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Multi-mycotoxin determination in rice, maize and peanut products most consumed in Côte d'Ivoire by UHPLC-MS/MS

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Abstract

The aim of this study was to determine the multi-mycotoxin occurrence in cereal and oilseed products most consumed in Côte d'Ivoire. A total of 238 samples of rice (88 produced locally or imported), maize (79, cracked or flour) and peanut paste (71) were collected in the main markets of Abidjan, Bouaké and Korhogo. An UHPLC-MS/MS method allowed the analysis of 77 mycotoxins. All the peanut paste samples were contaminated by aflatoxin B_1 (AFB1) with 99% exceeding the EU limits of 2 $\mu g.kg^{-1}$ for AFB1 and 4 $\mu g.kg^{-1}$ for total aflatoxins (AFT: B1+B2+G1+G2), and concentrations reaching up to 4535 $\mu g.kg^{-1}$ (AFB1) and 8094 $\mu g.kg^{-1}$ (AFT). Maize (96%) and rice (57%) samples were also contaminated by AFB1 with 58% and 24% respectively above the EU limits and maximum levels of 80 $\mu g.kg^{-1}$ for maize and 14 $\mu g.kg^{-1}$ for rice. Only 6% of the cereal samples (3 rice and one maize samples) had ochratoxin A content above the EU limit (3 $\mu g.kg^{-1}$). Fumonisins and zearalenone were detected, respectively, in 91% and 8% of the maize samples, and in 18% and 5% of the rice samples but at levels below EU limits. Out of the 238 samples, 91% were contaminated with more than one mycotoxin including EU regulated mycotoxins and/or other

mycotoxins mainly beauvericin (79% of the samples), equisetin (71%), aflatoxin M1 (45%), cyclopiazonic acid (32%), fumonisin B3 (29%), sterigmatocystin (24%), citrinin (18%), ochratoxin B (16%) and fusaric acid (15%). The peanut paste samples represented the highest risk to consumer health followed by maize and rice samples.

Keywords: Mycotoxins, Cereals, Rice, Maize, Peanut, Côte d'Ivoire

1. Introduction

Mycotoxins are secondary metabolites of filamentous fungi that colonize a wide range of crops, including cereals and oilseeds, both in the field and after harvest, especially during storage. Due to their thermal and chemical stability, mycotoxins can also be found in processed foods of plant origin, or by transfer, in food products of animal origin such as milk, eggs, meat and offal from animals consuming contaminated feed. These natural contaminants represent a major concern for human and animal health since they can cause acute or chronic intoxications which are sometimes fatal due to their various toxic effects (carcinogenic, hepatotoxic, nephrotoxic, neurotoxic, genotoxic, immunotoxic, oestrogenic...). More than 300 mycotoxins have been identified, but attention is given mainly to those with greatest public health and agro-economic significance. The most common genera of mycotoxigenic fungi in food and feed are *Aspergillus*, *Fusarium*, *Penicillium*, *Claviceps* and *Alternaria* (AFSSA, 2006; Zain, 2011). In order to protect consumer health, many countries worldwide, and in particular Europe, have established regulatory limits and guidance values for certain mycotoxins in foodstuffs (European Commission, 2006, 2013; Van Egmond, Schothorst, & Jonker, 2007). Recently, there is a growing interest in the occurrence and toxicity of emerging *Fusarium* mycotoxins (beauvericin, fusaproliferin, moniliformin, and enniatins A, A1, B, B1) and biologically modified forms of certain mycotoxins (EFSA, 2014a, 2014b; Jestoi, 2008). The latter include *Fusarium* mycotoxins conjugated to polar compounds by plants (the so called masked mycotoxins) or fungi (EFSA, 2014a; Rychlik et al., 2014). The co-occurrence of different mycotoxins within the same food almost always occurs and may result in a greater toxicity to humans due to possible additive or synergistic effects (Alassane-Kpembi et al., 2017; Grenier & Oswald, 2011). A consumer survey conducted under the 3CIvoire project (EuropeAid/129596/L/ACT/CI DCI-NSAPVD/2010/64) has revealed that rice, maize and peanut are among the most consumed cereals and oilseeds in Côte d'Ivoire. In this country, rice consumption has been reported to account for more than half of the cereal intake (Boansi, 2013). As its production satisfies only about 50% of the domestic consumption, it is massively imported mainly from Asian countries (USDA, 2015). Maize is consumed in various forms, in particular as "Kabatoh" which is a cooked dough prepared from maize flour with or without potash (Yeo, 2011).

Like rice and maize, peanut is considered a staple food crop because of its use not only as an important oil crop, but also for the paste produced from ground roasted seeds (Sangare, Koffi, Akamou, & Fall, 2009). So far, data on multiple mycotoxins in rice, maize, peanut and derived products (maize flour and peanut paste) are limited in Côte d'Ivoire and whereby the attention has been mainly concerned with those under EU regulation. The aim of the present study was to investigate the occurrence of 77 mycotoxins in the cereal (rice grains, cracked maize and maize flour) and oilseed (peanut paste) products that are most consumed by the Ivorian population in order to contribute to food safety risk assessment in Côte d'Ivoire.

2. Materials and methods

2.1. Food samples

In April 2013, 238 food samples (between 260 and 560 g each) intended for direct human consumption or use as an ingredient in foodstuffs were collected in the main markets of Abidjan, Bouaké and Korhogo, Côte d'Ivoire: 88 rice samples (47 produced locally and 41 imported), 79 maize samples (29 cracked maize, 32 and 18 flour samples respectively with potash and without potash) and 71 peanut paste samples. They were sampled in new plastic bags following the sales practices and immediately kept at 4°C, then stored at -20°C until sample preparation.

2.2 Sample preparation and storage

Frozen rice and cracked maize samples were finely ground with a Retsch mill (ZM 200, sieve 1-mm). The ground samples and the maize flour samples previously thawed were homogenized for 30 min using a Chopin MR2L mixer. Peanut paste samples were thawed and homogenized for 15 min with a blender (Thermomix VORWERK TM 21) by selecting the speed 2.5. Homogenized samples were kept at -20°C until mycotoxin analysis.

2.3 Multi-mycotoxin analysis

The QuEChERS extraction procedure and UHPLC-MS/MS method described in detail by Oplatowska-Stachowiak et al. (2015) was used to determine and quantify (or semi-quantify) 77 mycotoxins (Table 1) in the 238 homogenized maize and rice flour and peanut paste samples. Standards obtained in powder form were prepared at the concentration 1 mg/mL in the appropriate amount of solvent (MeCN or MeOH) according to the manufacturer's instructions. Three different solutions of standards were prepared as calibrants for the instrument to determine the limits of detection (LOD) and quantification (LOQ) (Table 1) for each mycotoxin analysed. Standard set 1 contained a concentrated stock solution of all the mycotoxins included in the method except for

fumonisins B1 (FB1), B2 (FB2) and B3 (FB3); the four masked zearalenone metabolites; zearalenone-14-sulfate (ZEN-14-Sulf); deoxynivalenol-3-glucoside (DON-3-Glc); and moniliformin (MON). Standard set 2 contained FB1, FB2, and FB3. Standard set 3 contained the four masked zearalenone metabolites, ZEN-14-Sulf, DON-3-Glc, and MON. The mycotoxins were assigned to seven different calibration groups as described by Oplatowska-Stachowiak et al. (2015) depending on the requirements and/or sensitivity. These concentrated standard sets were also used for preparing calibrants in matrix. The mycotoxin quantitation was achieved by preparing matrix-matched calibration curves with blank maize or rice flour and peanut paste samples spiked before extraction to correct for the recovery losses. If the measured mycotoxin content in a sample was higher than the highest calibration point, the extracted sample was diluted and analysed again. The data was analyzed using TargetLynx processing software (Waters, Wilmslow, UK) whereby linear 1/x weighted calibration curves were calculated.

3. Results and discussion

3.1 Multi-mycotoxin occurrence

Out of the 238 food samples collected in Côte d'Ivoire, 91% were contaminated with more than one mycotoxin (about 21% between 2 and 4 mycotoxins, 21% between 5 and 7 and 48% with more than 8), 4% with only one mycotoxin (9 rice samples) and 5% (12 rice samples) were not contaminated. The largest number of mycotoxins detected in the same sample was found in maize samples (14 in 3 samples) followed by peanut paste (13 in 6 samples) and rice samples (8 in 2 samples). However, the peanut paste samples were the most contaminated with multiple mycotoxins followed by the maize then rice samples, with at least 5 mycotoxins detected in the same sample (against 2 in both maize and rice samples). Furthermore, 94% of the peanut samples contained more than 8 mycotoxins (against 58% and 2% for maize and rice samples, respectively) (Table 2). Among the maize and rice samples, the maize flour samples without potash and the local rice samples were the most contaminated with multiple mycotoxins. A total of 24 mycotoxins (8 EU regulated and 16 others) were detected in all types of food samples, with 18, 17 and 15 from the rice, maize and peanut paste samples, respectively (Figure 1, Tables 3-6). Similar studies have also highlighted the co-existence of EU regulated and/or other mycotoxins in maize from Burkina Faso and Mozambique (Warth et al., 2012), Democratic Republic of Congo (DRC) (Mulunda, Dzoma, Nyirenda, & Bakunzi, 2013), Nigeria (Ogara et al., 2016), South Africa (Chilaka et al., 2012; Shephard et al., 2013) and Tanzania (Kamala et al., 2015) and maize flour from Cameroon (Abia et al., 2013); rice from Nigeria (Makun, Dutton, Njobeh, Mwanza & Kabiru, 2011; Makun, Gbodi, Akanya, Salako, &

Ogbadu, 2007); peanut from DRC (Mulunda et al., 2013) and peanut paste from Cameroon (Abia et al., 2013).

3.2 EU regulated mycotoxins

To date, there is no regulation for mycotoxins in foodstuffs in Côte d'Ivoire. Therefore, the EU maximum levels for mycotoxins in cereals (maize and rice), peanuts and derived products intended for direct human consumption (European Commission, 2006, 2007, 2010, 2012) are used for reference in the present article.

Aflatoxin B1 (AFB1) which is the predominant form of aflatoxins (AFs) in foods (AFSSA, 2006) was the most frequently detected EU regulated mycotoxin in the analysed food samples. Aflatoxins B2 (AFB2), G1 (AFG1) and G2 (AFG2) were also found in these samples but at a lower frequency and concentration than AFB1. AFs have been classified as Group 1 human carcinogens (IARC, 2002) with AFB1 being the most toxic followed by AFG1 then AFB2 and AFG2 (EFSA, 2007). The liver is the primary target organ. Acute high level aflatoxin exposure has resulted in deaths in some parts of the world, particularly in African countries, and chronic low-level aflatoxin exposure can increase the risk for human hepatocellular carcinoma and result in immune suppression (IARC, 2015; Zain, 2011). The peanut paste samples were the most contaminated with AFB1 and total aflatoxins (AFT: B1+B2+G1+G2) followed by the maize and rice samples (Table 3). This can be explained by the high peanut susceptibility to aflatoxin-producing fungi. AFB1 was recovered in 100%, 96% and 57% of the peanut paste, maize and rice samples, respectively; AFB2 in 99%, 67% and 30%; AFG1 in 100%, 57% and 22%; and AFG2 in 87%, 24% and 1%. Contamination levels higher than the EU limit for AFB1 (2 $\mu g.kg^{-1}$) were found in 99%, 61% and 42% of the contaminated peanut paste, maize and rice samples, respectively, and for AFT (4 $\mu g.kg^{-1}$) in 99%, 53% and 32% of these respective samples. The highest levels and means were obtained in peanut paste samples for AFB1, AFG1 and AFT (Table 3), and for AFB2 (1098 $\mu g.kg^{-1}$, mean 113 $\mu g.kg^{-1}$) and AFG2 (267 $\mu g.kg^{-1}$, mean 18 $\mu g.kg^{-1}$). The rice samples had the lowest maximum levels and means for AFB1, AFG1 and AFT (Table 3) and for AFB2 (1.1 $\mu g.kg^{-1}$, mean 0.6 $\mu g.kg^{-1}$) and AFG2 (0.9 $\mu g.kg^{-1}$ in one local rice sample). The highest levels quantified in maize samples were 80 $\mu g\ kg^{-1}$ for AFB1 (mean 8.6 $\mu g.kg^{-1}$), 84 $\mu g.kg^{-1}$ for AFG1 (mean 5.3 $\mu g.kg^{-1}$), 7 $\mu g.kg^{-1}$ for AFB2 (mean 1.5 $\mu g.kg^{-1}$), 3.8 $\mu g.kg^{-1}$ for AFG2 (mean 0.8 $\mu g.kg^{-1}$) and 173 $\mu g.kg^{-1}$ for AFT (mean 13 $\mu g.kg^{-1}$). Higher AFB1 contamination frequency and levels were also found in peanut paste or seed samples than in maize flour or grain samples from Cameroon and DRC (Abia et al., 2013 ; Mulunda et al., 2013), but Warth et al. (2012) in Burkina Faso and Mozambique found contrary results for the same types of samples. The high proportion of the food samples analysed in the present study with AFB1 levels exceeding the EU limit suggested that Ivoirians are at risk of chronic aflatoxin exposure.

Concentrations up to around 2000 (in peanut paste), 40 (in maize) and 7 (in rice) fold more than the EU AFB1 and AFT limits can also induce severe acute forms of aflatoxicosis. Previous studies have also reported very high aflatoxin levels in peanut, maize and rice samples from African countries. AFT and AFB1 concentrations up to 11900 $\mu g.kg^{-1}$ and 937 $\mu g.kg^{-1}$, respectively, were found in peanut samples (Chala, Mohammed, Ayalew, & Skinnes, 2013; Kamika & Takoy, 2011; Mutegi, Ngugi, Hendriks, & Jones, 2009). AFB1 levels up to 1081 $\mu g.kg^{-1}$ were reported in maize samples (Kamala et al., 2015) and up to 1642 $\mu g.kg^{-1}$ in mouldy rice samples (Makun et al., 2007). In Côte d'Ivoire, Sangare-Tigori, Moukha, et al. (2006) detected AFB1 in 100% of maize, rice and peanut samples from markets in Abidjan but with lower levels (up to 20 $\mu g.kg^{-1}$) than those quantified in the present study. Contrariwise, higher average levels of AFB1 (108 $\mu g.kg^{-1}$) and AFT (129 $\mu g.kg^{-1}$) with ranges of 5.7-309 $\mu g.kg^{-1}$ and 4.5-330 $\mu g.kg^{-1}$, respectively, were found in maize flour samples from markets in Abidjan, with 100% of the samples contaminated (Kouadio, Lattanzio, Ouattara, Kouakou, & Visconti, 2014). These latter researchers also detected AFB2, AFG1 and AFG2 at lower frequency and concentration than those of AFB1 but at higher average levels (8 $\mu g.kg^{-1}$ for both AFB2 and AFG1 and 6 $\mu g.kg^{-1}$ for AFG2) than those of the maize flour samples analysed in the present study.

Ochratoxin A (OTA) was detected in 65%, 22% and 15% of the peanut paste, maize and rice samples respectively, with only one maize sample and 3 rice samples exceeding the EU limit of 3 $\mu g.kg^{-1}$. For the cereal samples, the highest OTA level and mean were found in the rice samples (Table 4). OTA has been classified as a group 2B possible human carcinogen (IARC, 1993) and was associated with the Balkan Endemic Nephropathy (BEN). It is also thought to be teratogenic, hepatotoxic, neurotoxic and immunotoxic (Kőszegi & Poór, 2016). Sangare-Tigori, Dem, et al. (2006) reported OTA average levels higher than those found in the present study, in maize (mean 37 $\mu g.kg^{-1}$, range 9.8-86 $\mu g.kg^{-1}$), rice (mean 44 $\mu g.kg^{-1}$, range 9-92 $\mu g.kg^{-1}$) and peanuts (mean 23 $\mu g.kg^{-1}$, range 0.6-64 $\mu g.kg^{-1}$) from Côte d'Ivoire, whereas Sangare-Tigori, Moukha, et al. (2006) quantified OTA levels ranging from 0.09 to 0.86 $\mu g.kg^{-1}$ in maize, from 0.16 to 0.92 $\mu g.kg^{-1}$ in rice and from 0.20 to 0.64 $\mu g.kg^{-1}$ in peanuts. The frequency of OTA occurrence (30%) in the maize flour samples analysed in the present study was higher than that (13%) reported by Kouadio et al. (2014) in Côte d'Ivoire but the mean (1.9 $\mu g.kg^{-1}$ against 21 $\mu g.kg^{-1}$) and range (<LOQ to 7.6 $\mu g.kg^{-1}$ against not detected to 114 $\mu g.kg^{-1}$) were lower. In other African countries, OTA was found in peanut samples at lower concentrations than those quantified in the present study, but not or seldom in maize and rice samples (Abia et al., 2013; Kamala et al., 2015; Shephard et al., 2013; Toffa et al., 2013). There was the exception of rice samples from Nigeria for which more than 40% were contaminated with OTA at levels up to 1164 $\mu g.kg^{-1}$ (Makun et al., 2011, 2007) and of maize

samples from South Africa for which 68% contained OTA at concentrations up to 194 µg.kg^{-1} (Chilaka et al., 2012).

Fusarium mycotoxins are frequently detected in cereals and derived products (AFSSA, 2006). FB1 has been classified as a group 2B possible human carcinogen (IARC, 2002) and associated with oesophageal cancer and neural tube defects in humans. FB2 and FB3 toxicity is at the same level of magnitude than that of FB1 (Stockmann-Juvala & Savolainen, 2008). Zearalenone (ZEN) has been implicated in hyper estrogenic syndromes in humans and it has also been shown to be hepatotoxic, haematotoxic, immunotoxic and genotoxic (Zinedine, Soriano, Moltó, & Mañes, 2007). The maize samples analysed in the present study were more contaminated with FB1+FB2 than the rice samples but without exceeding the EU limit of 1000 µg.kg^{-1} (Table 5). FB1 levels were higher than FB2 in all the cereal samples (respective means 78 and 28 µg.kg^{-1} for maize samples; 5.3 and 2.2 µg.kg^{-1} for rice samples). Very few maize and rice samples were contaminated with ZEN and at very low concentrations not exceeding the EU limit of 75 µg.kg^{-1}. Fumonisins (FUMs) and ZEN were not detected in the peanut paste samples (Table 5). Warth et al. (2012) and Mulunda et al. (2013) reported similar results in peanut samples that can be explained by the fact that FUMs and ZEN are produced by *Fusarium* spp. which primarily infect cereal grains, in particular maize (Udomkun et al., 2017). In Côte d'Ivoire, Sangare-Tigori, Moukha, et al. (2006) found FB1 levels in maize samples (range 0.3-1.5 µg.kg^{-1}) much lower than those analysed in the present study (range 10-587 µg.kg^{-1}) and they did not detect FB1 in rice samples, whereas they quantified FB1 levels ranging from <0.3 to 6 µg.kg^{-1} in peanut samples. Contrariwise, Kouadio et al. (2014) determined higher FB1+FB2 levels (mean 356 µg.kg^{-1}, range 93-2210 µg.kg^{-1}) in maize flour samples. Sangare-Tigori, Moukha, et al. (2006) also analysed higher ZEN concentrations in maize (50 µg.kg^{-1}), rice and peanut samples (range 50-200 µg.kg^{-1}) as well as Kouadio et al. (2014) with ZEN determined in 57% of maize flour samples (mean 14 µg.kg^{-1}; range not detected-109 µg.kg^{-1}). In other African countries, higher ZEN contamination frequencies and levels were also found in rice (52%, up to 42 µg.kg^{-1}) and maize (15-92%, up to 135-811 µg.kg^{-1}) samples. In addition, higher FB1+FB2 contamination frequencies and levels have been reported in maize than rice samples with FB1 concentrations higher than FB2 in maize samples, but with higher FUM concentrations reaching up to 323948 µg.kg^{-1} for FB1 and 123814 µg.kg^{-1} for FB2 (Abia et al., 2013; Chilaka et al., 2012; Makun et al., 2011 ; Mulunda et al., 2013; Ogara et al., 2016; Shephard et al., 2013; Warth et al., 2012).

Among the rice samples, the local rice samples were the most contaminated with EU regulated mycotoxins. Except AFG2 that was not detected in the maize flour samples with potash, AFB1, AFG1, AFB2, OTA, FB1+FB2 and ZEN were detected in all types of maize samples. Maize flour

samples with potash had the lowest levels of AFB1, AFT and FB1+FB2 but the highest OTA and ZEN concentrations with one sample having an OTA level exceeding the EU limit (Tables 3-5).

3.3 Other mycotoxins

A total of 16 additional mycotoxins were detected in the analysed food samples (Table 6). Ten were recovered from the peanut paste samples including: beauvericin (BEA), equisetin (EQUS), aflatoxin M1 (AFM1), cyclopiazonic acid (CPA) and sterigmatocystin (STER) with each in more than 70% of the samples; ochratoxin B (OTB), enniatin B (ENN-B) and B1 (ENN-B1) at a frequency of 42%, 25% and 30% respectively; diacetoxyscirpenol (DAS) and aurofusarin (AUF) with each in less than 10% of the samples. Those toxins detected in the rice samples included: EQUS and BEA in 70% and 57% of the samples, respectively; AFM1, CPA, STER, citrinin (CIT), OTB, DAS, skyrin (SKY) and cytochalasin B (CYT-B) with each in less than 10% of the samples. Nine toxins were determined in the maize samples including: BEA, FB3, EQUS and AFM1 in 87%, 86%, 47%, and 37% of the samples, respectively; fusaric acid (FA) and CIT each in 46% of the samples; OTB, CPA and agroclavine (AGC) at a frequency comprised between 5 and 14%.

AFM1 was classified as a group 2B possible human carcinogen (IARC, 1993). Its acute toxicity is nearly equal to that of AFB1, but its potential carcinogenic hazard is about one order of magnitude less than that of AFB1 (Pietri & Piva, 2007). AFM1 is excreted into the milk of dairy animals and human nursing mothers after AFB1 ingestion and bioconversion in the liver. Its occurrence in plants, produced by *Aspergillus* spp. through a biosynthetic pattern not involving AFB1 or possibly by insect pests' metabolism from AFB1, was also reported (Giovati et al., 2015). This could explain AFM1 presence in the cereal and peanut paste samples analysed in the present study, with the peanut paste samples being the most contaminated with this toxin. Warth et al. (2012) have also detected AFM1 in maize samples from African countries at higher concentrations, as well as Ogara et al. (2016) up to 1744 $\mu g.kg^{-1}$, but not in peanut samples.

OTB, CIT and CPA are the three non EU regulated mycotoxins produced by *Aspergillus* / *Penicillium* spp. that were found in the analysed food samples. OTB which is clearly less toxic than OTA (Heussner & Bingle, 2015), was detected in all types of samples except maize flour samples without potash. Contamination frequency and levels were the highest in the peanut paste samples and low in the cereal samples. Abia et al. (2013) quantified lower OTB levels in peanut samples and did not detect this mycotoxin in maize samples. CIT is reported to possess nephrotoxic, cytotoxic and genotoxic effects. It is frequently found in food in combination with OTA, and suspected to be involved in the BEN (Flajs & Peraica, 2009; Föllmann, Behm, & Degen, 2014). CIT was found in all types of cereal samples but not in peanut paste samples. Previous studies in Africa also reported no CIT in peanut samples but levels up to 44029 $\mu g.kg^{-1}$ in maize samples (Abia et al., 2013; Ogara

et al., 2016; Warth et al., 2012). CPA was detected in all types of samples except the local rice samples, with the highest contamination frequency and levels in peanut paste samples, and concentrations above the LOQ only in 4 maize flour samples. This mycotoxin was quantified at higher concentrations (up to 1347 µg.kg^{-1}, mean 249 µg.kg^{-1}) in maize samples from Nigeria (Ogara et al., 2016) and in none or only one peanut sample from Burkina Faso and Mozambique (Warth et al., 2012). Eight non EU regulated mycotoxins produced by *Fusarium* spp. were detected in the analysed food samples. BEA was detected in all types of samples at high frequencies of occurrence (>40%), with the highest found in the peanut paste samples followed by the maize and rice samples, and at concentrations not exceeding 22 µg.kg^{-1}. ENN-B and ENN-B1 were detected at low concentrations only in peanut paste samples, as well as AUF. BEA and ENNs were found to produce cytotoxic effects in several mammalian cell lines (Jestoi, 2008; Prosperini, Meca, Font, & Ruiz, 2012). DAS was detected only in 2 local rice samples and 4 peanut paste samples at levels up to 4 µg kg^{-1}. EQUS was found in all types of samples with the highest contamination frequency and levels in the peanut paste samples. FB3 and FA were detected only in the maize samples at levels up to 94 and 83 µg.kg^{-1}, respectively. Previous studies performed in African countries have reported higher levels of BEA, EQUS, FB3 and FA in maize samples (respectively up to 3724 µg.kg^{-1}, 242 µg.kg^{-1}, 63509 µg.kg^{-1} and 19466 µg.kg^{-1}) but also AUF and DAS levels respectively up to 3878 µg.kg^{-1} and 34 µg.kg^{-1}, and very low ENN-B and ENN-B1 concentrations not exceeding 0.4 µg.kg^{-1} and 1 µg.kg^{-1}, respectively (Abia et al., 2013; Ogara et al., 2016; Shephard et al., 2013). Abia el al. (2013) quantified higher levels of BEA (up to 12 µg.kg^{-1}, mean 4 µg.kg^{-1}) and ENN-B1 (up to 5 µg.kg^{-1}, mean 1.4 µg.kg^{-1}) and lower ENN-B levels (up to 0.6 µg.kg^{-1}, mean 0.1µg.kg^{-1}) in peanut samples as well as FB3 and FA levels up to 5 µg.kg^{-1} and 189 µg.kg^{-1}, respectively. AGC was the *Claviceps* mycotoxin detected only in the maize samples at low concentrations. This toxin was also quantified by Shephard et al., (2013) in maize samples from South Africa at low concentrations (up to 1.8 µg.kg^{-1}). STER which is the most toxic AFB1 precursor, is both mutagenic and tumorigenic but less potent than AFs. It has been classified as a group 2B possible human carcinogen (Bennett & Klich, 2003; Ezekiel, Sulyok, Warth, Odebode, & Krska, 2012). STER was detected only in rice and peanut paste samples, with the latter being the most contaminated in frequency and levels. Contrariwise, Abia et al. (2013) and Ogara et al. (2016) found STER up to 242 µg.kg^{-1} in maize samples, respectively from Cameroon and Nigeria, and Warth et al. (2012) did not detect this toxin in peanut samples from Burkina Faso and in only one from Mozambique. SKY and CYT-B were detected in only one and 3 imported rice samples, respectively. SKY was found by Shephard et al. (2013) in 94% of maize samples from South Africa at levels up to 60 µg.kg^{-1}. Among the rice samples, the local rice samples were the most contaminated by the non EU regulated mycotoxins, except for OTB, STER, SKY and CYT-B.

4. Conclusion

The present study is the first to provide data on such a wide range of mycotoxins in the cereal (rice grains, cracked maize and maize flour) and oilseed (peanut paste) products most consumed in Côte d'Ivoire. It revealed that these food products were highly susceptible to contamination by several EU regulated and/or other mycotoxins. Furthermore, mycotoxin concentrations exceeding EU limits were found, especially for AFB1 which is the most toxic form of AFs and the most frequently detected mycotoxin in the samples. The low concentrations found for certain mycotoxins in the analysed food samples do not exclude potential toxic risks to Ivorian consumers due to a cumulative effect depending on their dietary habits. In addition, although data on the toxic effects of non EU regulated mycotoxins as well as on the possible interactions between mycotoxins are still limited or non-existent, the occurrence of the non EU regulated mycotoxins in foods is a concern and exposure to multiple mycotoxins, especially through high-consumption food products, is expected to result in greater human health problems. Therefore, the foods analysed in this study pose a risk to the health of Ivorian consumers, with the peanut paste samples representing the highest risk in terms of number of mycotoxins detected in the same sample, frequency and levels of contamination, followed by maize then rice samples. The local rice samples were the most contaminated compared to the imported ones. This can be explained by a better quality of rice exported by Asian countries and /or by the fact that imported rice is subject to quality controls before entering the market in Côte d'Ivoire.

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References

Abia, W. A., Warth, B., Sulyok, M., Krska, R., Tchana, A. N., Njobeh, P. B., Moundipa, P. F. (2013). Determination of multi-mycotoxin occurrence in cereals, nuts and their products in

Cameroon by liquid chromatography tandem mass spectrometry (LC-MS/MS). *Food Control*, *31*(2), 438–453. https://doi.org/10.1016/j.foodcont.2012.10.006

AFSSA (Agence française de sécurité sanitaire des aliments). (2006). Risk assessment for mycotoxins in human and animal food chains. Retrieved from https://www.anses.fr/fr/system/files/RCCP-Ra-MycotoxinesEN.pdf

Alassane-Kpembi, I., Schatzmayr, G., Taranu, I., Marin, D., Puel, O., & Oswald, I. P. (2017). Mycotoxins co-contamination: Methodological aspects and biological relevance of combined toxicity studies. *Critical Reviews in Food Science and Nutrition*, *57*(16), 3489–3507. https://doi.org/10.1080/10408398.2016.1140632

Bennett, J. W., & Klich, M. (2003). Mycotoxins. *Clinical Microbiology Reviews*, *16*(3), 497–516. https://doi.org/10.1128/CMR.16.3.497-516.2003

Boansi, D. (2013). Acreage response of rice in Côte D'ivoire: Macro-level response and some policy implications. *International Journal of Agricultural Policy and Research*, *1*(9), 288–297.

Chala, A., Mohammed, A., Ayalew, A., & Skinnes, H. (2013). Natural occurrence of aflatoxins in groundnut (Arachis hypogaea L.) from eastern Ethiopia. *Food Control*, *30*(2), 602–605. https://doi.org/10.1016/j.foodcont.2012.08.023

Chilaka, C. A., De Kock, S., Phoku, J. Z., Mwanza, M., Egbuta, M. A., & Dutton, M. F. (2012). Fungal and mycotoxin contamination of South African commercial maize. *Journal of Food, Agriculture & Environment Vol.10 (2): 296-303*

EFSA (European Food Safety Authority). (2007). Opinion of the scientific panel on contaminants in the food chain on a request from the commission related to the potential increase of consumer health risk by a possible increase of the existing maximum levels for aflatoxins in almonds, hazelnuts and pistachios and derived products. Question N° EFSA-Q-2006-174. *EFSA Journal*, (446), 1–127.

EFSA (European Food Safety Authority) Panel on Contaminants in the Food Chain (CONTAM). (2014a). Scientific Opinion on the risks for human and animal health related to the presence of modified forms of certain mycotoxins in food and feed: Modified mycotoxins in food and feed. *EFSA Journal*, *12*(12), 3916. https://doi.org/10.2903/j.efsa.2014.3916

EFSA (European Food Safety Authority) Panel on Contaminants in the Food Chain (CONTAM). (2014b). Scientific Opinion on the risks to human and animal health related to the presence of beauvericin and enniatins in food and feed: Beauvericin and enniatins in food and feed. *EFSA Journal*, *12*(8), 3802. https://doi.org/10.2903/j.efsa.2014.3802

European Commission. (2006). Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs (Text with EEA relevance). *Official Journal of European Union*, (364), 5–24.

European Commission. (2007). Commission Regulation (EC) No 1126/2007, amending Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs as regards Fusarium toxins in maize and maize products (Text with EEA relevance). *Official Journal of European Union*, (255), 14–16.

European Commission. (2010). Commission regulation (EU) No 165/2010 of 26 February 2010, amending Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs as regards aflatoxins (Text with EEA relevance). *Official Journal of European Union*, (50), 8.

European Commission. (2012). Commission regulation (EU) No 594/2012/EU of 5 July 2012 amending Regulation (EC) 1881/2006 as regards the maximum levels of the contaminants ochratoxin A, non-dioxin-like PCBs and melamine in foodstuffs (Text with EEA relevance). *Official Journal of European Union*, (176), 43–45.

European Commission. (2013). Recommendations (EU) No 165/2013 of 27 March 2013 on the presence of T-2 and HT-2 toxin in cereals and cereal products (Text with EEA relevance). *Official Journal of European Union*, (91), 12–15.

Ezekiel, C. N., Sulyok, M., Warth, B., Odebode, A. C., & Krska, R. (2012). Natural occurrence of mycotoxins in peanut cake from Nigeria. *Food Control*, *27*(2), 338–342. https://doi.org/10.1016/j.foodcont.2012.04.010

Flajs, D., & Peraica, M. (2009). Toxicological Properties of Citrinin. *Archives of Industrial Hygiene and Toxicology*, *60*(4). https://doi.org/10.2478/10004-1254-60-2009-1992

Föllmann, W., Behm, C., & Degen, G. H. (2014). Toxicity of the mycotoxin citrinin and its metabolite dihydrocitrinone and of mixtures of citrinin and ochratoxin A in vitro. *Archives of Toxicology*, *88*(5), 1097–1107. https://doi.org/10.1007/s00204-014-1216-8

Giovati, L., Magliani, W., Ciociola, T., Santinoli, C., Conti, S., & Polonelli, L. (2015). AFM1 in Milk: Physical, Biological, and Prophylactic Methods to Mitigate Contamination. *Toxins*, *7*(10), 4330–4349. https://doi.org/10.3390/toxins7104330

Grenier, B., & Oswald, I. (2011). Mycotoxin co-contamination of food and feed: meta-analysis of publications describing toxicological interactions. *World Mycotoxin Journal*, *4*(3), 285–313. https://doi.org/10.3920/WMJ2011.1281

Heussner, A., & Bingle, L. (2015). Comparative Ochratoxin Toxicity: A Review of the Available Data. *Toxins*, *7*(10), 4253–4282. https://doi.org/10.3390/toxins7104253

IARC (International Agency for Research on Cancer). (1993). IARC monographs on the evaluation of carcinogenic risks to humans. *Some naturally occurring substances: food items and constituents, heterocyclic aromatic amines and mycotoxins.* Volume 56, 599 p., IARC, Lyon, France.

IARC (International Agency for Research on Cancer). (2002). IARC monographs on the evaluation of carcinogenic risks to humans. *Some traditional herbal medicines, some mycotoxins, naphthalene and styrene.*Volume 82, 601 p. IARC, Lyon: France.

IARC (International Agency for Research on Cancer). (2015). *Mycotoxin control in low- and middle-income countries*. Retrieved from http://www.ncbi.nlm.nih.gov/books/NBK350558/

Jestoi, M. (2008). Emerging *Fusarium* -Mycotoxins Fusaproliferin, Beauvericin, Enniatins, And Moniliformin—A Review. *Critical Reviews in Food Science and Nutrition*, *48*(1), 21–49. https://doi.org/10.1080/10408390601062021

Kamala, A., Ortiz, J., Kimanya, M., Haesaert, G., Donoso, S., Tiisekwa, B., & De Meulenaer, B. (2015). Multiple mycotoxin co-occurrence in maize grown in three agro-ecological zones of Tanzania. *Food Control*, *54*, 208–215. https://doi.org/10.1016/j.foodcont.2015.02.002

Kamika, I., & Takoy, L. L. (2011). Natural occurrence of Aflatoxin B1 in peanut collected from Kinshasa, Democratic Republic of Congo. *Food Control*, *22*(11), 1760–1764. https://doi.org/10.1016/j.foodcont.2011.04.010

Kőszegi, T., & Poór, M. (2016). Ochratoxin A: Molecular Interactions, Mechanisms of Toxicity and Prevention at the Molecular Level. *Toxins*, *8*(4), 111. https://doi.org/10.3390/toxins8040111

Kouadio, J. H., Lattanzio, V. M. T., Ouattara, D., Kouakou, B., & Visconti, A. (2014). Assessment of mycotoxin exposure in Côte D'Ivoire (Ivory Coast) through multi-biomarker analysis and possible correlation with food consumption patterns. *Toxicol Int*, *21*(3), 248–257. https://doi.org/10.4103/0971-6580.155336

Makun, H. A., Gbodi, T. A., Akanya, O. H., Salako, E. A., & Ogbadu, G. H. (2007). Fungi and some mycotoxins contaminating rice (Oryza sativa) in Niger state, Nigeria. *African Journal of Biotechnology*, *6*(2). Retrieved from https://www.ajol.info/index.php/ajb/article/view/56106

Makun, H. A., Dutton, M. F., Njobeh, P. B., Mwanza, M., & Kabiru, A. Y. (2011). Natural multi-occurrence of mycotoxins in rice from Niger State, Nigeria. *Mycotoxin Research*, *27*(2), 97‑104. https://doi.org/10.1007/s12550-010-0080-5

Mulunda, M., Dzoma, B., Nyirenda, M., & Bakunzi, F. (2013). Mycotoxins occurrence in selected staple food in main markets from Lubumbashi, Democratic Republic of Congo. *Journal of Food, Agriculture & Environment*, *11*(3&4), 51–54.

Mutegi, C. K., Ngugi, H. K., Hendriks, S. L., & Jones, R. B. (2009). Prevalence and factors associated with aflatoxin contamination of peanuts from Western Kenya. *International Journal of Food Microbiology*, *130*(1), 27–34. https://doi.org/10.1016/j.ijfoodmicro.2008.12.030

Ogara, I. M., Zarafi, A. B., Alabi, O., Banwo, O., Ezekiel, C. N., Warth, B., Krska, R. (2016). Mycotoxin patterns in ear rot infected maize: A comprehensive case study in Nigeria. *Food Control*, *73*, 1159–1168. https://doi.org/10.1016/j.foodcont.2016.10.034

Oplatowska-Stachowiak, M., Haughey, S. A., Chevallier, O. P., Galvin-King, P., Campbell, K., Magowan, E., Elliott, C. T. (2015). Determination of the Mycotoxin Content in Distiller's Dried Grain with Solubles Using a Multianalyte UHPLC–MS/MS Method. *Journal of Agricultural and Food Chemistry*, *63*(43), 9441–9451. https://doi.org/10.1021/acs.jafc.5b03844

Pietri, A., & Piva, G. (2007). Aflatoxins in foods. *Italian Journal of Public Health*, *4*(1). Retrieved from http://ijphjournal.it/article/view/5899

Prosperini, A., Meca, G., Font, G., & Ruiz, M. J. (2012). Study of the cytotoxic activity of beauvericin and fusaproliferin and bioavailability in vitro on Caco-2 cells. *Food and Chemical Toxicology*, *50*(7), 2356–2361. https://doi.org/10.1016/j.fct.2012.04.030

Rychlik, M., Humpf, H.-U., Marko, D., Dänicke, S., Mally, A., Berthiller, F., Lorenz, N. (2014). Proposal of a comprehensive definition of modified and other forms of mycotoxins including 'masked' mycotoxins. *Mycotoxin Research*, *30*(4), 197–205. https://doi.org/10.1007/s12550-014-0203-5

Sangare, A., Koffi, E., Akamou, F., & Fall, C. A. (2009). Rapport national sur l'état des ressources phytogénétiques pour l'alimentation et l'agriculture. Ministère de l'agriculture, République de Côte d'Ivoire. 65p.

Sangare-Tigori, Dem, A., Kouadio, H., Betbeder, A.-M., Dano, D., Moukha, S., & Creppy, E. (2006). Preliminary survey of ochratoxin A in millet, maize, rice and peanuts in Côte d'Ivoire from 1998 to 2002. *Human & Experimental Toxicology*, *25*(4), 211–216. https://doi.org/10.1191/0960327106ht605oa

Sangare-Tigori, Moukha, S., Kouadio, H. J., Betbeder, A.-M., Dano, D. S., & Creppy, E. E. (2006). Co-occurrence of aflatoxin B_1, fumonisin B_1, ochratoxin A and zearalenone in cereals and peanuts from Côte d'Ivoire. *Food Additives and Contaminants*, *23*(10), 1000–1007. https://doi.org/10.1080/02652030500415686

Shephard, G. S., Burger, H.-M., Gambacorta, L., Krska, R., Powers, S. P., Rheeder, J. P., … van der Westhuizen, L. (2013). Mycological Analysis and Multimycotoxins in Maize from Rural Subsistence Farmers in the Former Transkei, South Africa. *Journal of Agricultural and Food Chemistry*, *61*(34), 8232–8240. https://doi.org/10.1021/jf4021762

Stockmann-Juvala, H., & Savolainen, K. (2008). A review of the toxic effects and mechanisms of action of fumonisin B1. *Human & Experimental Toxicology*, *27*(11), 799–809. https://doi.org/10.1177/0960327108099525

Toffa, D. D., Mahnine, N., Ouaffak, L., Abidi, A. El, Alaoui Faris, F. Z. El, & Zinedine, A. (2013). First survey on the presence of ochratoxin A and fungi in raw cereals and peanut available in the Republic of Niger. *Food Control*, *32*(2), 558–562. https://doi.org/10.1016/j.foodcont.2013.01.028

Udomkun, P., Wiredu, A. N., Nagle, M., Bandyopadhyay, R., Müller, J., & Vanlauwe, B. (2017). Mycotoxins in Sub-Saharan Africa: Present situation, socio-economic impact, awareness, and outlook. *Food Control*, *72*, 110–122. https://doi.org/10.1016/j.foodcont.2016.07.039

USDA (United States Departement of Agriculture), Foreign Agricultural Service. 2015 Update West Africa Rice Annual. Grain and Feed Annual. Gain report, *Global Agricultural Information Network*, Senegal, 04/29/2015, 18 p.

Van Egmond, H. P., Schothorst, R. C., & Jonker, M. A. (2007). Regulations relating to mycotoxins in food: Perspectives in a global and European context. *Analytical and Bioanalytical Chemistry*, *389*(1), 147–157. https://doi.org/10.1007/s00216-007-1317-9

Warth, B., Parich, A., Atehnkeng, J., Bandyopadhyay, R., Schuhmacher, R., Sulyok, M., & Krska, R. (2012). Quantitation of Mycotoxins in Food and Feed from Burkina Faso and Mozambique Using a Modern LC-MS/MS Multitoxin Method. *Journal of Agricultural and Food Chemistry*, *60*(36), 9352–9363. https://doi.org/10.1021/jf302003n

Yeo, Y. A. (2011). Analyse de la compétitivité du maïs local en Côte d'Ivoire. PRESAO (*Programme de Renforcement et de Recherche sur la Sécurité Alimentaire en Afrique de l'Ouest)*. Composante SRAI (Strengthening Regional Agricultural Integration in West Africa), Résumé N°2, 2011-12, Maïs, Décembre 2011, 10 p. Retrieved from http://fsg.afre.msu.edu/srai/index.htm

Zain, M. E. (2011). Impact of mycotoxins on humans and animals. *Journal of Saudi Chemical Society*, *15*(2), 129–144. https://doi.org/10.1016/j.jscs.2010.06.006

Zinedine, A., Soriano, J. M., Moltó, J. C., & Mañes, J. (2007). Review on the toxicity, occurrence, metabolism, detoxification, regulations and intake of zearalenone: An oestrogenic mycotoxin. Food and Chemical Toxicology, 45(1), 1–18. https://doi.org/10.1016/j.fct.2006.07.030

Table 1: Limits of detection and quantification of the UHPLC-MS/MS method used for the analysis of 77 mycotoxins in rice, maize and peanut samples collected in Côte d'Ivoire

Mycotoxins / Abbreviation	Toxin Source	Limit of detection (LOD) in solvent [µg.kg^{-1}]	Limit of quantification (LOQ) in solvent [µg.kg^{-1}]
EU regulated mycotoxins in foodstuffs			
Aspergillus mycotoxins			
Aflatoxin B1 / AFB1	Romer Labs	0.05	0.25
Aflatoxin B2 / AFB2	Sigma	0.12	0.25
Aflatoxin G1 / AFG1		0.12	0.25
Aflatoxin G2 / AFG2		0.12	0.25
Aflatoxin M1 / AFM1		0.05	0.25
Fusarium mycotoxins			
Deoxynivalenol / DON	Romer Labs	5	12.5
Fumonisin B1 / FB1	Sigma	0.5	1
Fumonisin B2 / FB2	Sigma	0.5	1
Zearalenone / ZEN	Romer Labs	2.5	5
Aspergillus / *Penicillium* mycotoxins			
Citrinin / CIT	Tebu-Bio	10	25
Ochratoxin A / OTA	Romer Labs	0.25	0.62
Patulin	Fisher	50	125
***Fusarium* mycotoxins with EU recommended limits in cereal and cereal products**			
T-2 toxin	Romer Labs	2.5	5
HT-2 toxin	EnzoLife Sciences	25	50
Metabolites of EU regulated *Fusarium* mycotoxins			
3-acetyldeoxynivalenol / 3-AcDON	Romer Labs	12.5	25
15-AcDON	Romer Labs	25	50
Deepoxy-DON	Sigma	2.5	5
Zearalanone	Tebu-Bio	5	10
α-zearalanol / α-ZAL		125	250
β-ZAL		125	250
α-zearalenol / α-ZEL		50	125
β-ZEL		50	125
Emerging *Fusarium* mycotoxins			
Beauvericin / BEA	Romer Labs	0.005	0.01
Enniatin A	Tebu-Bio	0.02	0.05
Enniatin A1		0.02	0.05
Enniatin B / ENN-B		0.02	0.05
Enniatin B1 / ENN-B1		0.02	0.05
Moniliformin / MON		62.5	125
Fusarium* mycotoxins conjugated by plants (masked mycotoxins) or fungi			
DON-3-glucoside / DON-3-Glc	Romer Labs	125	250
ZEN-14-Glc	Department of Agrobiotechnology, IFA-Tulln	5	12.5
α-ZEL-14-Glc		25	50
β-ZEL-14-Glc		12.5	25
ZEN-16-Glc		1	2.5
ZEN-14-sulfate*		1	2.5
Other *Fusarium* mycotoxins			
Apicidin	EnzoLife Sciences	5	10
Aurofusarin / AUF	Insight Biotechnology	0.62	1.25
Diacetoxyscirpenol / DAS	Discovery Fine Chemicals	1.25	2.5
Equisetin / EQUS	Insight Biotechnology	0.25	0.62
Fumonisin B3 / FB3	Romer Labs	0.5	1
Fusarenon X		12.5	25
Fusaric acid / FA	Fisher Scientific	2.5	5
Neosolaniol	Romer Labs	1	2.5
Nivalenol		62.5	125
Verrucarol	Sigma	25	50

***Penicillium* mycotoxins**			
Curvularin	Insight Biotechnology	2.5	5
Meleagrin	Tebu-Bio	0.06	0.12
Mycophenolic acid		0.62	1.25
Penicillic acid	Cambridge Biosciences	5	10
Roquefortine C	Romer Labs	0.05	0.12
***Aspergillus* / *Penicillium* mycotoxins**			
Cyclopiazonic acid / CPA	Abcam	2.5	6.25
Ochratoxin B / OTB	Insight Biotechnology	0.25	0.62
Verruculogen	Romer Labs	0.62	1.25
***Alternaria* mycotoxins**			
Altenuene	Analyticon Discovery	2.5	5
Alternariol	Insight Biotechnology	2.5	5
Alternariolmonomethylether		5	10
Macrosporin	EnzoLife Sciences	25	50
Tentoxin		1.25	2.5
***Claviceps* mycotoxins (ergot alkaloids)**			
Agroclavine / AGC	Romer Labs	0.25	0.62
Ergocornine		0.62	1.25
Ergocorninine		0.02	0.06
Ergocristine		0.62	1.25
Ergocristinine		0.02	0.06
Ergocryptine		0.25	0.62
Ergocryptinine		0.02	0.06
Ergometrine		0.12	0.25
Ergometrinine		0.06	0.12
Ergosine		0.25	0.62
Ergosinine		0.06	0.12
Ergotamine		0.12	0.25
Ergotaminine		0.06	0.12
Other fungal mycotoxins			
Cytochalasin B / CYT-B	Tebu-Bio	1.25	2.5
Emodin	Cambridge Biosciences	25	50
Gliotoxin	Tebu-Bio	2.5	5
Penitrem A		0.5	1
Skyrin / SKY	EnzoLife Sciences	10	25
Stachybotrylactam	Insight Biotechnology	0.25	0.62
Sterigmatocystin / STER	EnzoLife Sciences	0.62	1.25

Table 2: Number of mycotoxins found in the same sample for the different types of food samples collected in Côte d'Ivoire

Food sample (n)	Minimum number of mycotoxins >1	Maximum number of mycotoxins	% (number) of samples according to number of mycotoxins detected in the same sample				
			0	1	2-4	5-7	≥ 8
Rice samples (88)	**2**	**8**	**14 (12)**	**10 (9)**	**49 (43)**	**25 (22)**	**2 (2)**
Imported rice (41)	2	6	29 (12)	20 (8)	39 (16)	12 (5)	-
Local rice (47)	2	8	-	2 (1)	58 (27)	36 (17)	4 (2)
Maize sample (79)	**2**	**14**	**-**	**-**	**10 (8)**	**32 (25)**	**58 (46)**
Cracked maize (29)	2	12	-	-	10 (3)	38 (11)	52 (15)
Maize flour with potash (32)	2	14	-	-	16 (5)	34 (11)	50 (16)
Maize flour without potash (18)	6	14	-	-	-	17 (3)	83 (15)
Peanut paste samples (71)	**5**	**13**	**-**	**-**	**-**	**6 (4)**	**94 (67)**

n = total number of collected samples

Table 3: Occurrence of aflatoxins B1 (AFB1) and G1 (AFG1), and total aflatoxins (AFT=AFB1+AFB2+AFG1+AFG2) in food samples collected in Côte d'Ivoire

Mycotoxins	Food sample (n)	Frequency % (n')	Mean (Range) µg.kg^{-1}	Number of contaminated samples according to mycotoxin levels (X) µg kg^{-1}						
				X<2	2*≤X<4	4**≤X<20	20≤X<50	50≤X<100	100≤X<500	X≥500
AFB1	Imported rice (41)	46 (19)	1.0 (<LOQ-2.9)	18	1	-	-	-	-	-
	Local rice (47)	66 (31)	3.9 (<LOQ-14)	11	8	12	-	-	-	-
	Cracked maize (29)	97 (28)	8.2 (0.3-80)	10	5	11	1	1	-	-
	Maize flour with potash (32)	94 (30)	7.2 (<LOQ-49)	15	5	6	4	-	-	-
	Maize flour without potash (18)	100 (18)	12 (0.3-57)	5	3	8	1	1	-	-
	Peanut paste (71)	100 (71)	260 (0.6-4535)	1	1	6	12	12	32	7
AFG1	Imported rice (41)	7 (3)	0.4 (0.3-0.7)	3	-	-	-	-	-	-
	Local rice (47)	34 (16)	3.3 (<LOQ -17)	10	3	3	-	-	-	-
	Cracked maize (29)	66 (19)	7.3 (0.3-84)	13	1	3	1	1	-	-
	Maize flour with potash (32)	38 (12)	1.9 (0.4-7.5)	8	3	1	-	-	-	-
	Maize flour without potash (18)	89 (16)	5.6 (0.5-11)	5	2	9	-	-	-	-
	Peanut paste (71)	100 (71)	143 (0.7-2194)	3	-	17	15	11	21	4
AFT	Imported rice (41)	46 (19)	1.1 (<LOQ-4.1)	18	-	1	-	-	-	-
	Local rice (47)	66 (31)	5.8 (<LOQ-33)	9	8	13	2	-	-	-
	Cracked maize (29)	97 (28)	14 (0.3-173)	9	3	12	3	-	1	-
	Maize flour with potash (32)	94 (30)	8.7 (<LOQ-56)	14	5	7	3	1	-	-
	Maize flour without potash (18)	100 (18)	18 (0.8-67)	2	3	4	8	1	-	-
	Peanut paste (71)	100 (71)	530 (1.4-8094)	1	-	4	3	12	35	16

n = total number of collected samples; n' = number of contaminated samples; *, ** EU limits respectively for AFB1 and AFT in cereals, peanuts and derived products intended for direct human consumption; <LOQ = less than the limit of quantification

Table 4: Ochratoxin A (OTA) occurrence in food samples collected in Côte d'Ivoire

Food sample (n)	Frequency % (n')	Mean (Range) µg.kg^{-1}	**Number of contaminated samples according to mycotoxin levels (X) µg kg^{-1}**				
			X<3*	3≤X<15	15≤X<30	30≤X<60	X≥60
Imported rice (41)	12 (5)	2.7 (<LOQ -7.9)	4	1	-	-	-
Local rice (47)	17 (8)	6.3 (<LOQ -15)	6	1	1	-	-
Cracked maize (29)	7 (2)	1.8 (1.8)	2	-	-	-	-
Maize flour with potash (32)	16 (5)	3.4 (<LOQ -7.6)	4	1	-	-	-
Maize flour without potash (18)	56 (10)	1.3 (0.7-1.9)	10	-	-	-	-
Peanut paste (71)	65 (46)	9.8 (<LOQ -147)	24	17	2	2	1

n = total number of collected samples; n' = number of contaminated samples; * EU limit for OTA in cereals and cereal products intended for direct human consumption, no EU limit for peanut products; <LOQ = less than the limit of quantification

Table 5: Occurrence of fumonisins B1 and B2 (FB1+FB2) and zearalenone (ZEN) in food samples collected in Côte d'Ivoire

Mycotoxins	**Food sample (n)**	**Frequency % (n')**	**Mean (Range) µg.kg^{-1}**
FB1+FB2	Imported rice (41)	2 (1)	2.6
	Local rice (47)	32 (15)	6.0 (2.7-13)
	Cracked maize (29)	97 (28)	99 (11-706)
	Maize flour with potash (32)	81 (26)	73 (14-214)
	Maize flour without potash (18)	100 (18)	162 (2.3-319)
	Peanut paste (71)	nd	nd
ZEN	Imported rice (41)	nd	nd
	Local rice (47)	9 (4)	6.6 (<LOQ -7.5)
	Cracked maize (29)	3 (1)	< LOQ
	Maize flour with potash (32)	13 (4)	6.6 (<LOQ -7.7)
	Maize flour without potash (18)	6 (1)	< LOQ
	Peanut paste (71)	nd	nd

n = total number of collected samples; n' = number of contaminated samples; nd = not detected, i.e. less than the limit of detection; <LOQ = less than the limit of quantification

Table 6: Occurrence of other mycotoxins, non EU regulated in cereals (maize and rice), peanuts and derived products intended for direct human consumption, in food samples collected in Côte d'Ivoire

Mycotoxins	Food sample											
	Imported rice (n=41)		Local rice (n=47)		Cracked maize (n=29)		Maize flour with potash (n=32)		Maize flour without potash (n=18)		Peanut paste (n=71)	
	F % (n')	Mean (range) µg.kg $^{-1}$	F % (n')	Mean (range) µg.kg $^{-1}$	F % (n')	Mean (range) µg.kg $^{-1}$	F % (n')	Mean (range) µg.kg $^{-1}$	F % (n')	Mean (range) µg.kg $^{-1}$	F % (n')	Mean (range) µg.kg $^{-1}$
Aflatoxin M1	2 (1)	0.4	13 (6)	0.5 (<LOQ-0.8)	38 (11)	1.1 (0.4-5.3)	25 (8)	0.9 (0.4-1.7)	56 (10)	0.9 (0.4-2.8)	99 (70)	20 (0.4-381)
Agroclavine	nd	nd	nd	nd	24 (7)	1.0 (<LOQ-1.4)	3 (1)	0.7	11 (2)	2.9 (2.4-3.5)	nd	nd
Aurofusarin	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	7 (5)	3.2 (2.6-4.2)
Beauvericin	41 (17)	0.3 (0.1-0.6)	70 (33)	1.2 (0.1-12)	97 (28)	2.7 (0.3-22)	94 (30)	1.9 (0.4-5.0)	61 (11)	1.6 (0.5-4.6)	99 (70)	1.5 (0.1-11)
Citrinin	2 (1)	29	13 (6)	56 (<LOQ-101)	52 (15)	57 (<LOQ-146)	56 (18)	386 (<LOQ-2394)	17 (3)	84 (52-122)	nd	nd
Cyclopiazonic acid	2 (1)	<LOQ	nd	nd	7 (2)	<LOQ	19 (6)	13 (<LOQ-19)	17 (3)	10 (<LOQ -15)	90 (64)	31 (<LOQ-132)
Cytochalasin B	7 (3)	4.3 (<LOQ-5.0)	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Diacetoxyscirpenol	nd	nd	4 (2)	2.9 (<LOQ-2.9)	nd	nd	nd	nd	nd	nd	6 (4)	4.0 (<LOQ-4.0)
Enniatin B	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	25 (18)	0.9 (0.4-2.6)
Enniatin B1	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	30 (21)	0.7 (0.3-2.4)
Equisetin	41 (17)	1.7 (<LOQ-6.2)	96 (45)	10 (0.6-115)	38 (11)	1.8 (0.7-4.1)	59 (19)	1.9 (<LOQ-4.7)	39 (7)	2.2 (<LOQ-3.8)	99 (70)	15 (<LOQ-140)
Fumonisin B3	nd	nd	nd	nd	86 (25)	17 (2.2-83)	78 (25)	14 (3.1-35)	100 (18)	25 (2.1-55)	nd	nd
Fusaric acid	nd	nd	nd	nd	24 (7)	23 (<LOQ-44)	53 (17)	30 (7.0-94)	67 (12)	22 (8.5-29)	nd	nd
Ochratoxin B	5 (2)	1.5 (<LOQ-1.5)	4 (2)	0.9 (0.7-1.1)	7 (2)	<LOQ	6 (2)	1.1 (<LOQ-1.1)	nd	nd	42 (30)	19 (0.7-260)
Skyrin	2 (1)	50	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Sterigmatocystin	7 (3)	4.7 (<LOQ-7.8)	9 (4)	2.6 (<LOQ-4.8)	nd	Nd	nd	nd	nd	nd	70(50)	8.1 (<LOQ-40)

n = total number of collected samples; n' = number of contaminated samples; F = frequency; nd = not detected, i.e. less than the limit of detection; <LOQ = less than the limit of quantification

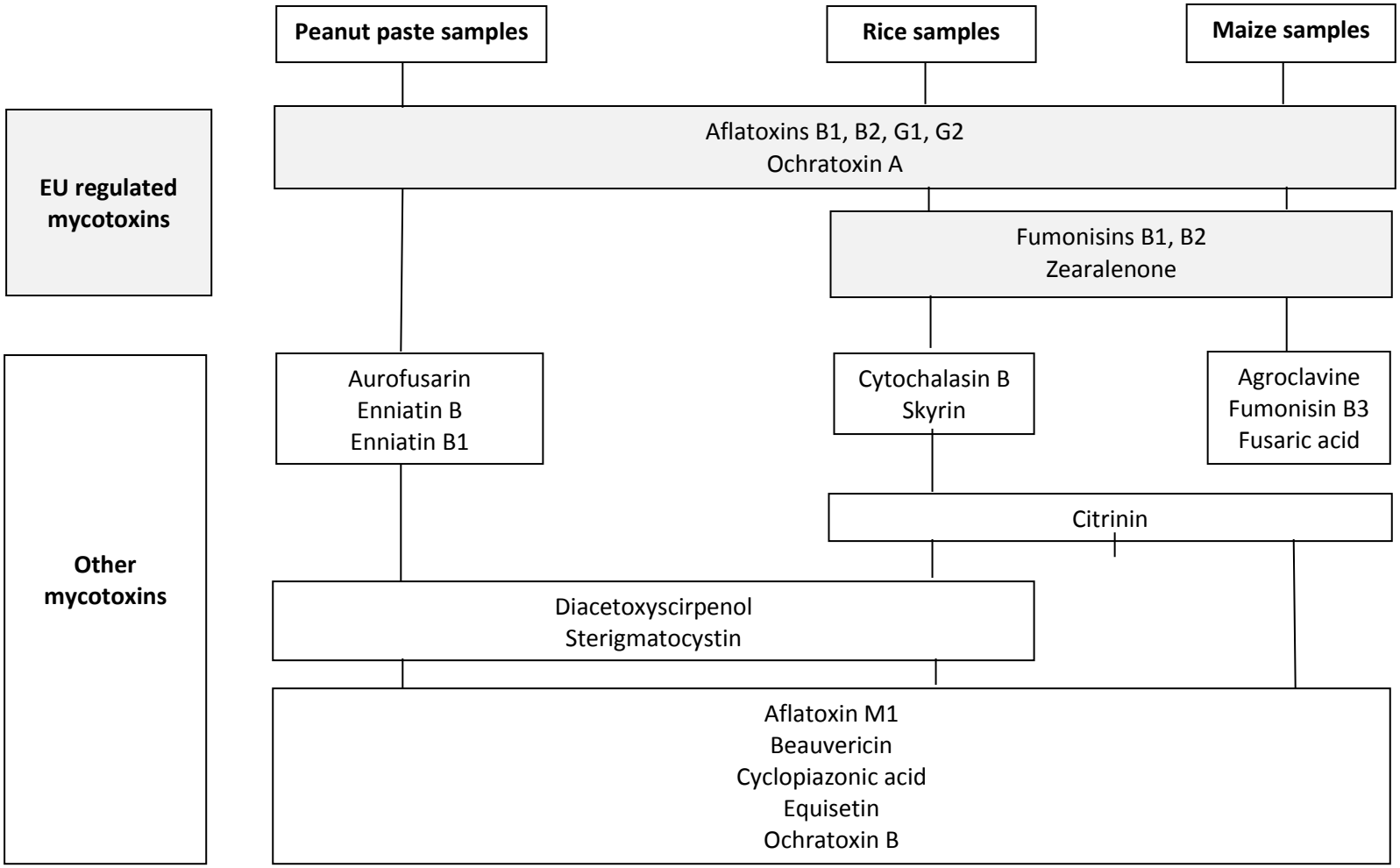

Figure 1: Multi-mycotoxin occurrence in food samples collected in Côte d'Ivoire