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Fully automated online sample extraction and analysis of mycotoxins in foods by online SFE-SFC-MS

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Introduction

Mycotoxins are low-molecular-weight natural products produced by fungi and are capable of causing disease and death. They are strictly regulated around the world because of their strong carcinogenic effects. A simple and reliable method to analyze mycotoxins is required to ensure food safety. The current methods require time-consuming sample pretreatment. Here we report a fully automated online sample extraction and analysis of mycotoxins in foods by online SFE-SFC-MS.

Materials and Method

Reagents and standards

Reagents: T-2 toxin, HT-2 toxin, deoxynivalenol, zearalenone, aflatoxin G2, aflatoxin G1, aflatoxin B2, and aflatoxin B1 were purchased from Cayman chemical.

Samples: A certified reference material for aflatoxin contaminated peanut butter (BCR-385R) was purchased from Sigma-Aldrich. A certified reference material for multi-toxin contaminated corn (MTC-9999A, Trilogy Analytical Laboratory, Inc.) was provided by a customer.

Methods

Samples were analyzed using a Shimadzu Nexera UC online SFE-SFC-MS system consisting of LC-30ADSF CO_2 pump, LC-30AD modifier pump, LC-30AD makeup pump, DGU-20A_{3R} and DGU-20A_{5R} degassing units, SIL-30AC autosampler, CTO-20AC column oven, two SFC-30A back pressure regulators, CBM-20A system controller, FCV-20AH_2 divert valve and LCMS-8060 mass spectrometer. The flow diagram is shown in Fig. 1.

1g of peanut butter was mixed with 1g of absorbent before loading to the extraction vessels, while 1g of powdered corn was put into the vessels as is (Fig. 2). The vessels were set on the online SFE-SFC-MS system. They were extracted on the SFE unit and then transferred to a 4.6 x 150mm column. The mycotoxins were detected by the triple quadrupole mass spectrometer.

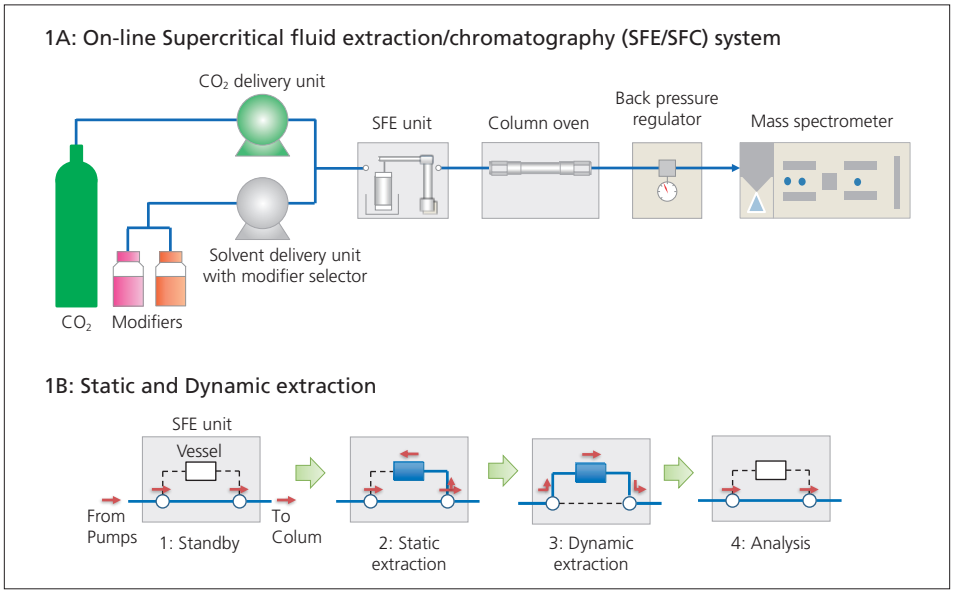

Fig.1 Flow diagrams of Nexera UC online SFE-SFC-MS system

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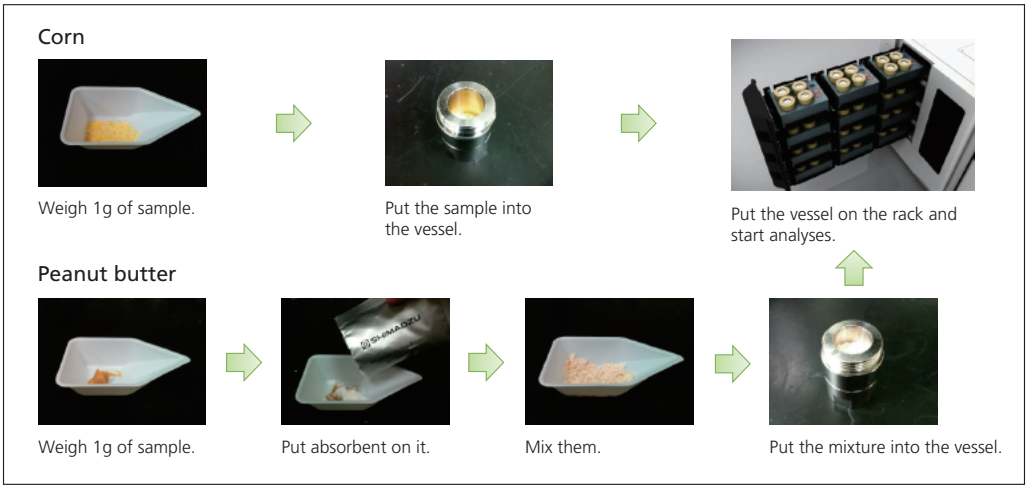

Fig.2 Sample preparation

Table 1 Analytical Conditions

System	: Shimadzu Nexera UC System with LCMS-8060		
[SFE] Nexera UC			
Extraction vessel	: 5 mL		
Static extraction	: Extraction time	: 2 min	
	B. Conc.	: 5 % (0-1 min), 3 % (1.01-2 min)	
	BPR pressure	: A: 149 bar (to column), B: 150 bar (to waste)	
	Flow rate	: 5.0 mL/min	
Dynamic extraction	: Extraction time	: 2 min	
	B. Conc.	: 3 % (2-6 min)	
	BPR pressure	: A: 149 bar (to column), B: 150 bar (to waste)	
	Flow rate	: 5.0 mL/min	
[SFC] Nexera UC			
Column	: Cosmosil π-NAP (4.6 mm x 150 mm, 5 μm)		
Mobile phase	: A: CO_2, B: 10 mmol/L ammonium acetate in methanol		
Flow Rate	: 3.0 mL/min		
Gradient program	: B. Conc. 3 % (6-7 min) – 18 % (32 min)		
Make-up solution	: Methanol		
Make-up flow rate	: 0.1 mL/min		
Column Temperature	: 40 °C		
BPR pressure	: A: 150 bar (to column), B: 400 bar (to waste)		
[MS Detection] LCMS-8060			
Ionization	: ESI (+/-)		
Probe voltage	: Used tuning file		
DL temperature	: 250 °C		
Block heater temperature	: 400 °C		
Interface temperature	: 300 °C		
Nebulizing gas flow	: 3 L/min		
Drying gas flow	: 5 L/min		
Heating gas flow	: 5 L/min		
Mode	: MRM		

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Results

Analysis of standard solution

A paper filter was placed in the extraction vessel, then spiked with 100 ng standard. Fig. 3 shows chromatogram of a 100 ng standard. T-2 toxin, HT-2 toxin, deoxynivalenol, zearalenone, and aflatoxins were successfully analyzed by online SFE-SFC-MS system.

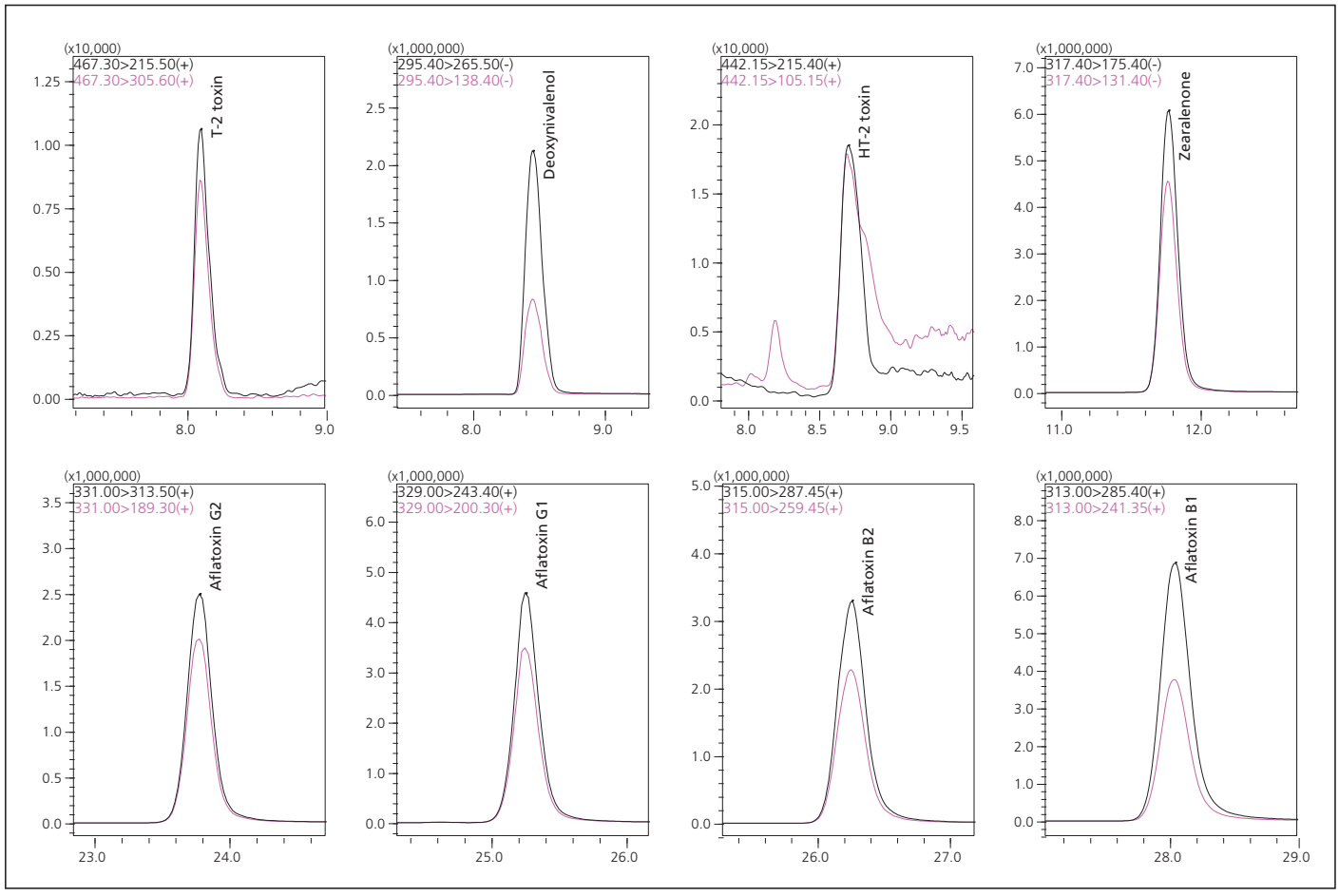

Fig.3 Chromatogram of 100 ng standard

Analysis of corn and peanut butter

Two certified reference materials (aflatoxin contaminated peanut butter and multi-toxin contaminated corn) were analyzed on the online SFE-SFC-MS system. Table 2 shows their certified value. Fig. 4 and 5 show chromatograms of corn and peanut butter, respectively. Aflatoxin G2 was not detected from the peanut butter, but almost all the analytes were successfully detected.

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Table 2 Certified values for mycotoxins in peanut butter and corn

	Corn MTC-9999A	Peanut butter BCR-385R
T-2 toxin	300 ± 57 ppb	N/A
HT-2 toxin	510 ± 83 ppb	N/A
Deoxynivalenol	2.2 ± 0.6 ppm	N/A
Zearalenone	265 ± 36 ppb	N/A
Ochratoxin A	N/A	N/A
Fumonisin B1*	19.9 ± 1.6 ppm	N/A
Fumonisin B2*	6.3 ± 0.8 ppm	N/A
Fumonisin B3*	1.5 ± 0.2 ppm	N/A
Aflatoxin B1	13.0 ± 3.2 ppb	1.77 ± 0.30ppb
Aflatoxin B2	0.6 ± 0.2 ppb	0.48 ± 0.08 ppb
Aflatoxin G1	1.6 ± 0.7 ppb	0.9 ± 0.4 ppb
Aflatoxin G2	N.D.	0.30 ± 0.12 ppb

*Note: Fumonisins were not analyzed in this study.

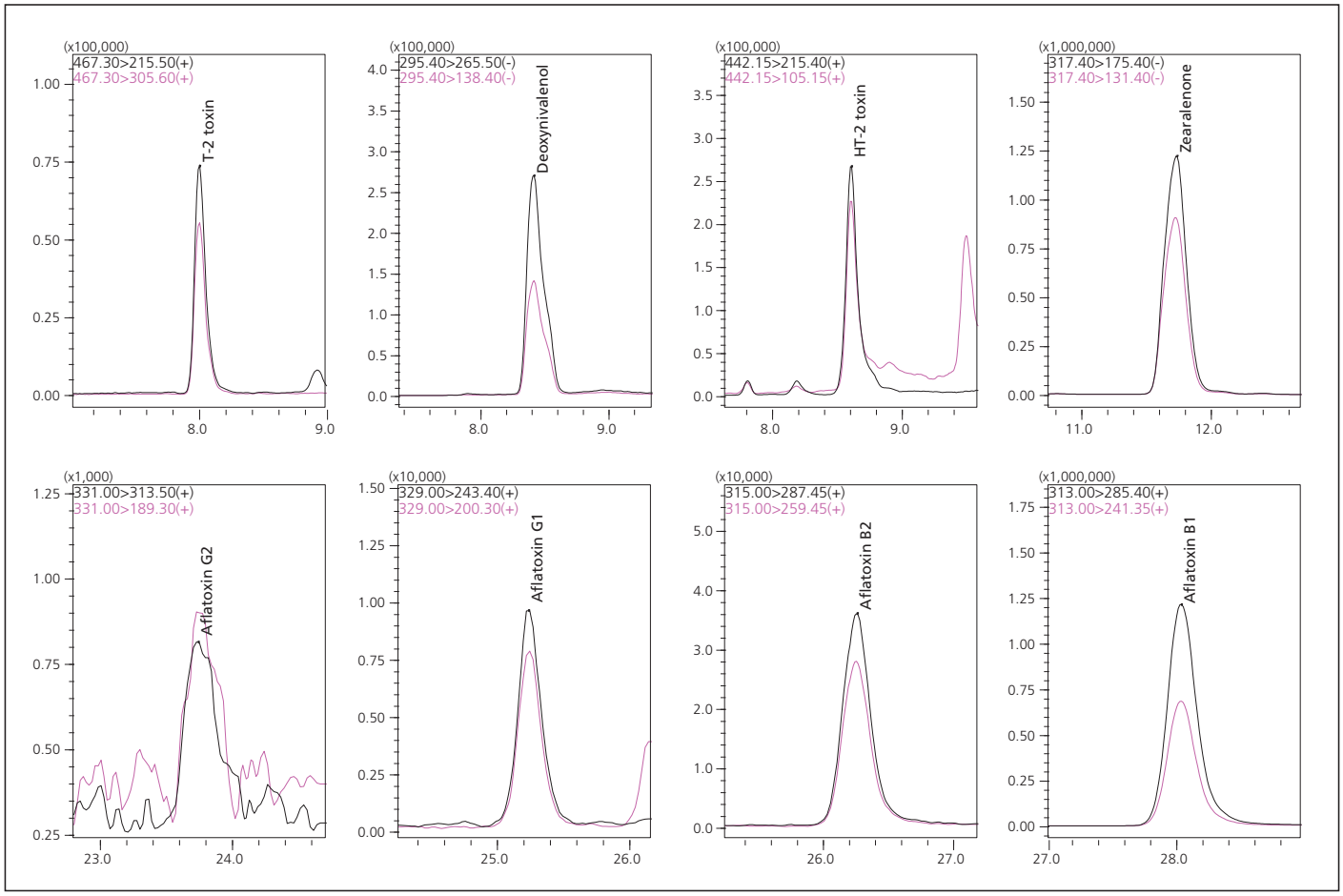

Fig.4 Chromatogram of multi-toxin contaminated corn

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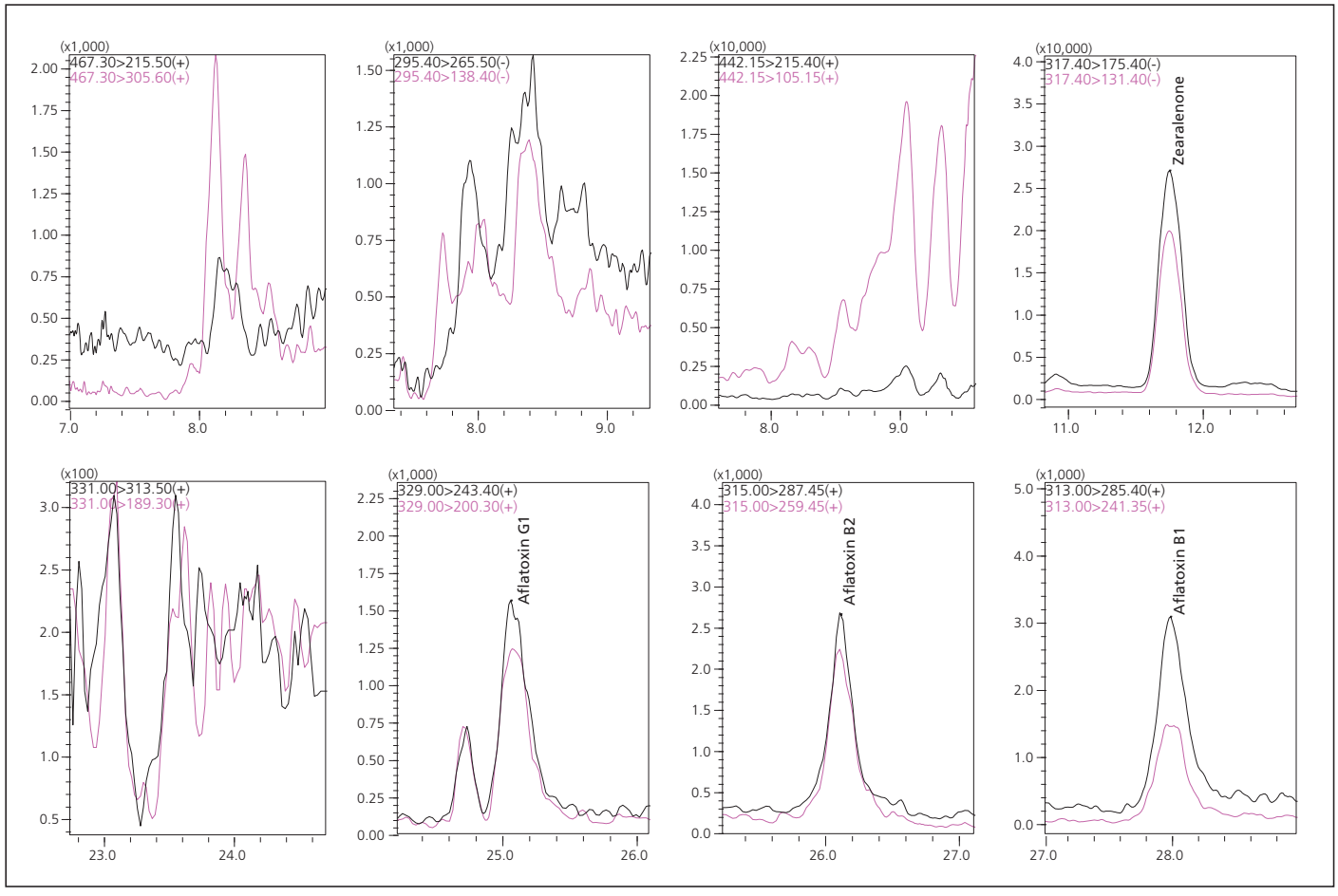

Fig.5 Chromatogram of aflatoxin contaminated peanut butter

Conclusions

- Nexera UC enables online SFE-SFC analysis which reduces labor for sample pretreatment.
- Established method was successfully applied to actual food samples.

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