

Supplementary Materials: Fe₃O₄@COF(TAPT-DHTA) nanocomposites as magnetic solid-phase extraction adsorbents for simultaneous determination of 9 mycotoxins in fruits by UHPLC-MS/MS

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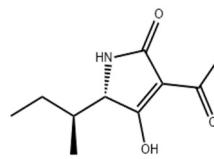
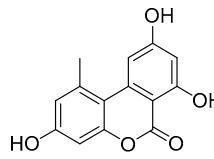
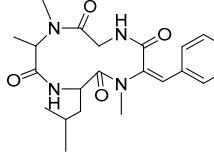
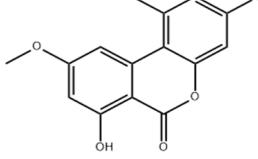
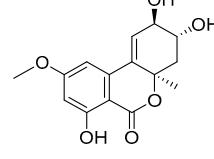
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Table S1. Structure and physicochemical parameters of 9 mycotoxins

Mycotoxins	Structure	Molecular weight	pKa	Molecular Formula	Hydrogen Bond Donor Count	Hydrogen Bond Acceptor Count
Tenuazonic acid (TeA)		197.23	4.50±1.00	C ₁₀ H ₁₅ NO ₃	2	3
Alternariol (AOH)		258.23	7.16±0.20	C ₁₄ H ₁₀ O ₅	3	5
Tentoxin (TEN)		414.50	—	C ₂₂ H ₃₀ N ₄ O ₄	2	4
Alternariol monomethyl ether (AME)		272.25	7.00±0.20	C ₁₅ H ₁₂ O ₅	2	5
Altenuene (ALT)		292.28	7.41±0.70	C ₁₅ H ₁₆ O ₆	3	6

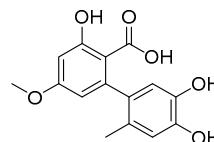
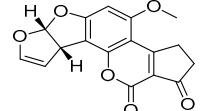
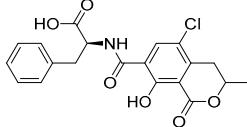
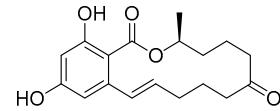
Mycotoxins	Structure	Molecular weight	pKa	Molecular Formula	Hydrogen Bond Donor Count	Hydrogen Bond Acceptor Count
Altenusin (ALS)		290.27	3.08±0.34	C ₁₅ H ₁₄ O ₆	4	6
Aflatoxin B ₁ (AFB ₁)		312.27	—	C ₁₇ H ₁₂ O ₆	0	6
Ochratoxin A (OTA)		403.81	3.40±0.10	C ₂₀ H ₁₈ ClNO ₆	3	6
Zearalenone (ZEN)		318.40	7.58±0.40	C ₁₈ H ₂₂ O ₅	2	5

Table S2. Mass spectrometry parameters of 9 mycotoxins

Mycotoxins	Precursor Ions (m/z)	Retention time (min)	Product Ions (m/z)	Dwell Time (s)	Cone Voltage (V)	Collision Energy (eV)
TeA	198.1 [M+H] ⁺	1.75	153.1058 ^a	0.005	42	16
			125.0214 ^b	0.005	42	12
AOH	259.0 [M+H] ⁺	4.45	185.1184 ^a	0.005	64	28
			213.1284 ^b	0.005	64	24
AME	273.0 [M+H] ⁺	5.65	258.0000 ^a	0.012	54	26
			128.0600 ^b	0.012	54	25
ALT	292.9 [M+H] ⁺	3.64	257.0693 ^a	0.005	30	14
			275.0738 ^b	0.005	30	8
TEN	415.5 [M+H] ⁺	4.76	132.1335 ^a	0.005	14	34
			256.2291 ^b	0.005	14	34
ALS	288.8 [M-H] ⁻	2.62	245.0728 ^a	0.005	8	20
			230.0566 ^b	0.005	45	17
AFB ₁	313.2 [M+H] ⁺	3.78	241.1575 ^a	0.009	74	36
			284.9710 ^b	0.009	74	22
OTA	404.3 [M+H] ⁺	4.28	239.0344 ^a	0.005	30	34
			221.0280 ^b	0.005	30	22

ZEN	317.2 [M-H] ⁻	5.33	175.0464 ^a	0.012	56	26
			131.0360 ^b	0.012	56	24

^a quantitation ion; ^b qualifier ions

Table S3. Significance analysis of adsorbed solution

Mycotoxins	Adsorption Solution (Recovery±SD %)					
	0%	1%	2%	3%	4%	5%
TeA	67.88±4.55 ^b	82.77±5.31 ^a	61.51±6.51 ^{bc}	56.64±3.63 ^{cd}	58.52±6.02 ^c	48.81±2.21 ^d
AOH	74.06±3.15 ^b	104.18±3.60 ^a	69.66±1.62 ^{bc}	74.10±5.59 ^b	67.32±6.41 ^{bc}	61.48±4.60 ^c
AME	81.23±10.28 ^b	99.62±5.91 ^a	80.73±11.58 ^b	79.31±4.54 ^b	54.19±6.29 ^c	40.48±5.12 ^c
TEN	69.31±5.76 ^b	87.71±12.93 ^a	42.84±2.48 ^c	34.68±3.93 ^{cd}	28.26±4.66 ^{de}	20.79±2.74 ^e
ALT	65.74±3.22 ^b	87.50±8.3 ^a	34.76±1.79 ^c	28.58±4.6 ^{cd}	25.53±1.18 ^d	20.71±1.57 ^d
ALS	63.29±4.93 ^b	85.81±12.76 ^a	53.21±6.89 ^b	33.83±3.17 ^c	20.70±2.87 ^d	15.47±2.86 ^d
AFB ₁	74.50±7.50 ^{ab}	79.15±9.81 ^{ab}	81.32±8.69 ^a	72.99±9.95 ^{ab}	64.18±9.76 ^{bc}	55.54±5.03 ^c
OTA	87.73±3.66 ^a	90.71±2.66 ^a	79.62±4.22 ^{ab}	79.86±14.06 ^{ab}	60.09±11.08 ^{bc}	46.78±8.03 ^c
ZEN	77.34±2.40 ^{bc}	95.94±6.18 ^a	91.45±5.87 ^{ab}	85.69±10.75 ^{ab}	65.70±12.43 ^{cd}	54.05±4.43 ^d

Table S4. Significance analysis of pH value of adsorbed solution

Mycotoxins	pH of Adsorption Solution (Recovery±SD %)					
	pH=3	pH=4	pH=5	pH=6	pH=7	pH=8
TeA	62.15±10.15 ^b	83.01±4.11 ^a	61.26±7.34 ^b	54.28±2.44 ^{bc}	48.93±3.32 ^c	50.50±2.89 ^c
AOH	83.49±7.06 ^{ab}	95.99±7.95 ^a	94.92±1.23 ^a	83.86±4.26 ^{ab}	79.01±8.35 ^{ab}	71.14±1.11 ^b
AME	94.96±7.36 ^a	104.51±5.25 ^a	83.36±4.24 ^{ab}	66.26±0.87 ^c	51.31±11.75 ^c	47.87±11.26 ^c
TEN	74.34±5.19 ^b	89.95±6.48 ^a	92.88±4.96 ^a	81.03±2.08 ^{ab}	54.14±1.31 ^c	50.55±4.46 ^c
ALT	57.45±3.82 ^b	87.64±9.59 ^a	68.03±1.13 ^b	68.52±8.17 ^b	67.94±3.59 ^b	62.89±11.75 ^b
ALS	64.06±9.09 ^b	82.81±4.02 ^a	83.79±1.88 ^a	63.01±5.34 ^b	54.50±0.88 ^c	51.20±1.70 ^c
AFB ₁	57.98±1.04 ^b	76.42±3.92 ^a	55.95±0.70 ^b	39.09±0.62 ^c	39.30±3.25 ^c	36.93±4.58 ^c
OTA	50.43±3.84 ^b	88.29±0.93 ^a	53.94±0.62 ^b	39.42±1.07 ^c	41.15±5.64 ^c	28.23±1.07 ^d
ZEN	63.94±3.70 ^b	92.12±6.32 ^a	62.07±5.13 ^b	40.19±2.91 ^c	34.67±3.86 ^c	33.96±0.86 ^c

Table S5. Significance analysis of NaCl concentration in adsorbed solution

Mycotoxins	Concentration of NaCl (Recovery±SD %)					
	0 mg mL ⁻¹	2 mg mL ⁻¹	4 mg mL ⁻¹	6 mg mL ⁻¹	8 mg mL ⁻¹	10 mg mL ⁻¹
TeA	85.10±10.52 ^a	76.62±1.64 ^b	67.87±3.04 ^c	62.54±1.39 ^c	59.34±1.33 ^c	49.91±2.48 ^d
AOH	103.11±5.38 ^a	95.00±7.56 ^a	81.63±3.99 ^{ab}	71.01±2.99 ^b	59.63±3.05 ^{bc}	43.69±4.25 ^c
AME	100.28±5.87 ^a	95.88±2.09 ^a	61.61±4.72 ^b	54.77±10.44 ^{bc}	47.84±1.35 ^c	43.84±7.11 ^c
TEN	94.67±6.53 ^a	73.01±5.57 ^b	69.21±1.79 ^b	58.18±4.38 ^c	55.36±3.71 ^{cd}	47.92±3.30 ^d
ALT	83.22±2.68 ^a	69.68±5.56 ^b	63.43±2.09 ^c	68.46±1.48 ^{bc}	72.07±4.47 ^b	54.96±0.57 ^d
ALS	84.88±8.35 ^a	75.66±4.23 ^a	60.68±5.88 ^b	55.42±4.90 ^b	54.07±2.97 ^b	59.46±4.12 ^b
AFB ₁	76.83±3.76 ^a	65.97±7.32 ^b	64.89±3.53 ^b	58.69±6.10 ^{bc}	49.10±1.57 ^{cd}	43.52±5.01 ^d
OTA	87.43±2.43 ^a	61.69±0.50 ^b	59.38±3.17 ^b	48.51±6.29 ^c	35.55±5.50 ^d	36.71±2.77 ^d
ZEN	94.10±6.01 ^a	62.32±7.32 ^b	49.90±5.80 ^c	41.82±0.72 ^{cd}	35.48±2.34 ^{de}	29.85±1.15 ^e

Table S6. Significance analysis of Adsorption time

Mycotoxins	Adsorption time (Recovery±SD %)				
	2 min	4 min	6 min	8 min	10 min
TeA	22.73±5.42 ^d	49.39±5.19 ^c	72.92±8.54 ^b	88.30±11.50 ^a	88.79±4.91 ^a
AOH	41.34±5.65 ^d	59.93±4.12 ^c	82.08±7.92 ^b	96.43±4.57 ^a	95.80±7.52 ^a
AME	32.58±2.22 ^d	46.02±2.27 ^c	74.66±1.91 ^b	96.58±1.55 ^a	99.66±4.02 ^a
TEN	31.00±1.00 ^d	62.92±1.10 ^c	74.50±8.64 ^b	95.97±0.72 ^a	92.66±8.90 ^a
ALT	53.48±4.49 ^b	76.77±9.62 ^a	88.26±8.01 ^a	85.46±7.79 ^a	93.18±1.74 ^a
ALS	49.84±3.18 ^b	61.23±8.91 ^b	79.39±4.90 ^a	89.42±1.79 ^a	93.76±9.50 ^a
AFB ₁	19.75±11.71 ^b	31.63±6.87 ^b	64.74±0.31 ^a	78.16±2.73 ^a	77.41±4.22 ^a
OTA	49.23±8.70 ^c	65.93±5.84 ^b	72.23±7.82 ^b	91.29±6.68 ^a	86.36±8.04 ^a
ZEN	29.34±1.29 ^c	36.57±1.91 ^c	58.31±5.81 ^b	94.87±7.99 ^a	101.09±5.72 ^a

Table S7. Significance analysis of adsorbent amount

Mycotoxins	The amount of adsorbent (Recovery±SD %)				
	10 mg	15 mg	20 mg	25 mg	30 mg
TeA	71.26±4.03 ^c	85.57±4.75 ^{ab}	90.58±8.38 ^a	88.27±1.05 ^{ab}	75.02±2.30 ^{bc}
AOH	59.90±2.87 ^d	69.81±3.21 ^c	96.95±2.56 ^a	72.97±1.18 ^{bc}	74.39±1.13 ^b
AME	63.38±2.82 ^c	75.14±0.26 ^b	96.49±5.08 ^a	83.07±7.95 ^b	76.78±8.78 ^b
TEN	68.41±3.44 ^b	70.17±1.49 ^b	87.73±6.07 ^a	69.85±0.94 ^b	67.92±0.61 ^b
ALT	68.79±3.66 ^c	75.97±9.37 ^{bc}	90.63±3.29 ^a	83.79±2.16 ^{ab}	81.46±2.58 ^{ab}
ALS	64.40±1.99 ^b	72.28±5.09 ^b	82.32±8.99 ^a	76.08±4.34 ^b	79.34±5.31 ^b
AFB ₁	35.83±0.99 ^c	54.81±0.68 ^b	75.26±0.81 ^a	73.63±1.28 ^a	77.81±6.10 ^a
OTA	45.47±4.09 ^b	48.52±1.38 ^b	83.80±7.59 ^a	72.22±5.67 ^a	76.53±7.57 ^a
ZEN	58.40±6.36 ^c	75.73±0.92 ^b	97.18±11.02 ^a	86.47±3.09 ^{ab}	84.37±10.31 ^{ab}

Table S8. Significance analysis of elution kinds

Mycotoxins	The kinds of Elution solvent (Recovery±SD %)							
	A	B	C	D	E	F	G	H
TeA	81.93±3.98 ^b	45.03±7.73 ^d	79.90±0.52 ^b	55.31±5.38 ^{cd}	68.78±1.74 ^{bc}	72.75±6.43 ^b	106.28±3.42 ^a	66.81±8.84 ^{bc}
AOH	99.82±9.16 ^{ab}	103.53±6.22 ^{ab}	94.33±1.87 ^{abc}	93.29±6.94 ^{bc}	81.73±8.30 ^{cd}	82.12±1.25 ^{cd}	107.33±5.08 ^a	76.05±2.60 ^d
AME	98.92±6.83 ^a	72.39±2.97 ^{cde}	70.19±2.47 ^{de}	75.16±1.97 ^{cd}	85.18±0.73 ^b	78.83±6.26 ^{bc}	93.64±6.23 ^a	65.49±2.96 ^e
TEN	91.04±7.38 ^a	66.18±4.34 ^d	74.69±3.87 ^{cd}	89.22±2.46 ^{ab}	80.64±0.77 ^{bc}	78.20±5.03 ^c	83.33±7.55 ^{abc}	77.78±4.42 ^c
ALT	89.25±2.61 ^a	52.42±8.83 ^{bcd}	65.96±5.28 ^{bc}	60.23±5.22 ^{bc}	64.08±4.38 ^b	47.28±7.59 ^{cd}	90.35±5.02 ^a	44.5±6.07 ^d
ALS	86.21±3.99 ^a	67.46±3.54 ^{bc}	76.44±0.72 ^{ab}	76.22±4.82 ^{ab}	72.05±1.002 ^{abc}	38.36±6.47 ^d	86.62±2.75 ^a	58.69±5.44 ^c
AFB ₁	80.86±1.53 ^a	70.52±8.59 ^b	64.74±0.31 ^{bc}	61.49±2.73 ^c	40.19±2.51 ^{de}	35.23±0.64 ^e	72.19±8.54 ^b	45.04±0.89 ^d
OTA	87.69±5.48 ^a	71.48±6.72 ^{bc}	61.12±1.89 ^c	80.18±2.87 ^{ab}	62.47±2.15 ^{bc}	43.32±1.42 ^d	87.36±1.42 ^a	44.39±1.85 ^d
ZEN	94.24±3.43 ^a	69.91±1.91 ^b	65.53±0.51 ^b	72.65±4.95 ^b	67.76±5.72 ^b	66.07±0.73 ^b	68.23±8.63 ^b	53.85±7.39 ^c

(A) Methanol, (B) Acetonitrile, (C) Methanol/Formic acid (99/1), (D) Acetonitrile/Formic acid (99/1), (E) Methanol/Acetonitrile/Formic acid (50/49/1), (F) Methanol/Acetonitrile/Ammmonium hydroxide (50/49/1), (G) Methanol/Acetonitrile/Formic acid (80/19/1), (H) Methanol/Acetonitrile/Ammonium hydroxide (80/19/1)

Table S9. Significance analysis of Elution Volume

	3 mL	9 mL	15 mL
TeA	88.15±7.30 ^a	80.53±3.95 ^a	80.65±7.49 ^a
AOH	99.69±2.73 ^a	84.97±2.63 ^a	93.91±1.66 ^a
AME	96.80±3.02 ^a	104.35±5.29 ^a	98.41±3.92 ^a
TEN	88.99±7.09 ^{ab}	82.04±3.49 ^b	94.90±2.21 ^a
ALT	83.41±3.99 ^a	71.26±3.24 ^a	67.62±7.81 ^a
ALS	81.39±8.74 ^a	86.55±4.75 ^a	83.93±2.73 ^a
AFB ₁	78.40±5.08 ^a	75.67±0.34 ^a	76.39±1.20 ^a
OTA	88.85±3.48 ^a	89.31±7.16 ^a	81.88±6.15 ^a
ZEN	98.6±5.71 ^{ab}	103.88±10.15 ^a	84.70±3.06 ^b

Table S10. Significance analysis of Elution time

Mycotoxins	Elution time (Recovery±SD %)				
	1 min	2 min	3 min	4 min	5 min
TeA	38.49±3.30 ^c	65.98±2.38 ^b	70.14±3.29 ^b	91.19±1.98 ^a	90.06±2.70 ^a
AOH	42.29±6.55 ^c	76.57±7.40 ^b	103.03±10.37 ^a	101.99±6.40 ^a	99.96±5.85 ^a
AME	48.12±2.97 ^c	64.14±5.67 ^b	99.39±7.20 ^a	103.62±2.06 ^a	100.29±5.14 ^a
TEN	37.85±0.93 ^c	63.64±0.27 ^b	85.58±0.87 ^a	86.26±0.60 ^a	83.34±1.44 ^a
ALT	41.53±4.79 ^c	54.18±5.08 ^b	82.20±2.27 ^a	88.02±7.98 ^a	83.44±6.11 ^a
ALS	40.69±2.85 ^b	65.91±6.75 ^a	72.07±4.50 ^a	80.22±9.80 ^a	81.76±5.85 ^a
AFB ₁	33.26±4.93 ^c	52.23±8.38 ^c	65.50±6.30 ^a	75.08±3.05 ^a	77.34±4.96 ^a
OTA	32.77±1.78 ^c	59.66±6.41 ^b	64.28±7.4 _b	90.96±1.50 ^a	87.81±3.07 ^a
ZEN	41.35±1.54 ^c	51.78±3.74 ^{bc}	59.41±2.97 ^b	96.71±8.00 ^a	97.80±3.68 ^a

Table S11. Comparison of the Fe₃O₄@COF(TAPT-DHTA) based MSPE method developed in this study with the sample pretreatment approaches reported in the previous studies.

Mycotoxins	Matrix	Sorbent	Sample pretreatment	Analytical technique	Adsorption/ Elution time of MSPE (min)	Recovery (%)	LOD ($\mu\text{g kg}^{-1}$)	Ref.
FB ₁ , ZON, OTA	maize oil, rapeseed oil, soybean oil	Fe ₃ O ₄ @nSiO ₂ @mSiO ₂	MSPE	UPLC-MS/MS	10/5	89.40-97.10	0.08-1.03	[1]
AFB ₁ , AFB ₂ , AFG ₁ , AFG ₂ , OTA, OTB	vegetable oils	PDA@Fe ₃ O ₄ -MWCNTs	MSPE	HPLC-FLD	10/3	70.15-89.25	0.20-0.50	[2]
AFB ₁ , ZON	Wheat flour	Fe ₃ O ₄ -MWCNTs-NH ₂	MSPE	HPLC	25/15	88.80-96.00	0.15-0.24	[3]
AFB ₁ , AFG ₁ , STER, ZEN, OTA	licorice	Fe ₃ O ₄ @PDA/MIL-101(Cr)	MSPE	UHPLC-MS/MS	30/2	78.53-116.28	0.01-0.09	[4]
AOH, AME, TEN, TeA	jujube	MgSO ₄ , NaCl	QuEChERS	UPLC-MS/MS		83.50-109.60	0.14-0.26	[5]
AOH, AME, TEN, TeA	tomato, fruit-based products	MgSO ₄ , NaCl	QuEChERS	LC-MS/MS		98.80-108.90	0.25-20.00	[6]
AOH, AME, TEN, TeA, ALT	wolfberry	C18	QuEChERS	UPLC-MS/MS		73.80-111.50	0.07-0.24	[7]
9 mycotoxins	Tomato, Strawberry, Watermelon, Melon, Hawthorn	Fe ₃ O ₄ @COF(TAPT-DHTA)	MSPE	UHPLC-MS/MS	8/4	74.25-111.75	0.01-0.50	This work

Table S12. Contamination levels of 9 mycotoxins in five fruits (tomato, strawberry, watermelon, melon, hawthorn)

Mycotoxins	Tomato		Strawberry		Watermelon		Melon		Hawthorn	
	Positive/Total samples	Content range ($\mu\text{g kg}^{-1}$)	Positive/Total samples	Content range ($\mu\text{g kg}^{-1}$)	Positive/Total samples	Content range ($\mu\text{g kg}^{-1}$)	Positive/Total samples	Content range ($\mu\text{g kg}^{-1}$)	Positive/Total samples	Content range ($\mu\text{g kg}^{-1}$)
TeA	10/20	3.8-6.5	9/20	1.9-5.6	0/20	ND ^a	1/20	<LOQ	0/20	ND
AOH	2/20	3.05-4.0	3/20	4.9-20.0	1/20	<LOQ	0/20	ND	5/20	3.7-14.2
AME	0/20	ND	0/20	ND	0/20	ND	1/20	1.4	6/20	10.9-16.8
TEN	1/20	18.2	0/20	ND	0/20	ND	0/20	ND	3/20	0.6-1.3
ALT	8/20	2.2-44.5	11/20	3.4-54.8	5/20	29.5-56.3	10/20	43.4-123.7	9/20	38.8-190.4
ALS	1/20	1.88	3/20	<LOQ	0/20	ND	4/20	1.3-5.9	2/20	4.3-8.1
AFB ₁	0/20	ND								
OTA	0/20	ND								
ZEN	0/20	ND								

^a: means not detected

Table S13. Comparison between the developed UHPLC-MS/MS method and the reference methods by using tomato sample No. 7,14 and 20 ($\mu\text{g kg}^{-1}$)

	Samples	TeA	AOH	AME	ALT	AFB ₁	OTA	TEN	ALS	ZEN
Current method	7		3.05 \pm 0.08 ^a		9.09 \pm 0.19 ^a					
	14				7.65 \pm 0.27 ^a			18.20 \pm 0.26 ^a		
	20				13.79 \pm 0.75 ^a				1.88 \pm 0.12 ^a	
Reference method*	7		2.89 \pm 0.18 ^a		9.28 \pm 0.97 ^a					
	14				8.39 \pm 0.53 ^a			17.09 \pm 1.14 ^a		
	20				13.62 \pm 3.06 ^a				2.01 \pm 0.53 ^a	

*Reference method: SN/T 4259-2015

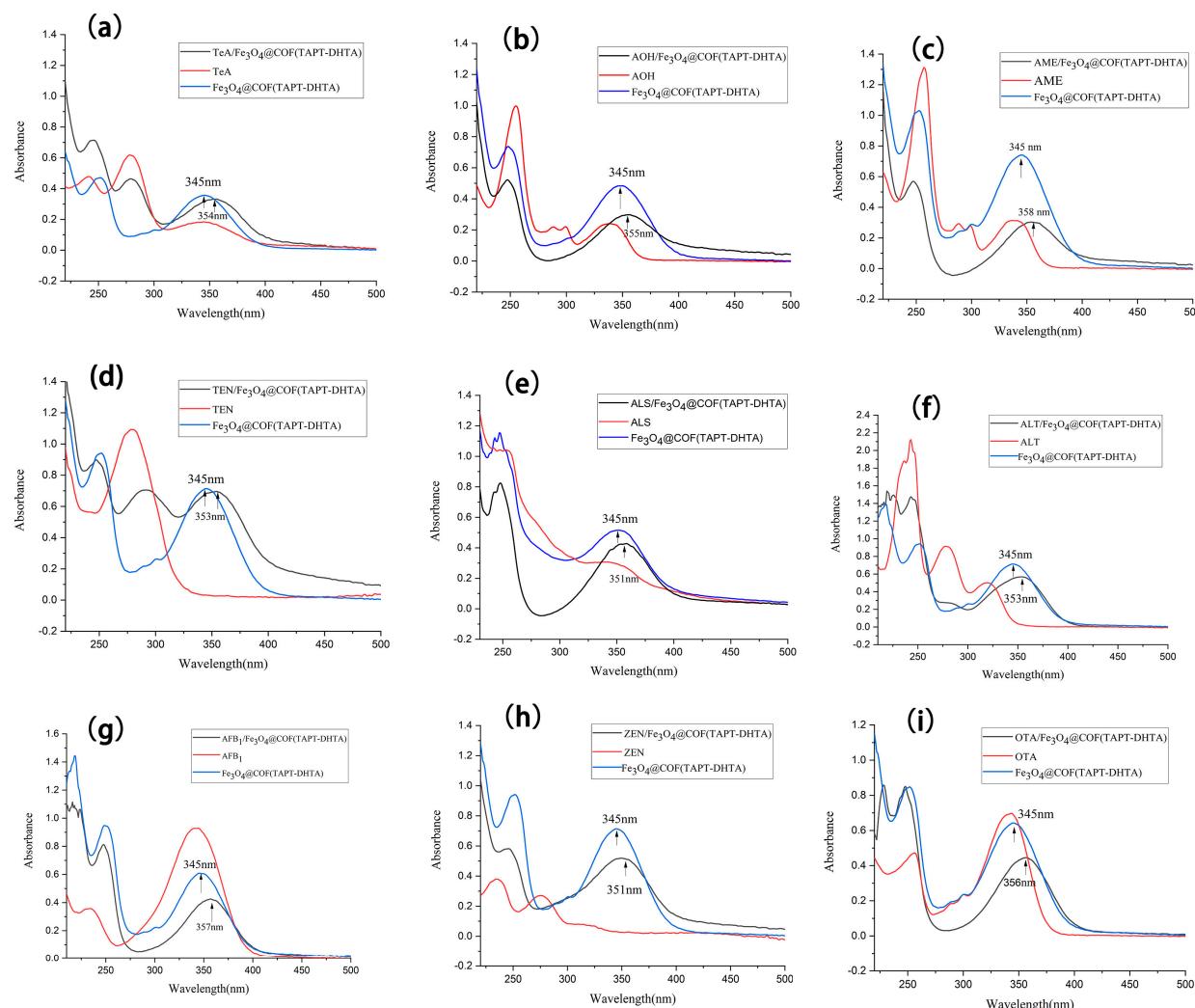


Figure S1. UV-Vis spectra of targeted mycotoxins, Fe₃O₄@COF(TAPT-DHTA) and mycotoxin/Fe₃O₄@COF(TAPT-DHTA)

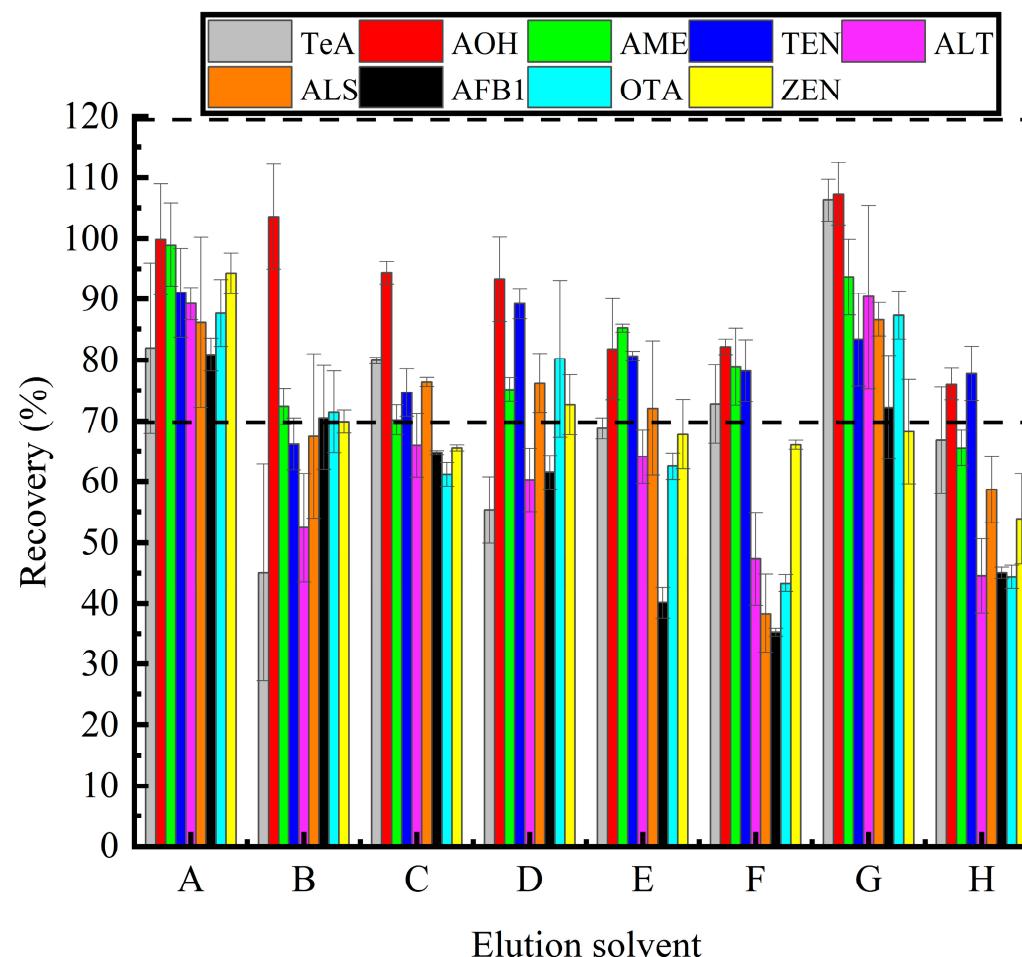


Figure S2. Comparison of the purification efficiency of 9 mycotoxins in the spiked tomato sample by 8 candidate elution solutions, (A) Methanol, (B) Acetonitrile, (C) Methanol/Formic acid (99/1), (D) Acetonitrile/Formic acid (99/1), (E) Methanol/Acetonitrile/Formic acid (50/49/1), (F) Methanol/Acetonitrile/Ammonium hydroxide (50/49/1), (G) Methanol/Acetonitrile/Formic acid (80/19/1), (H) Methanol/Acetonitrile/Ammonium hydroxide (80/19/1)

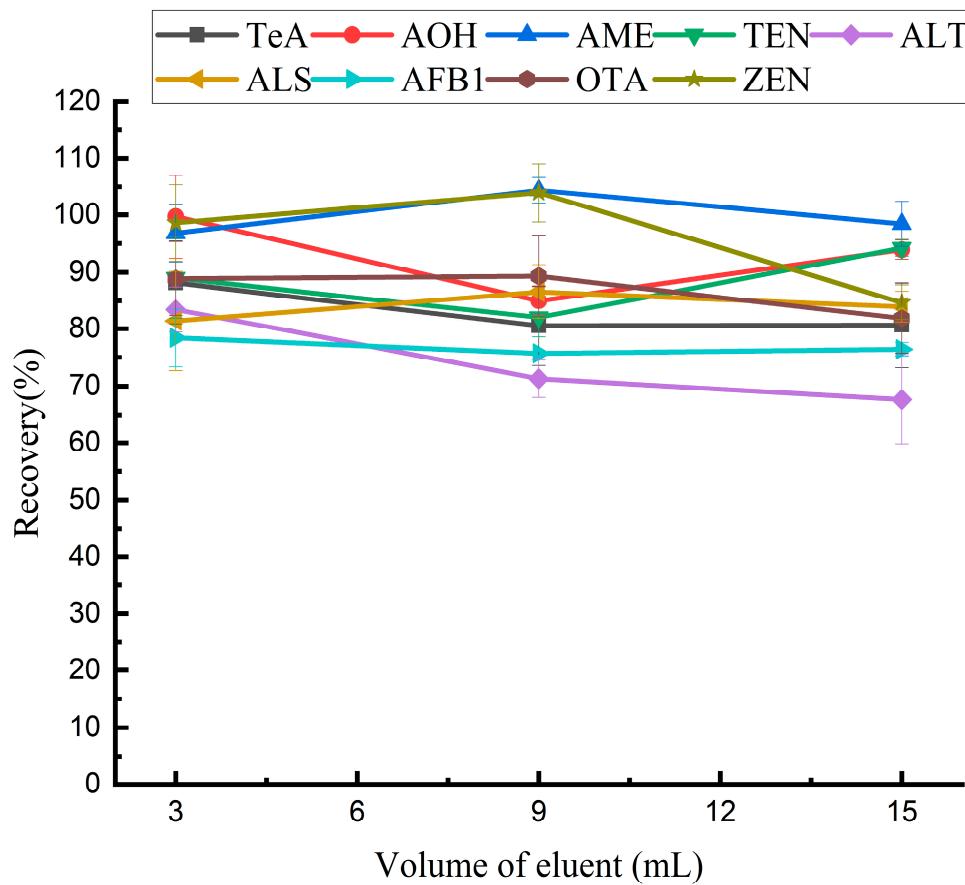


Figure S3. Effects of eluent volume on $\text{Fe}_3\text{O}_4@\text{COF}$ (TAPT-DHTA) MSPE process

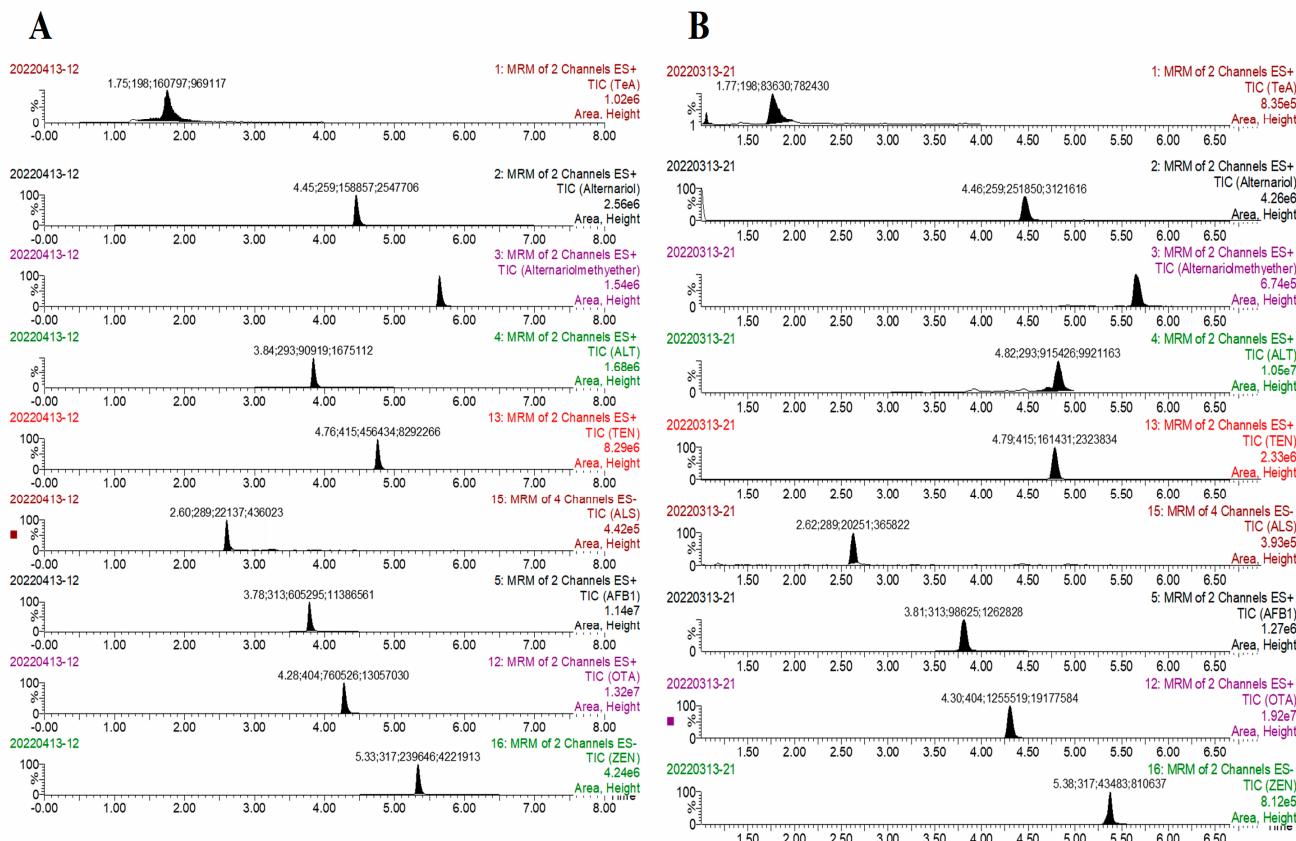


Figure S4. MRM chromatography of 9 mycotoxins in (A)solvent standard solution and (B) matrix standard solution (100 µg kg⁻¹). The retention time of peak corresponding to each toxin were as follow: TeA (1.75), AOH (4.45), AME (5.65), ALT (3.64), TEN (4.76), ALS (2.62), AFB₁ (3.78), OTA (4.28), ZEN (5.33)

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