)[a]	Min	Max	Mean	SD	N(%)[a]	Min	Max	Mean	SD
Averufin	0(0)	<LOD	<LOD	<LOD	<LOD	12(100)	0.81	41.2	11.6	12.2	5(42)	0.30	1.31	0.80	0.40
Brevianamid F	12(100)	75.8	91.8	85.0	6.01	6(50)	3.34	44.1	14.7	15.1	0(0)	<LOD	<LOD	<LOD	<LOD
Citreorosein	8(67)	0.96	79.7	32.3	38.5	12(100)	0.96	14.7	6.14	4.31	8(67)	2.47	17.9	6.76	5.12
Curvularin	1(8)	1.00	1.00	1.00	–	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD
Cyclo(L-Pro-L-Tyr)	12(100)	10.4	23.9	17.1	5.02	12(100)	1.20	54.4	7.21	14.9	6(50)	8.41	38.0	24.8	11.6
Cyclo(L-Pro-L-Val)	12(100)	33.7	96.2	63.6	20.4	12(100)	4.97	266	39.9	72.5	12(100)	3.51	37.7	15.8	13.4
Dihydroxymellein	4(33)	0.85	8.71	4.70	4.45	10(83)	0.85	29.9	10.6	10.8	4(33)	6.98	51.5	28.4	24.4
Emodin	6(50)	1.23	23.1	14.1	9.53	12(100)	1.30	32.7	14.6	10.6	12(100)	1.09	5.48	2.73	1.32
Equisetin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	8(67)	0.36	7.34	2.30	2.42
Fallacinol	12(100)	0.93	908	243	396	12(100)	0.52	13.8	4.29	4.19	0(0)	<LOD	<LOD	<LOD	<LOD
Fellutanine A	12(100)	9.60	20.5	13.7	3.83	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD
Integracin A	0(0)	<LOD	<LOD	<LOD	<LOD	7(58)	0.16	14.9	2.95	5.38	1(8)	0.92	0.92	0.92	–
Integracin B	0(0)	<LOD	<LOD	<LOD	<LOD	5(42)	0.15	11.4	4.34	4.69	0(0)	<LOD	<LOD	<LOD	<LOD
Iso-Rhodoptilometrin	0(0)	<LOD	<LOD	<LOD	<LOD	8(67)	0.96	2.66	1.17	0.60	1(8)	0.96	0.96	0.96	–
Kojic acid	12(100)	114	477	199	122	12(100)	166	11 467	3 516	3 777	12(100)	186	7 380	1 412	2 393
Macrosporin	1(8)	0.53	0.53	0.53	–	1(8)	0.53	0.53	0.53	–	7(58)	0.89	2.08	1.52	0.45
Malformin C	2(17)	4.08	11.6	7.82	5.29	5(42)	0.60	35.9	12.6	16.9	0(0)	<LOD	<LOD	<LOD	<LOD
Methylsulochrin	2(17)	0.39	0.56	0.48	0.12	1(8)	0.68	0.68	0.68	–	0(0)	<LOD	<LOD	<LOD	<LOD
Monocerin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	12.2	12.2	12.2	–
Mycophenolic acid	0(0)	<LOD	<LOD	<LOD	<LOD	2(17)	1.03	2.94	1.99	1.35	0(0)	<LOD	<LOD	<LOD	<LOD
N-Benzoyl-Phenylal	9(75)	1.39	88.7	28.2	33.4	12(100)	24.9	130	50.9	27.3	10(83)	2.53	174	54.0	78.6
Neoechinulin A	12(100)	16.1	15 536	4 003	6 544	11(92)	11.0	243	72.8	73.5	10(83)	5.00	104	37.8	41.4
Nidurufin	0(0)	<LOD	<LOD	<LOD	<LOD	4(33)	0.24	4.07	1.59	1.70	0(0)	<LOD	<LOD	<LOD	<LOD
Cytochalasin J	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	2(17)	317	436	376	83.7
Norsolorinic acid	0(0)	<LOD	<LOD	<LOD	<LOD	3(25)	1.20	4.31	2.73	1.56	1(8)	2.84	2.84	2.84	–
O-MethylSTER	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	0.48	0.48	0.48	–	0(0)	<LOD	<LOD	<LOD	<LOD
Oxaline	0(0)	<LOD	<LOD	<LOD	<LOD	4(33)	0.60	0.60	0.60	0.00	0(0)	<LOD	<LOD	<LOD	<LOD
Pestalotin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	0.60	0.60	0.60	–
Questiomycin A	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	3.00	3.00	3.00	–	0(0)	<LOD	<LOD	<LOD	<LOD
Quinolactacin A	10(83)	0.27	1.18	0.59	0.33	11(92)	0.78	4.09	1.78	1.05	3(25)	0.77	3.13	2.24	1.28
Rugulusovin	11(92)	6.93	12.6	9.86	2.07	4(33)	2.76	7.66	4.57	2.16	2(17)	4.04	8.42	6.23	3.10
seco-STER	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	0.61	0.61	0.61	–
Skyrin	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	0.23	0.23	0.23	–	5(42)	0.23	0.94	0.43	0.31
Sterigmatocystin	1(8)	0.45	0.45	0.45	–	3(25)	0.29	2.01	0.95	0.93	2(17)	7.28	7.68	7.48	0.29
Sydowinin A	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	15.9	15.9	15.9	–
Tryptophol	12(100)	273	572	344	98.4	5(42)	51.5	127	88.1	37.1	12(100)	401	18 651	6 008	6 252
Versicolorin A	0(0)	<LOD	<LOD	<LOD	<LOD	11(92)	1.33	12.5	4.61	3.57	3(25)	0.90	1.07	1.00	0.09
Versicolorin C	0(0)	<LOD	<LOD	<LOD	<LOD	11(92)	1.62	10.7	4.26	3.05	9(75)	0.36	1.51	0.79	0.43
WIN-64821	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	1(8)	2.18	2.18	2.18	–
Xanthotoxin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	4(33)	1.70	3.37	2.31	0.74
Bacterial metabolites															
Chloramphenicol	4(33)	0.10	0.86	0.52	0.35	3(25)	0.30	1.13	0.66	0.42	1(8)	0.14	0.14	0.14	–
Monactin	0(0)	<LOD	<LOD	<LOD	<LOD	3(25)	0.09	0.20	0.13	0.06	9(75)	0.09	1.82	0.81	0.60
Nonactin	0(0)	<LOD	<LOD	<LOD	<LOD	0(0)	<LOD	<LOD	<LOD	<LOD	5(42)	0.09	0.60	0.22	0.22

[a]Number (percentage) of positive samples.

Only 3/12 (25%) of the dried okra samples analyzed in the present study were contaminated with AFB_1 and the levels reached 8.27 μg/kg. Previously, aflatoxins were not detected in 20 okra

from Niger state (northern Nigeria)[40], whereas, the levels we found were higher than those reported in dried okra from markets in Benin, Mali and Togo (max: 5.4 μg/kg)[41] and Turkey (max: 1.7 μg/kg)[42]. Here, we report on OTA contamination of a dried okra sample (3.4 μg/kg); data that was not previously available for okra. Okra is a perishable food crop often freshly harvested from the farm; however, in order to prolong its shelf life, vendors cut the okra fruit into small parts prior to sun drying. In most cases, the okra is dried on bare soil, such that, it is exposed to soil fungi and mycotoxigenic fungal spores from the environment[13]. Perhaps, this poor processing/vending practice contributed to the presence of up to seven mycotoxins, aflatoxin B_1 inclusive, in the analyzed okra samples.

Aflatoxins were not detected in any of the cashew nut samples; this agrees with the reports of Lamboni et al.[43] and Sombie et al.[44], who did not find aflatoxins in 84 and 50 cashew nuts from Benin Republic and Sierra Leone, respectively. Our finding is, however, in contrast to the reports of Milhome et al.[45] and Adetunji et al.[22], who documented up to 31.5 μg/kg and 6.8 ng/g aflatoxins in cashew nuts from Brazil and Nigeria, respectively. In view of the disparities in the type and patterns of fungal metabolites reported for cashew over the years and in order to better gain understanding of the contamination scenario, a comprehensive inter-laboratory study of this food matrix kept under different handling conditions and from different continents is required. Several 51 other fungal metabolites were also detected at varying levels in the bush mango, cashew nut and dried okra samples. In addition, three bacterial metabolites were quantified, including chloramphenicol which was the only one detected in the food crops.

3.4 Public health impacts of mycotoxins detected in the foods

With respect to the public health impacts of occurring mycotoxins in the food samples, AFB_1 is the most potent mycotoxin, classed as category 1 human carcinogen[46]. Its ingestion has been linked to liver cancer and immune suppression[2,47]. All the contaminated okra, two bush mango and one sorghum sample contained concentrations of aflatoxins beyond the permissible limits of 4 μg/kg set for total aflatoxins in foods in Nigeria. Although the sample sizes investigated in this study seem rather little, the presence of aflatoxins in any food constitutes a potential health risk due to the daily consumption of these foods leading to chronic exposures. In addition, the co-occurrence of AFB_1 with other possible carcinogenic and nephrotoxic mycotoxins such as FB_1 and OTA, as well as other emerging mycotoxins (e.g., BEAU), in the food samples calls for concern in view of the widespread consumption of these food crops in the region[2,46,48]. AFB_1 co-occurred with FB_1 in at least 19% of the sorghum samples albeit at low FB_1 concentrations, while more than 17% and 8% of the bush mango and dried okra samples, respectively, were co-contaminated with AFB_1 and OTA. These two mycotoxins have been reported to interact to exacerbate adverse health effects in humans especially children [49]; however, the effects of the concentrations reported in the present study are unknown. In view of recent knowledge gained from *in vitro* combinatory toxicological studies suggesting potential additive and/or synergistic effects of several mycotoxin combinations[50-51], further studies may be required to look into the effects of the various toxins reported herein.

4. CONCLUSIONS

This study indicates that the commonly consumed bush mango, cashew nut, dried okra, sesame and sorghum vended in local markets Nasarawa state, Nigeria, are prone to mycotoxin contamination. Sorghum was the most susceptible of the food crops due to the presence of 14 mycotoxins. In addition, the occurrence of OTA and its co-occurrence with aflatoxin B_1 in dried okra, which was not previously reported, is documented here. The data presented herein is highly relevant to inform viable interventions such as: 1) drying of these foods at household levels on elevated platforms to prevent contact with bare soil; 2) pulverization of okra and bush mango after sun drying, proper packaging in air-tight plastic containers or cellophane bags, and storage at low temperature (<10 ℃) to prevent mycotoxin development. Furthermore, government through relevant agencies should provide mechanical dryers at the community level for ease of drying of various agricultural products to minimize fungal growth.

REFERENCES

See in its Chinese version P81-P82.