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Short communication

Application of nanoring amino-functionalized magnetic polymer dispersive micro-solid-phase extraction and ultra fast liquid chromatography–tandem mass spectrometry in dicyandiamide residue analysis of powdered milk

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ABSTRACT

In this study, a rapid and accurate ultra-fast liquid chromatography–tandem quadrupole mass spectrometry (UFLC–MS/MS) method combined with dispersive micro-solid-phase extraction (d-μ-SPE) using a core–shell nanoring amino-functionalized magnetic polymer (CS-NR-MP) was established and validated to determine trace dicyandiamide (DCD) in powdered milk. The developed d-μ-SPE cleanup procedure can dramatically reduce the matrix in samples, and lead to a significant reduction in absolute matrix effects. Chromatographic separation was performed on an Acquity UPLC BEH Amide column by using water–acetonitrile (9:91, v/v) as the mobile phase within 2 min. DCD was quantitatively analyzed by using DCD-$^{15}N_2{}^{13}C_2$ as an internal standard. The results showed that the recoveries were between 99.8 and 105.6% with RSDs in the range of 0.5–4.9%. The target compound had good linearity in the range of 0.1–20.0 $\mu g\ L^{-1}$ with a correlation coefficient (r) of 0.9996. The limit of quantification (LOQ) was 0.06 $\mu g\ kg^{-1}$. This method can be used for the rapid and sensitive determination of ultratrace DCD residue in powdered milk samples.

1. Introduction

In recent years, the capacity for efficient and sensitive authentication of food ingredients is an important part of food safety programs, as illustrated by several incidents of adulteration of products such as milk. In 2007, the US Food and Drug Administration (FDA) determined that a wheat gluten ingredient was contaminated with melamine. In 2008, thousands of Chinese children experienced kidney problems as a result of melamine adulteration of infant formula. Unluckily, in September 2012, the presence of dicyandiamide (DCD, $C_2H_4N_4$) in powdered milk has been reported in New Zealand, which is used as nitrification inhibitor for increasing the nitrogen (N) utilization efficiency of grazed meadow. The Ministry for Primary Industries (MPI) reached the conclusion that DCD has not been considered to have any impact on food safety, even at the low levels detected by international regulators, however, most of customers are likely to view DCD residues as a contaminant [1]. Food regulators around the world are reflecting market demands with increasingly rigorous testing and in some countries there is a zero tolerance to detected residues outside agreed standards. Thus, there is a need to develop a rapid and accurate method to measure DCD in a wide variety of milk and milk products [2–4].

In the agricultural industry, for optimization of the application of DCD in agriculture and horticulture, high performance liquid chromatography (HPLC) method has been used for investigation of the degradation of DCD, as well as uptake by plants and loss by leaching [5–7]. However, the limit of quantification (LOQ) of HPLC method is too high to apply in the authentication of food ingredients in powdered milk. Recently, Qin et al. reported a new analytical method by using Raman chemical imaging for detecting trace DCD in powdered milk [4]. Though it is suited for screening of large-volume food samples, it is poor for detecting

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trace amounts of adulterants. High performance liquid chromatography coupled with tandem quadrupole mass spectrometry (LC–MS/MS) is an attractive alternative due to its simplicity, separation efficiency and excellent sensitivity and selectivity for both multi-residue determination and trace-level identification of a wide range of adulterants [8]. Recently, Abernethy and Higgs reported a LC–MS/MS method for qualitative analysis of DCD at mg L^{-1} level in fresh milk [9]. Yun et al. also reported a LC–MS/MS method for determination of DCD in milk and powdered milk, and the LOQ was 0.05 mg kg^{-1} [10]. Both the methods provide rapid and effective approaches to proactively combat economically motivated adulteration in protein-containing products rather than monitoring raw milk production made will contain trace DCD residues. However, according to the statement of MPI, DCD is one of a number of compounds that may be used to make the total nitrogen content of a food appear higher than it actually is, and DCD levels identified are very low and far below any level that could provide any economic advantage.

In LC–MS/MS method, matrix effects need to be evaluated during the method development because the accuracy and precision of a LC–MS/MS method could be significantly affected by this phenomena [11,12]. Endogenous matrix components co-eluting with the analyte of interest are believed to be the primary cause of ionization matrix effects. In the case of powdered milk and blood sample, phospholipids have been identified as a major contributing source of matrix effects in LC–MS/MS based bioanalytical methods [13–16]. Based on the amino-propyl SPE method used in the literature, we inspirited that a novel core–shell nanoring amino-functionalized magnetic polymer (CS-NR-MP) can be a powerful adsorbent to carry out dispersive micro-solid-phase extraction (d-μ-SPE) method for cleanup of the extracted powdered milk samples [17]. To the best of our knowledge, the application of d-μ-SPE coupled with LC–MS/MS method for quantification of trace level DCD in powdered milk has so far never been reported.

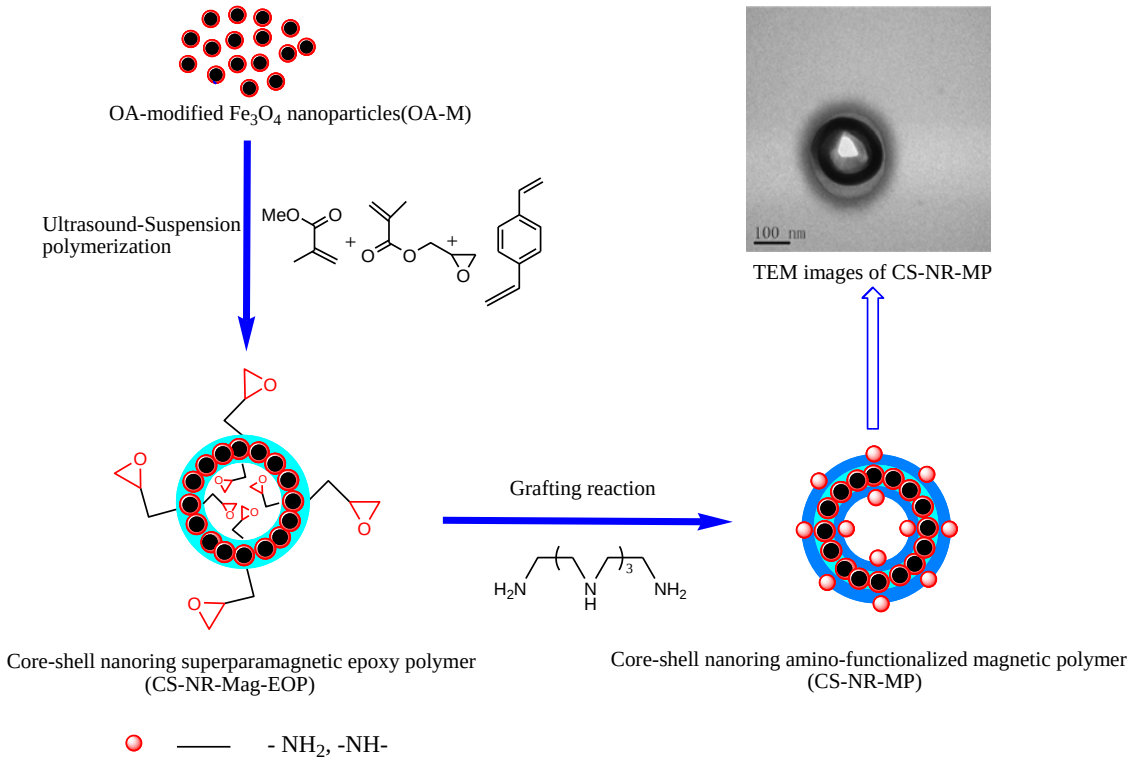
In this work, a new d-μ-SPE procedure using CS-NR-MP as adsorbent combined with ultra-fast liquid chromatography–tandem quadrupole mass spectrometry (UFLC–MS/MS) method has been developed for detection of trace DCD in powdered milk. The excellent sensitivity and selectivity of the developed method for DCD is investigated in laboratory batch tests, and it can be applied to the routine analyses for the determination of trace DCD residue in powdered milk samples.

2. Experimental

2.1. Reagents and materials

Acetonitrile of HPLC grade was obtained from Mallinckrodt Baker, Inc. (Phillipsburg, NJ, USA). Deionized water was purified using a Millipore water purification system (Millipore, Billerica, MA, USA). HPLC grade ammonium hydroxide solution and methanol were obtained from Merck Inc. (Darmstadt, Germany). Dicyandiamide (> 99.0%) and dicyandiamide-$^{15}N_2{}^{13}C_2$ (> 99.0%, internal standard) were obtained from Sigma-Aldrich (St. Louis, MO, USA). Powdered milk samples were collected from local markets.

CS-NR-MP used in the experiment was prepared in our laboratory according to the reported procedure for preparing core–shell nanoring amino-functionalized magnetic nonimprinted polymer (CS-NR-Mag-NIP) nanoparticles [18]. The CS-NR-MP obtained were ringed and exhibited a well-defined core–shell configuration. The Fe_3O_4 nanoparticles exhibited a uniform morphology with an average particle size of about 20 nm. The inside and outside diameters of the ringed CS-NR-MP nanoparticles were 70 and 150 nm, respectively, and the coated shell had an average thickness of approximately 10 nm. CS-NR-MP showed a saturation magnetization value of 7.12 emu g^{-1}. The preparation procedure of CS-NR-MP is illustrated in Scheme 1.



Scheme 1. Schematic procedures of CS-NR-MP nanoparticles.

2.2. Equipment

Ultra-fast liquid chromatography–tandem quadrupole mass spectrometry (UFLC–MS/MS) analyses were performed using a Prominence UFLC XR system equipped with a DGU-20A3 degasser, a CTO-20AC column oven, a LC-20AD pump, a SIL-20AC autosampler (Shimadzu Corporation, Tokyo, Japan) and an AB SCIEX TRIPLE QUAD™ 5500 mass spectrometer (Applied Biosystems, Foster City, CA, USA). The UFLC–MS/MS system was controlled and data were analyzed on a computer equipped with Applied Biosystems/MDS Sciex Analyst 1.5.1 (Applied Biosystems, Foster City, CA, USA).

2.3. UFLC–MS/MS analysis

The ultra-fast chromatographic separation was performed on an Acquity UPLC BEH Amide (100 mm × 2.1 mm i.d., 1.7 μm) by using water as eluent A (9%) and acetonitrile as eluent B (91%) at a constant flow of 0.35 mL min^{-1}, and the injection volume was 5.0 μL. The column temperature was held at 40 °C to increase the retention time reproducibility. The mass spectrometer was performed using an electrospray ionization (ESI) source in positive multiple reaction monitoring (MRM) mode, which was used for quantification. The operation conditions were as follows: ion spray voltage, 5500 V; curtain gas (CUR), 40 psi; interface heater, on; collision gas, medium; nebulizer gas (gas 1), 50 psi; heater gas (gas 2), 50 psi; turbo spray temperature, 500 °C; entrance potential (EP), 10 V; collision cell exit potential (CXP), 11 V. Nitrogen was used in all cases. The mass spectrometric information of the precursor ion, product ion, corresponding declustering potential (DP) and collision energy (CE) are shown in Table 1. The dwell time was set to 50 ms in the positive mode.

2.4. Sample preparation

Batch tests were conducted to evaluate excellent sensitivity and selectivity of the SPE UFLC–MS/MS method. 0.2 g sample was weighted into a 10 mL polypropylene centrifuge tube, and then 10 μL of 10.0 μg L^{-1} IS and 2.0 mL acetonitrile was added. The contents were homogenized for 2.0 min using an Ultra Turrax-mixer. Subsequently, the mixture was centrifuged at 6800 rpm for 3.0 min.

For liquid–liquid extraction (LLE) purification, the supernatant was transferred into another 10 mL polypropylene centrifuge tube, and then 2.0 mL *n*-hexane was added and vortexed for 5.0 min. The subnatant was transferred into a 5 mL polypropylene centrifuge tube, and concentrated with a nitrogen stream until the last drop of solution visibly disappeared. The residues were redissolved in 200 μL acetonitrile, vortexed, and transferred into an autosampler vial for UFLC–MS/MS analysis.

For PSA SPE purification, the cartridge was pretreated with 6.0 mL acetonitrile, afterwards, the supernatant was loaded onto the PSA cartridge, and washed by another 6.0 mL acetonitrile. The PSA cartridge was eluted with 6.0 mL 5% ammonia (v/v) in methanol at a flow rate of 1.0 mL min^{-1}. The final eluate was concentrated with a nitrogen stream until the last drop of solution visibly disappeared. The residues were redissolved in 200 μL acetonitrile, vortexed, and transferred into an autosampler vial for UFLC–MS/MS analysis.

For CS-NR-MP d-μ-SPE purification, 20 mg of CS-NR-MP were added to the supernatant, and the mixture was immediately machine-shaken for 1.0 min, then separation under a magnetic field for 2 min. The adsorbent was eluted with 6.0 mL 5% ammonia (v/v) in methanol. The final eluate was concentrated with a nitrogen stream until the last drop of solution visibly disappeared. The residues were redissolved in 200 μL acetonitrile, vortexed, and transferred into an autosampler vial for UFLC–MS/MS analysis.

Table 1
Q1/Q3 ion pairs, declustering potential (DP), collision energy (CE) of MRM and retention time for DCD and DCD-$^{15}N_2{}^{13}C_2$.

Compounds	Precursor ion (Q1, *m/z*)	Fragment ion (Q3, *m/z*)	DP (V)	CE (eV)	Retention time (min)
DCD	85.0	68.0*, 43.0	100, 100	34, 40	1.21
DCD-$^{15}N_2{}^{13}C_2$	89.1	71.0*, 45.0	70, 70	25, 25	1.22

* Quantitative ion.

2.5. Method validation

2.5.1. Standard preparation

Individual stock standard solutions were prepared at a concentration of 1000.0 mg L^{-1} by exact weighing and dissolution in water–acetonitrile (1:1, v/v). These solutions were stored at 4 °C in the dark. Working standard mixture solution at a concentration of 100.0 mg L^{-1} was prepared by appropriate dilution of the stock solutions with water–acetonitrile (1:1, v/v).

2.5.2. Spiked samples

Spiked recoveries were performed at concentrations of 0.1, 1.0, and 10.0 μg kg^{-1} (equivalent to 0.1, 1.0 and 10.0 μg L^{-1}) for DCD in the samples. For each spiked sample, the stock solution of the standards and 10 μL of 10.0 μg L^{-1} IS were added to 0.2 g of powdered milk, which was free from the target compounds. The spiked samples were stored at 4 °C for about 12 h to let the DCD permeate uniformly into the samples. Recoveries at each level were run along with both a reagent and a blank sample.

2.5.3. Validation parameters

The method was evaluated by linearity, LOD and LOQ, precision and accuracy. Calibration standards in acetonitrile with concentrations at 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, 10.0 and 20.0 μg L^{-1} were prepared for the calibration curves. All of the IS were prepared at a concentration of 0.5 μg L^{-1}. Calibration curves of peak area ratio of quantitative ion pairs and IS against the analyte concentration were used to calibrate the UFLC–MS/MS system and spike samples in recovery experiments. LOD and LOQ were determined based on a signal-to-noise ratio of 3 (S/N=3) and 10 (S/N=10), respectively. Both the method precision and accuracy were estimated. The intra-day and inter-day precision were investigated by injection of the three levels of spiked samples 0.1, 1.0, and 10.0 μg kg^{-1} with six replicates and replicated on six different days, respectively. Accuracy of the method was also checked by spiking 0.1, 1.0, 10.0 μg kg^{-1} of DCD in the samples.

3. Results and discussion

3.1. Optimization of UFLC–MS/MS conditions

A UFLC–MS/MS method was established to determine the presence of DCD in powdered milk, the Acquity UPLC BEH Amide (100 mm × 2.1 mm i.d., 1.7 μm) were employed to achieve the maximum sensitivity and the satisfactory peaks by using water and acetonitrile as eluent (9:91, v/v), at a constant flow of 0.35 mL/min.

The confirmation procedures by the Commission Decision 2002/657/EC (Commission Decision 2002/657/EC 2002) for banned substances were established by using a minimum of four identification points. In this experiment, four identification points, one parent (1.0 point) and two transitions (each 1.5 points) were monitored. In the work, UFLC–ESI-MS/MS methods of DCD residues

have investigated three ways to chose precursor ions including $[M+Na]^+$, $[M+H]^+$ and $[M-H]^-$. We chose $[M+H]^+$ as the precursor ion because the ion was the most abundant peak in the mass spectra, and MS/MS spectra of the precursor ion at m/z 85.0 is shown in Fig. 1. Furthermore, in order to improve the qualitative accuracy, the MS/MS spectrum of IS, i.e., DCD-$^{15}N_2{}^{13}C_2$, is illustrated in Fig. 1. According to the confirmation procedures by the Commission Decision 2002/657/EC, DCD was quantitatively analyzed by using DCD-$^{15}N_2{}^{13}C_2$ as an IS.

3.2. CS-NR-MP d-μ-SPE procedure and its optimization

Optimization of the sample preparing method was a necessary step to remove matrix and obtain good signal response. Matrix effects, a phenomenon of ion suppression or enhancement of the analyte of interest, the absolute matrix effect may be quantitatively assessed by comparing the response of the analyte spiked into extracted blank matrix with the response of the analyte spiked into matrix-free reconstitution solution. In this work, the matrix effect was evaluated, and the initial results showed that the absolute matrix effects ranged from 36.9% to 42.2% at the tested concentrations (Table 2).

Therefore, LLE and several kinds of SPE cartridges such as Alumina-N-SPE, NH_2-SPE, MCX-SPE, PSA-SPE, and CS-NR-MP d-μ-SPE were investigated. And CS-NR-MP d-μ-SPE was used to prepare the powdered milk at last, because it could make the sample contain less matrix interference than others which is protective to the column and the ESI source. The result is shown in Table 2. In addition, the effect of organic solvents, extraction

Table 2
Absolute matrix effect and recovery of DCD in powdered milk under various extraction conditions.

Analyte	Added ($\mu g\ kg^{-1}$)	Mean absolute matrix effect[a]			
		Without purification (%, $n=6$)	With LLE purification (%, $n=6$)	With PSA-SPE purification (%, $n=6$)	With CS-NR-MP d-μ-SPE purification (%, $n=6$)
DCD	0.1	36.9	49.6	76.0	106.2
	1.0	43.8	48.1	73.1	100.1
	10.0	42.2	50.6	69.2	102.8

[a] Expressed as (the mean peak area of analyte spiked after extraction/the mean peak area of the neat analyte standard) × 100%.

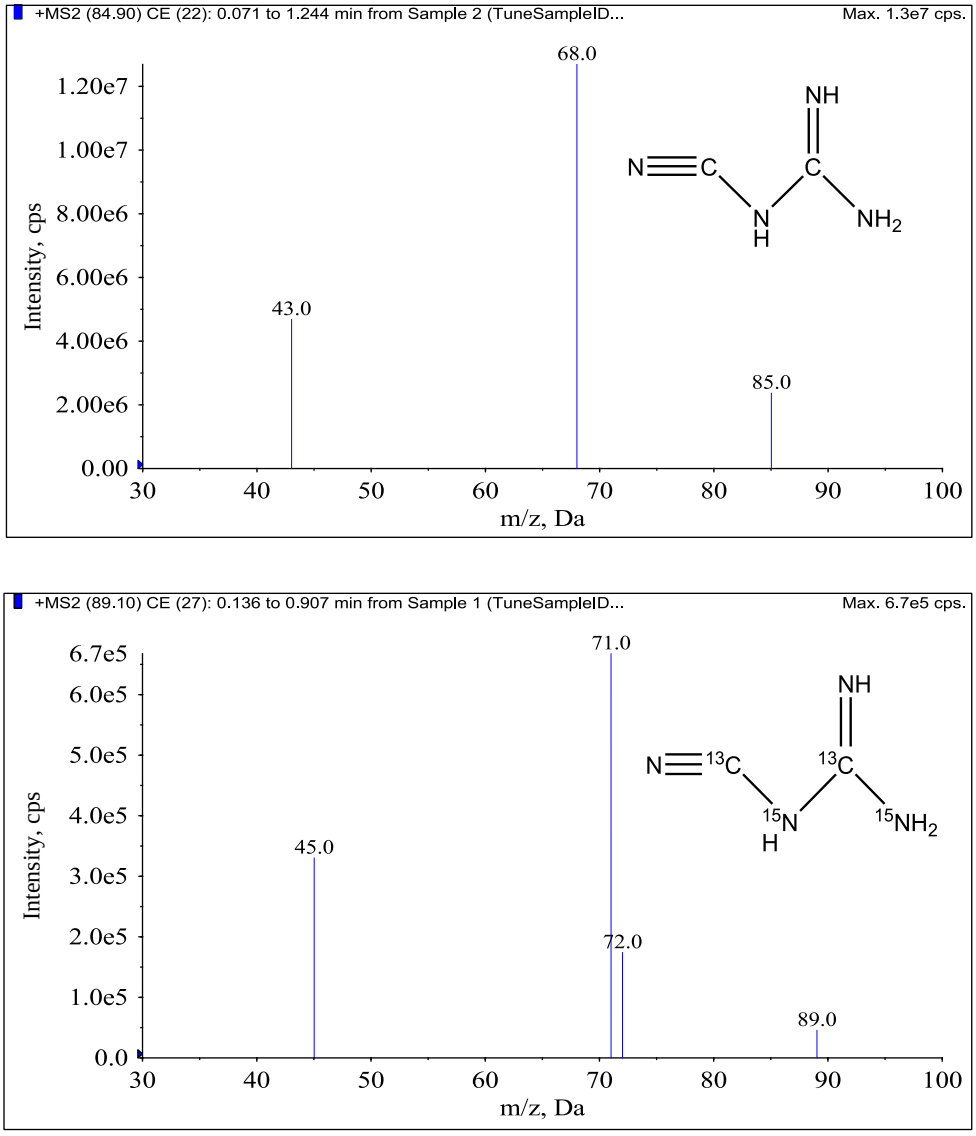


Fig. 1. MS/MS spectra of DCD and DCD-$^{15}N_2{}^{13}C_2$.

time, the amount of CS-NR-MP on the d-μ-SPE cleanup properties were investigated in detail. And the results of recycling experiment showed that CS-NR-MP could be reused at least six times without much sacrifice of the cleanup efficiency. The preparative reproducibility of the CS-NR-MP used for the cleanup procedure was deeply evaluated. And the results show that the absolute deviations of the recoveries for DCD by the three batches are less than 5.0%. This indicates that the preparation procedure of CS-NR-MP has good repeatability and reproducibility. So the CS-NR-MP d-μ-SPE combined with ultra-fast liquid chromatography–tandem quadrupole mass spectrometry (UFLC–MS/MS) method has been developed for detection of trace DCD in powdered milk.

3.3. Method linear range, precision, accuracy, LOD and LOQ

The calibration curve was obtained by plotting the relation between the peak area ratio ($Y=A_{sample}/A_{IS}$) against the analyte concentration ratio ($X=C_{sample}/C_{IS}$). The response function of DCD was linear with a correlation coefficient (r) higher than 0.9996 (linear equation: $Y=0.7961X+0.1830$). Precision and accuracy were assessed based on the analysis of DCD spiked at concentrations of 0.1, 1.0 and 10.0 $\mu g\ kg^{-1}$ in blank powdered milk. The majority of mean recoveries are in the range of 99.8–105.6% at the three spiking levels with associated intra-day relative standard deviations (RSDs) ranging from 0.5% to 3.9% and inter-day RSDs ranging from 0.8% to 4.9%. The limit of detection (LOD) and the limit of quantification (LOQ), which were calculated in blank extracts as the lowest analyte concentration that yielded a signal-to-noise (S/N) ratio of 3 and 10, were 0.02 $\mu g\ kg^{-1}$ and 0.06 $\mu g\ kg^{-1}$, respectively.

In our study, the total run time was just 2 min, and a good peak shape of the DCD was achieved under the specified UFLC–MS/MS conditions. No interference was found at the retention time of the DCD. The MRM chromatograms for DCD spiked at 0.2 $\mu g\ kg^{-1}$ and blank powdered milk sample are shown in Fig. 2. Furthermore, LC or LC–MS-based analysis for the determination of DCD in various milk samples have been listed in Table 3. It shows that the limit of quantification (LOQ) of the developed method is much lower than other LC or LC–MS methods.

3.4. Sample analysis

Three batches of powdered milk (ten samples for each batch) were analyzed by the developed method. Each batch of samples was processed together with a matrix blank (DCD-free sample), which was confirmed by using UFLC–MS/MS method. The blank matrix eliminated the false positive as result of contamination in the extraction process, instrument or chemicals. A blank extract spiked at the calibration level (5.0 $\mu g\ L^{-1}$) was used to control the extraction efficiency. The DCD was identified by comparison of its retention time and fragment ions with the related standard compound. The obtained results showed DCD was not detected because of lower LOQ (0.06 $\mu g\ kg^{-1}$) in the analyzed thirty samples.

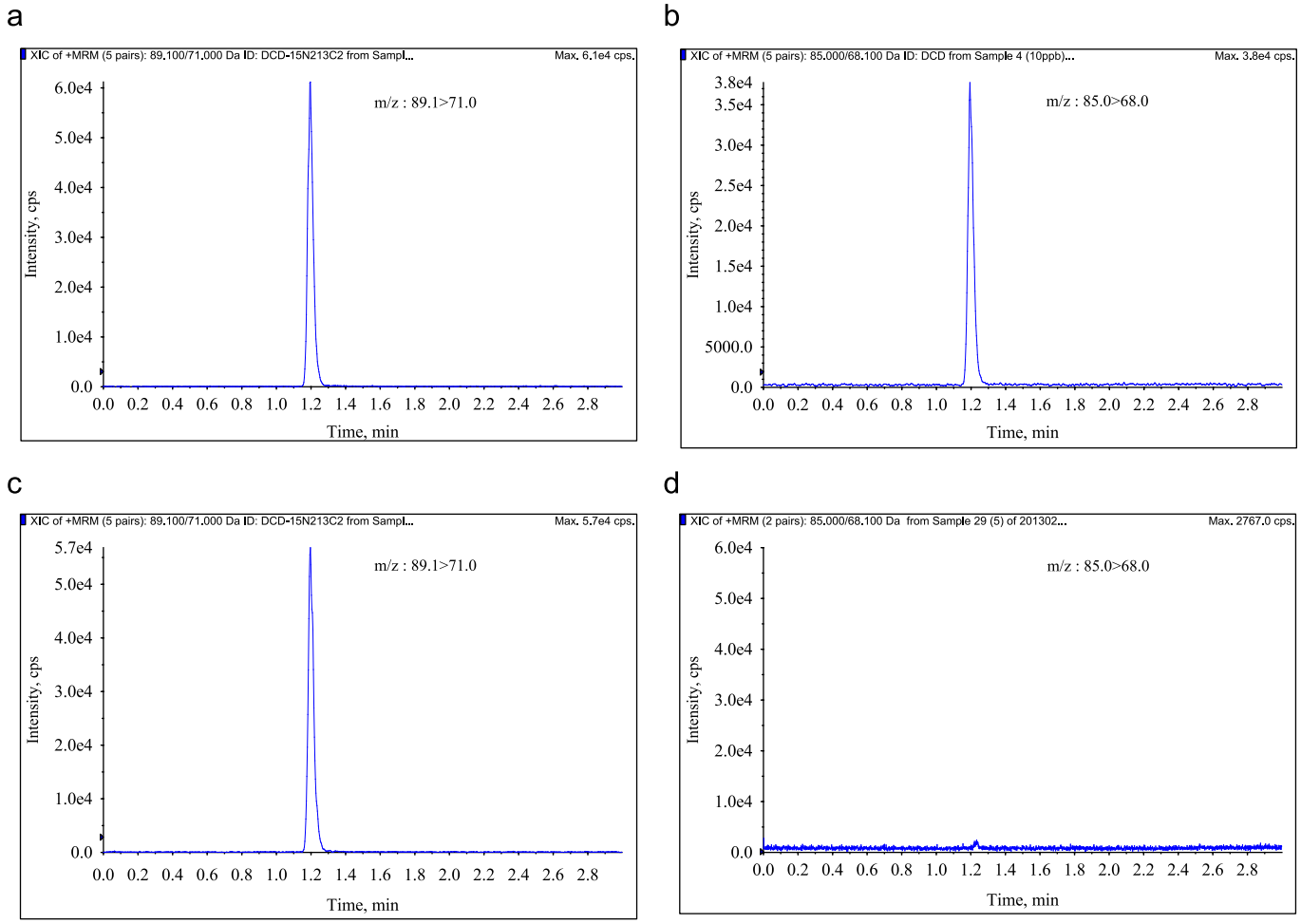


Fig. 2. Extract ion chromatograms (XIC) of spiked powdered milk at a concentration of 0.2 $\mu g\ kg^{-1}$ with IS at a concentration of 0.5 $\mu g\ kg^{-1}$ (a: DCD-$^{15}N_2{}^{13}C_2$; b: DCD) and blank powdered milk sample (c: DCD-$^{15}N_2{}^{13}C_2$; d: DCD). (a) m/z: 89.1 > 71.0, (b) m/z: 85.0 > 68.0, (c) m/z: 89.1 > 71.0 and (d) m/z: 85.0 > 68.0.

Table 3
Comparison of the analytical features of current LC/LC–MS-based methodologies for the determination of DCD in milk samples.

Sample	Analyte	Sample preparation (main steps)	LC column	LC parameters	Detector parameters	Linear range, method LOQ, recoveries (*R*)	Reference
Powdered milk	Dicyandiamide	d-μ-SPE	Acquity UPLC BEH Amide column 100 mm × 2.1 mm, 1.7 μm	Mobile phase: water–acetonitrile (9:91, v/v) 0.35 mL min^{-1}, Inj: 5 μL	ESI(+)–MS/MS: capillary voltage: 5.5 kV, source temperature: 500 °C	LR: 0.1–20 μg L^{-1}; LOQ: 0.06 μg kg^{-1}; *R*: 99.8–105.6%	This work
Skim milk	Cyromazine Triuret Biuret Dicyandiamide Melamine Amidinourea Urea	LLE	SeQuant ZIC-HILIC column 150 mm × 2.1 mm, 5.0 μm	Mobile phase: acetonitrile (0.1% formic acid)/water (10 mmol L^{-1} ammonium acetate), with gradient elution, 0.4 mL min^{-1}, Inj: 20 μL	ESI(+)–MS/MS: capillary voltage: 5.0 kV, source temperature: 550 °C	LR: 0.8–800 mg L^{-1}; LOQ: 0.18–28.8 mg · L^{-1}; *R*: 95.9–102.5	[8]
Milk and milk powder	Dicyandiamide Melamine Cyanuric acid	LLE	Acquity UPLC HSS T3 column 50 mm × 2.1 mm, 1.8 μm	Mobile phase: water (10 mmol L^{-1} ammonium acetate)/acetonitrile with gradient elution, 0.3 mL min^{-1}, Inj: 10 μL	ESI(+/−)–MS/MS	LR: 5–200 μg L^{-1}; LOQ: 0.02–0.05 mg kg^{-1}; *R*: 60.0–115.0%	[10]
Dairy products	Dicyandiamide	LLE	XBridge Amide column 250 mm × 4.6 mm, 3.5 μm	Mobile phase: water (0.2% formic acid)/acetonitrile (0.2% formic acid) (10/90, v/v) 1.0 mL · min^{-1}, Inj.: 20 μL	UV detector: wavelength: 218 nm	LR: 0.5–50 mg L^{-1}; LOQ: 0.5 μg kg^{-1}; *R*: 96.7–101%	[19]

Abbreviations: LLE, liquid–liquid extraction; d-μ-SPE, dispersive micro-solid-phase extraction; Inj., injection volume; ESI, electrospray ionization; LR, linear range; LOQ, limit of quantitative; *R*, recovery.

4. Conclusions

In this study, a rapid and accurate ultra-fast liquid chromatography–tandem quadrupole mass spectrometry (UFLC–MS/MS) method was established to determine dicyandiamide (DCD), and the total run time was 2 min. The effective d-μ-SPE extraction procedure using CS-NR-MP was optimized for the cleanup of DCD in powdered milk, and the matrix effects were effectively eliminated by the CS-NR-MP adsorbent. Acceptable recoveries for DCD were obtained in the range of 99.8–105.6%. The results demonstrate that the accuracy and precision of the proposed CS-NR-MP d-μ-SPE coupled with UFLC–MS/MS method are satisfactory for analysis of trace DCD in powdered milk samples.

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