

Dispersive Magnetic Solid-Phase Extraction as a Novelty Sample Treatment for the Determination of the Main Aflatoxins in Paprika

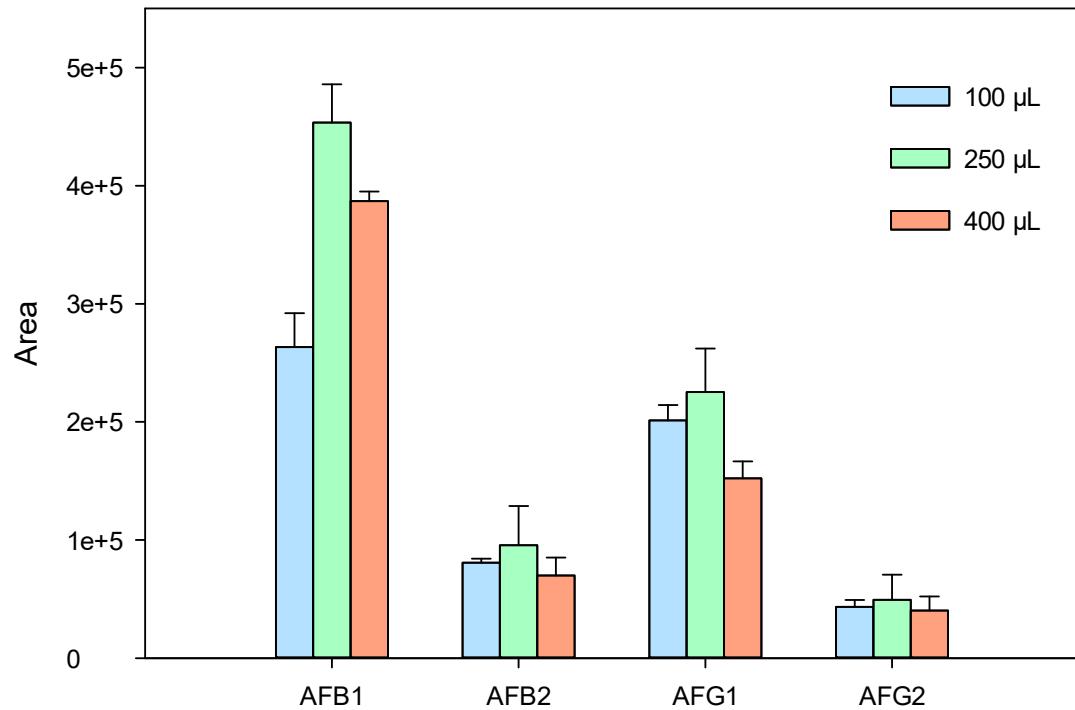


Figure S1. Influence of the $\text{Fe}_3\text{O}_4@\text{PPy}$ suspension volume on the sensitivity of the analytes.

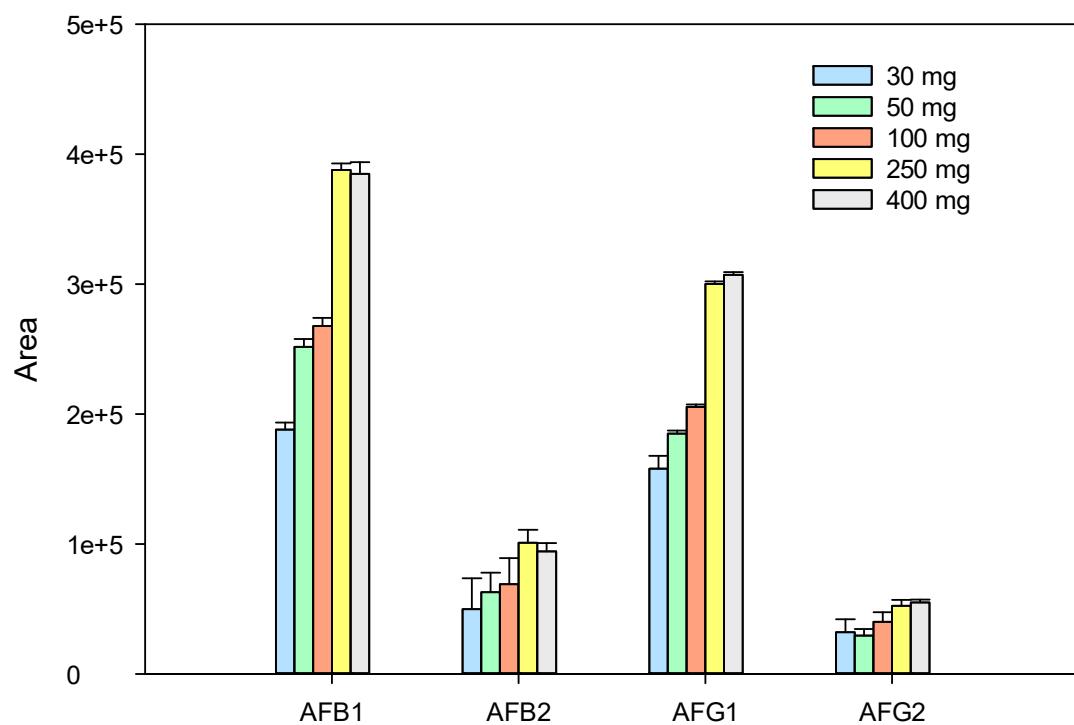


Figure S2. Influence of the $\text{Fe}_3\text{O}_4@\text{PPy}$ mass on the sensitivity of the analytes.

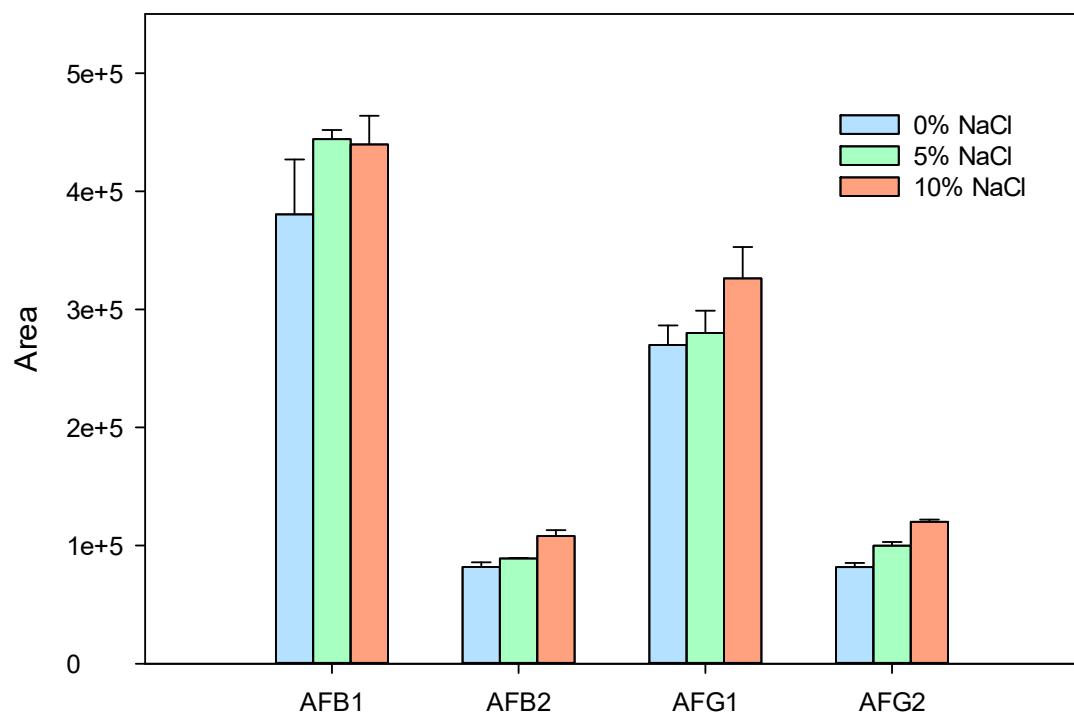


Figure S3. Influence of the NaCl content in the extraction medium on AFs preconcentration efficiency.

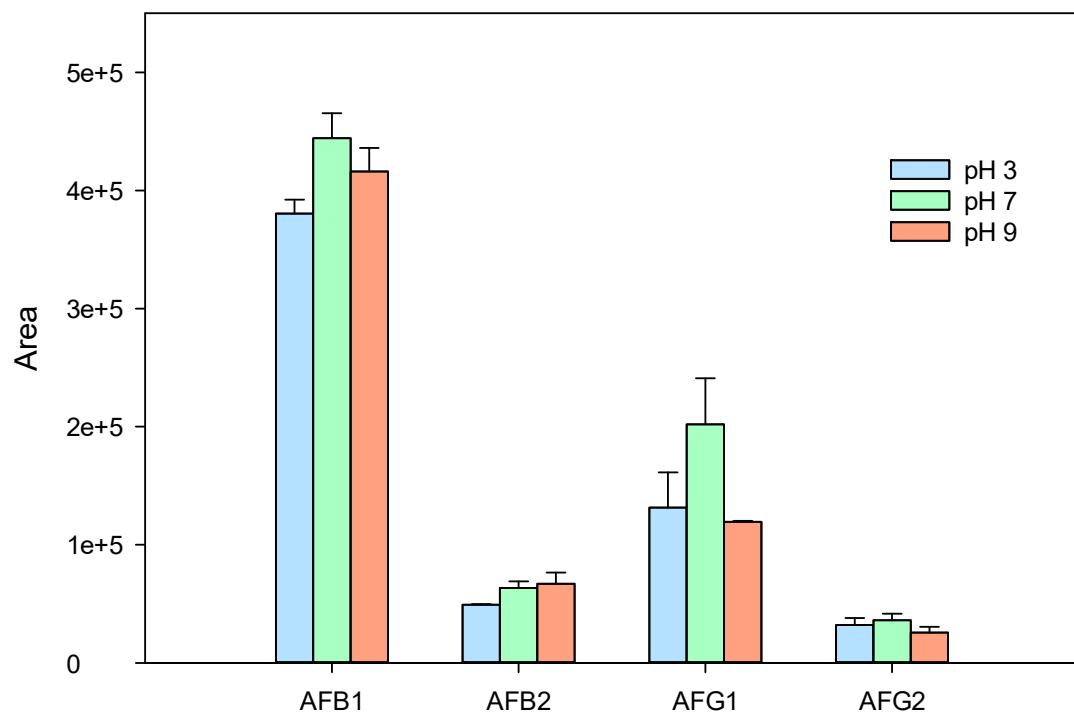


Figure S4. Influence of the pH on AFs preconcentration efficiency.

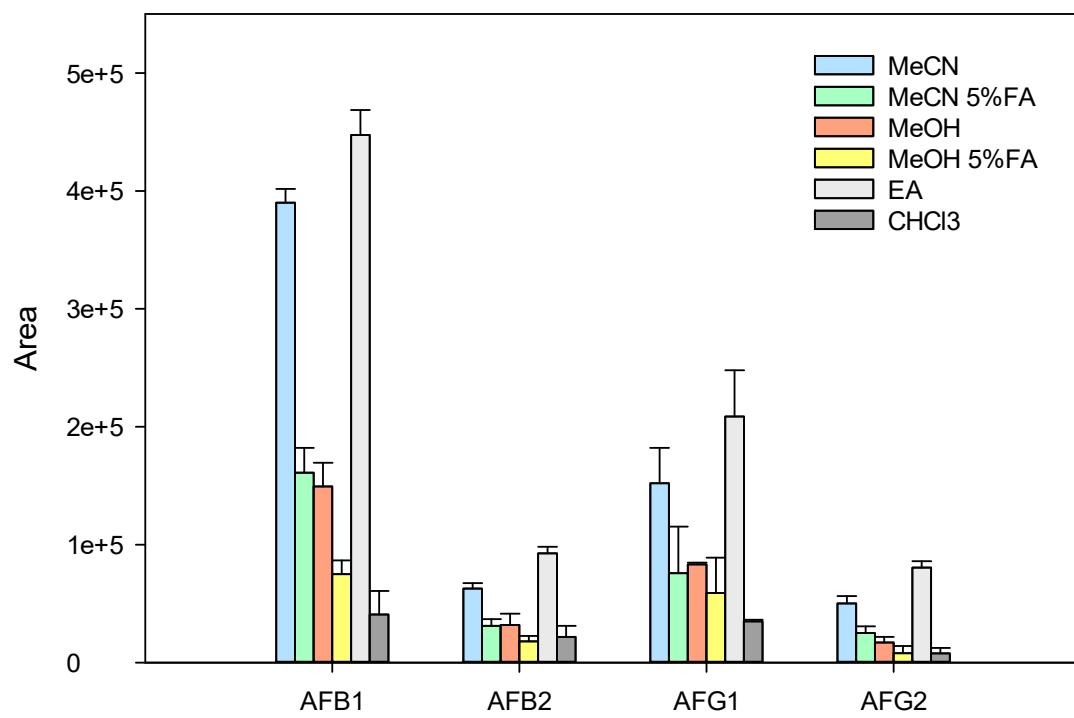


Figure S5. Influence of the desorption solvent nature on AFs preconcentration efficiency.

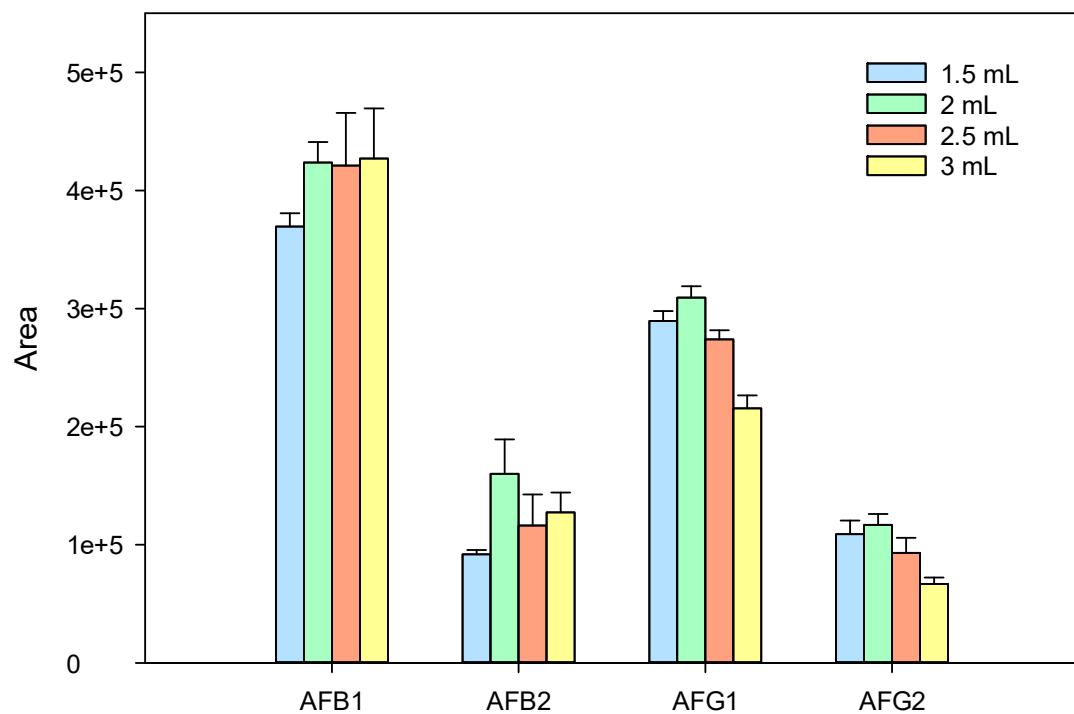


Figure S6. Influence of the desorption solvent volume on AFs preconcentration efficiency.

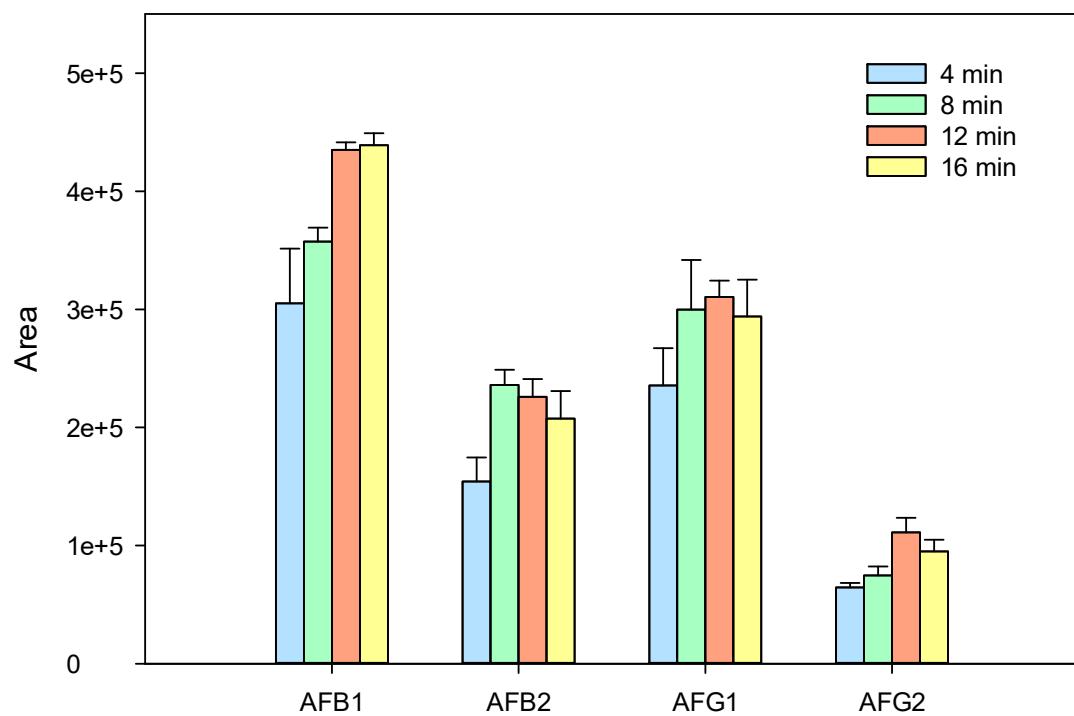


Figure S7. Influence of the desorption time on AFs preconcentration efficiency.

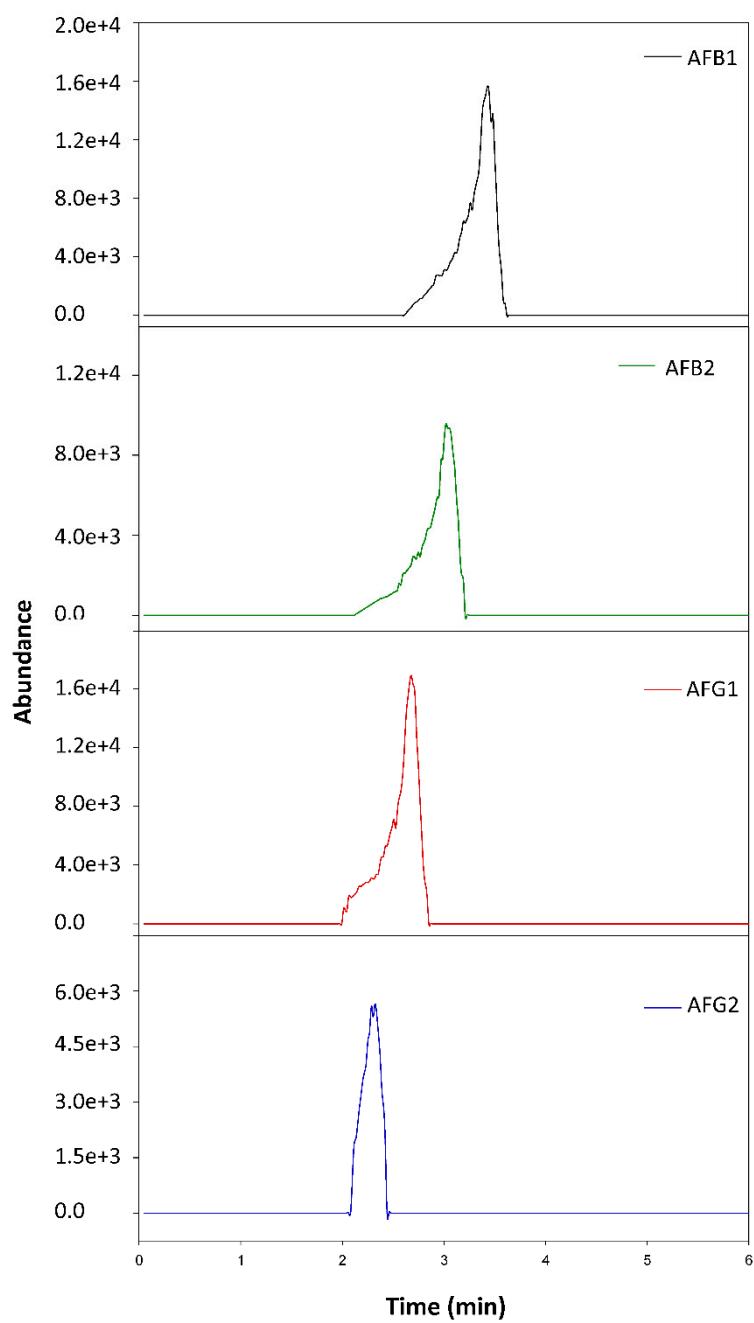


Figure S8. LC-HRMS chromatogram of AFB1, AFB2, AFG1 and AFG2 in spiked paprika at 10 µg kg¹.

Table S1. Comparison of the novel proposed method with other methods for the determination of AFs in food samples.

AFs	Matrix	NPs type	Determination technique	Sorbent amount	Method time	Organic solvent consumed	Linearity	Recovery (%)	RSD (%)	LOD	Ref
B1 B2 G1 G2	Fruit	HB-Zn/Co-ZIF-8	UHPLC-IT-MS	40 mg	60 min	150 µL of 0.1%FA-ACN	5-100 ng mL ⁻¹ (B1, G1, G2) 1-50 ng mL ⁻¹ (B2)	75.1- 102.4	5.3- 13.6	0.18-1.50 ng mL ⁻¹	(Wang et al., 2020)
B1 B2 G1 G2	Pistachio	Sp-M-Dp	HPLC-FLD	150 mg	20 min	1 mL ACN	2-10 ng g ⁻¹	72.0- 95.6	5.6- 7.3	0.02-0.07 ng g ⁻¹	Ahmadpoor, Nasrollahzadeh, & Maham, (Karami- Osboo, 2022)
B1 B2	Maize	MAMs	HPLC-PCD-FLD	NI	7 min	5 mL of BB and 1 mL of eluting buffer/MeOH (20:80, v/v)	0.5-50 µg kg ⁻¹	83.6- 97.8	1.6- 8.00	10-25 pg mL ⁻¹	(Liu et al., 2018)
B1 B2 G1 G2	Wheat	EGBMA-TEOS- MNP	IAC-HPLC-FLD	100 mg	12 min	2 mL of a mixture of Me ₂ CO/MeCN/CH ₂ Cl ₂ (1:1:2, v/v) and 2 mL of 0.5 mM Triton X-100 in 15% (v/v) ACN/water.	0.1-50 ng mL ⁻¹	92.0- 105.0	0.8- 2.6	0.03 ng mL ⁻¹	(Hanif, Allahyari, Pourghazi, & Amoli-diva, (Li, Zhang, & Shi, 2018)
B1	Wheat	Fe ₃ O ₄ - MWCNTs-NH ₂	HPLC-DAD	6 mg	40 min	0.2 mL of Me ₂ CO	1-100 ng g ⁻¹	88.8- 96.0	2.1- 2.8	0.15 ng g ⁻¹	(Hashemi & Taherimaslak, 2014)
B1 B2 G1 G2	Pistachio	AMT-TMSPT- MNP	HPLC -FLD	150 mg	8 min	2 mL Me ₂ CO/MeCN/CH ₂ Cl ₂ (1:1:2, v/v/v)	0.10-15 ng mL ⁻¹ (B1, G1) 0.04-3 ng mL ⁻¹ (B2, G2)	92.5- 103.2	2.3- 5.9	0.014- 0.037 µg kg ⁻¹	(Alilou, Amirzehni, & Eslami, 2021)
M2 M1 G2 G1 B2 B1	Rice and wheat flour	MIP-capped MGO/MOF-808	HPLC-FID	5 mg	25 min	0.5 mL ACN, 300 µL phosphate buffer (0.2 M, pH 7) and 250 µL GQDs.	0.03-5 ng mL ⁻¹	97.3- 98.6	0.9- 2.9	0.009- 3.69 ng mL ⁻¹	

B1	B2	G1	Pistachio	MIL-101(Cr) MIL-101(Cr)/Fe ₃ O ₄ @SiO ₂ @PTU	HPLC-FLD	60 mg	6 min	215 μL CH ₂ Cl ₂ /ACN/Me ₂ CO (2:1:1, v/v)	0.2-4 ng g ⁻¹ (B1, G1) 0.1-3.5 ng g ⁻¹ B2 0.3-0.4 ng g ⁻¹ G2	93.4- 97.8	6.4- 12.8	0.02- 0.09 ng g ⁻¹	(Bagher, Hamid, & Ghasempour, 2020)
B1	B2	G1	Paprika	Fe ₃ O ₄ @PPy	UHPLC-HRMS	244 mg	42 min	2 mL of EA	3.7 – 50 μg kg ⁻¹ (B1) 3.9 – 50 μg kg ⁻¹ (B2) 3.5 – 50 μg kg ⁻¹ (G1) 4.7 – 50 μg kg ⁻¹ (G2)	81.9- 99.4	5.3- 7.8	1.0 - 1.4 μg kg ⁻¹	This work

Table S2. Validation data for the determination of aflatoxins in paprika using HPLC-MS/MS.

Mycotoxin	Equation	Linearity ($\mu\text{g kg}^{-1}$)	Linearity R^2	LOD ($\mu\text{g kg}^{-1}$)	LOQ ($\mu\text{g kg}^{-1}$)	Matrix effect (%)
AFB1	$y = 2624 x + 1521$	2.3 - 50	0.992	0.7	2.3	61.9
AFB2	$y = 639.5 x + 173$	3.0 - 50	0.997	0.9	3.0	61.1
AFG1	$y = 2232 x + 302$	2.2 - 50	0.988	0.7	2.2	63.2
AFG2	$y = 373.6 x + 108$	2.5 - 50	0.998	0.8	2.5	60.1

Table S3. AFs and their derivatives investigated using untargeted processing.

Aflatoxin	Molecular Formula	<i>m/z</i>
Aflatoxin B2a	C ₁₇ H ₁₄ O ₇	330.0739
Aflatoxin G2a	C ₁₇ H ₁₄ O ₈	346.0689
Aflatoxin GM1	C ₁₇ H ₁₂ O ₈	344.0532
Aflatoxin M1	C ₁₇ H ₁₂ O ₇	328.0583
Aflatoxin M2	C ₁₇ H ₁₄ O ₇	330.0739
Aflatoxin M2a	C ₁₇ H ₁₄ O ₈	346.0689
Aflatoxin M4	C ₁₇ H ₁₂ O ₇	328.0583
Aflatoxin P1	C ₁₆ H ₁₀ O ₆	298.0477
Aflatoxin P2	C ₁₆ H ₁₂ O ₆	300.0634
Aflatoxin Q1	C ₁₇ H ₁₂ O ₇	328.0583
Aflatoxin Q2a	C ₁₇ H ₁₄ O ₈	346.0689
Aflatoxicol	C ₁₇ H ₁₄ O ₆	314.0790
Aflatoxicol B	C ₁₇ H ₁₄ O ₆	314.0790
Aflatoxicol H1	C ₁₇ H ₁₄ O ₇	330.0739
Aflatoxicol M1	C ₁₇ H ₁₄ O ₇	330.0739
<i>Biosynthesis pathway metabolites</i>		
Sterigmatocystin	C ₁₈ H ₁₂ O ₆	325.0707
O-methyl-sterigmatocystin	C ₁₉ H ₁₄ O ₆	339.0863
Dihydrosterigmatocystin	C ₁₈ H ₁₄ O ₆	327.0863
Dihydro-O-methylsterigmatocystin	C ₁₉ H ₁₆ O ₆	341.1020
<i>Degradation products</i>		
DP1	C ₁₆ H ₁₂ O ₄	269.0808
DP2	C ₁₇ H ₁₀ O ₆	311.0550
DP3	C ₁₇ H ₁₀ O ₇	327.0499
DP4	C ₁₇ H ₁₆ O ₆	317.1020
DP5	C ₁₆ H ₁₂ O ₆	301.0707
DP6	C ₁₆ H ₆ O ₅	279.0288
DP7	C ₁₆ H ₁₀ O ₅	283.0601
DP8	C ₁₆ H ₂₂ O ₅	295.1540
DP9	C ₁₆ H ₂₀ O ₅	293.1384
DP10	C ₁₆ H ₂₀ O ₆	309.1333

DP11	$C_{16}H_{18}O_6$	307.1176
DP12	$C_{16}H_{16}O_7$	321.0929

Table S4. UHPLC-HRMS parameters of the target aflatoxins.

Compound	RT ^a (min)	Formula	Precursor ion (<i>m/z</i>)	Error (ppm)	Product ion 1 (<i>m/z</i>)	Product ion 2 (<i>m/z</i>)
AFG2	2.319	$C_{17}H_{14}O_7$	331.0812	1.3	245.1	313.1
AFG1	2.679	$C_{17}H_{12}O_7$	329.0656	-0.37	243.1	311.1
AFB2	3.021	$C_{17}H_{14}O_6$	315.0863	-0.59	286.9	259
AFB1	3.432	$C_{17}H_{12}O_6$	313.0707	-0.75	241	284.9

^aRT: retention time