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Determination of triazines in infant nutrient cereal-based foods by pressurized microwave-assisted extraction coupled with high-performance liquid chromatography–mass spectrometry

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Abstract

A method for determining triazine herbicides in infant nutrient cereal-based foods by pressurized microwave-assisted extraction (PMAE), coupled with high-performance liquid chromatography–electrospray ionization mass spectrometry (HPLC–ESI/MS), is described. The key parameters of PMAE, including extraction solvent, extraction time and temperature, were optimized. The isolation of the target compounds from the matrix was found to be efficient when 2 g of nutrient cereal samples was extracted with 20 mL of methanol for 10 min at 105 °C. Final determination was accomplished by HPLC–ESI/MS. The recoveries from 66.2 to 88.6% were obtained for three compounds at fortification levels (5–500 $\mu g\,kg^{-1}$) with relative standard deviations (R.S.D.) ≤12.62%. Compared with atmospheric pressure microwave-assisted extraction (AMAE), ultrasonic extraction (UE) and soxhlet extraction (SE), the proposed method was more efficient, faster and more straightforward and required no additional cleanup steps. When the proposed method was applied to the aged spiked nutrient cereal samples, the results indicated that, although the recoveries of analytes were much lower than those obtained from fresh spiked samples, they were nevertheless satisfactory for the quantitative analysis of practical samples.

Keywords: Extraction methods; Infant nutrient cereal-based foods; Triazines

1. Introduction

Children can easily be exposed to pesticides by inhaling contaminated air, ingesting tainted food or non-dietary substances (i.e., dust or soil) or by absorbing them through the skin from contaminated media. It was suggested that [1] dietary ingestion may be an important pathway for children to be exposed to persistent organic pollutants including pesticides. Commission Directive 2006/125/EC on processed cereal-based foods and baby foods for infants and young children requires that baby food contains no detectable levels of pesticide residues, meaning not more than $10\,\mu g\,kg^{-1}$ based on the advice of the Scientific Committee on Food. In addition, the Directive prohibits the use of certain very toxic pesticides (excluding triazines) in the production of processed cereal-based baby foods and baby foods and establishes levels lower than the general maximum level of $10\,\mu g\,kg^{-1}$ for a few other very toxic pesticides. Triazines, important herbicides used in agriculture worldwide, are ubiquitous environmental pollutants. Furthermore, since they are designed to kill living organisms, they represent potentially hazardous chemicals, which can cause injury to humans if exposure is at inappropriately high levels. Infant cereal-based foods are important energy source for the nutrition of infants to form the basis of their weaning–feeding from the age of 4–6 months. The study of infant cereal from a health standpoint is important because ablactation can be an important process for eliminating triazine compounds, which are gradually ingested with the diet and accumulate in adipose tissue, from the human organism. Moreover, the high consumption of infant cereal-based foods by babies throughout the world makes the determination of triazines in baby foods the subject of much concern.

Soxhelt extraction (SE) and liquid–liquid extraction (LLE), commonly considered as the benchmark extraction techniques, have been the traditional methods used for the extraction of pes-

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ticides residues from matrix. The two methods are often effective but time consuming, labour intensive and usually require large amounts of solvent and sample. In recent years, extraction methods based on instrumental techniques such as microwave-assisted extraction (MAE) [2,3,9], supercritical fluid extraction (SFE) [4,5] and the ultrasonic extraction (UE) have also been reported. The rapidity, simplicity, low cost of operation and high sample throughput obtained by MAE make this technique to be developed into a good alternative to traditional extraction methods and become a popular routine technique in environmental analysis, especially in organic analysis [6]. Widespread use of microwaves for analytical purpose have been found in very different areas including clinical, food and environmental analysis [7–9], but to our knowledge, the reports about the use of microwaves for the extraction of triazine herbicides in nutrient cereal samples are scarce. Two types of microwave extraction system are commercially available: the closed and the open-vessel systems [10]. In the former, the extraction process is often carried out at high pressure, namely pressurized microwave-assisted extraction (PMAE), and accelerated with increasing temperature of extraction system. The latter is also referred as a focused microwave-assisted extraction performed in an open vessel, which is known as atmospheric pressure microwave-assisted extraction (AMAE), and is more suitable for preparing large amounts of sample [11,12].

Determination of pesticides in food, especially in heat-treated foods such as infant food, is often complicated in the presence of fats and proteins. The proper determination of triazines was predominantly performed by high-performance liquid chromatography–ultraviolet detection (HPLC-UV) [9,13] and gas chromatography–mass spectrometry (GC–MS) [14,16,17]. The high-performance liquid chromatography–mass spectrometry (HPLC–MS) [15], which is complementary to GC–MS analysis, is sensitive, selective and suitable for monitoring compounds that are thermally unstable or non-volatile.

In this paper, an analytical method using PMAE, coupled with high-performance liquid chromatography–electrospray ionization mass spectrometry (HPLC–ESI/MS) was developed for the determination of residues corresponding to the three triazine herbicides (Fig. 1), namely atrazine (6-chloro-N^2-ethyl-N^4-isopropyl-1,3,5-triazine-2,4-diamine), simazine (6-chloro-N^2,N^4-diethyl-1,3,5-triazine-2,4-diamine) and prometryn (N^2,N^4-diisopropyl-6-methylthio-1,3,5-triazine-2,4-diamine) from infant nutrient cereal-based samples. But for all samples spiked 500 μg kg^{-1}, quantification of three triazine compounds was based on HPLC–DAD.

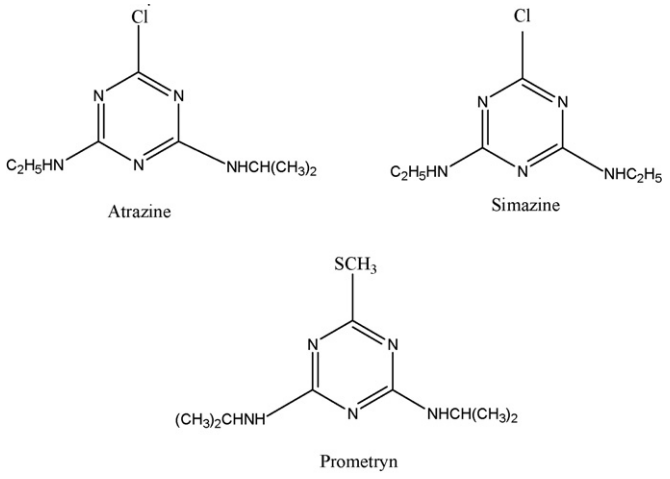

Fig. 1. Structures of three triazine herbicides.

2. Experimental

2.1. Chemicals and instrument

2.1.1. Chemicals

Chromatographic grade acetonitrile and methanol were from Fisher Scientific Company (UK). All other reagents were of analytical-reagent grade and from Beijing Chemical Factory (Beijing, China). Infant cereal-based foods were purchased from a major local supermarket. Atrazine, simazine and prometryn standards were obtained from National Institute for the Control of Pharmaceutical and Biological Products (Beijing, China). Standard stock solutions for triazines of the concentration level of 1000 μg/mL were prepared in acetonitrile. Pure water was obtained with a Milli-Q water purification system (Millipore Co., USA).

2.1.2. Spiked sample preparation

In the study, all experiment were performed on some representative infant nutrient cereal-based foods including six kinds of rice cereals suitable for infant at the age of 4–24 months (Sample 1), 6–24 months (Sample 2) and 8–24 months (Sample 3), nutrient cereal with Vitamins A and D and calcium (Sample 4), high-protein cereal (Sample 5) and nutrient cereal with fruit and vegetable granules (Sample 6). Except for the experiments mentioned in Section 3.2.3, which were performed on all six kinds of samples, all other results were obtained with Sample 3. The fresh spiked nutrient cereal-based samples containing atrazine, simazine and prometryn at concentration levels of 500, 100, 50 and 5 μg/kg were prepared by spiking the three stock standard triazine solutions into 2 g of triazine-free cereals mentioned above. To ensure the triazines to be well distributed, a reasonable amount of acetone was added to moisten the cereal and careful agitation was performed followed by an air-drying for 24 h at ambient temperature before extraction.

The aged spiked samples containing simazine, atrazine and prometryn at concentration level of 50 and 100 μg/kg were stored in the dark for 45 days at about 4 °C after air-drying overnight at ambient temperature.

2.1.3. Instruments

The 1100 series liquid chromatograph (Agilent Technologies Inc., USA) equipped with photodiode-array detector (DAD) and quaternary gradient pump was used. Separation of the analytes was performed upon Zorbax Eclipse XDB-C8 (5 μm, 4.6 mm × 150 mm, Agilent). An ABI Q-Trap Mass Spectrometer (Applied Biosystems Sciex, Foster City, USA) was used. The instrument is equipped with electrospray ionization (ESI)

source and interfaced to a computer running Applied Biosystems Analyst Version 1.4 software.

2.1.4. Chromatographic conditions

Before analysis by HPLC, the solution was filtered through a 0.45 μm filter membrane. The flow rate of the mobile phase was kept at 1 mL/min. Mobile phases A, B and D were methanol, water and acetonitrile, respectively. The gradient conditions were as follows: 0–7 min, 60% B and 20% D; 8–14 min, 60–50% B and 20%–50% D; 15–24 min, 50% B and 50% D. Under the optimal HPLC program, the retention time for simazine, atrazine and prometryn is 6.6, 11.8 and 18.6 min, respectively. When HPLC–DAD was applied, the injection volume was 20 μL. The detection was finished at a wavelength of 222 nm and a reference wavelength of 360 nm. The slit width was 16 nm.

2.1.5. Mass spectrometric conditions

Nitrogen (99.999%) was used for nebulizer gas and curtain gas. The ion polarity was set to positive mode. User controlled voltages, gas pressures and source temperature were optimized for the detection of the parent and product ions of triazines. The source temperature was set to 500 °C. The curtain gas, gas 1 (nebulizer gas) and gas 2 (turbo gas) was 35, 35 and 70 psi, respectively. The ion spray and entrance potential were 3000 and 10 V, respectively. The declustering potential, collision energy and collision cell exit potential were optimized at 80 V, 40 eV and 30 V, respectively, for triazines. Both Q1 and Q3 were set to unit resolution. When HPLC–ESI/MS was applied, the injection volume was 5 μL and the mass spectra of three triazines are given in Fig. 2. The following precursor → product ion pairs were monitored in multiple reaction monitoring (MRM) mode:

- atrazine $m/z = 216.2 \rightarrow 174.1$,
- simazine $m/z = 202.1 \rightarrow 67.7$,
- prometryn $m/z = 242.2 \rightarrow 158.2$.

2.2. Extraction procedure

2.2.1. PMAE

PMAE was performed on a WRT-C microwave preparation system (Michem Technology Co. Ltd., Beijing, China) with a pressure and temperature control system. In the present work, only temperature control system was applied. The extraction vessel consists of a vessel body (PEEK) and a liner vessel (TFM). Two grams of sample was transferred into the liner vessel of the extraction vessel and then 20 mL methanol was added into it. The liner vessel was put into the vessel body. To monitor the temperature, the control vessel was connected to the temperature control device. After being closed, the extraction vessels and control vessel were put into the microwave sample preparation system. The magnetron power output of the microwave unit was set at 100% (600 W). Then the system was turned on and the temperature in the extraction vessel gradually increased until it reached to the preset 105 °C. When the temperature reached the preset value, the irradiation time was counted. The extraction was continuously performed for the given 10 min under 105 °C. After the completion of extraction, the sample vessel was allowed to cool down to the room temperature for about 20 min. Finally, the extract was filtrated and transferred into a 100 mL flask. The liner vessel and the sediment were rinsed three times with methanol and the rinsed solvent was also transferred into the flask. The mixture was dried by rotatory evaporator at 45 °C, redissolved using 1 mL acetonitrile and followed by centrifugation.

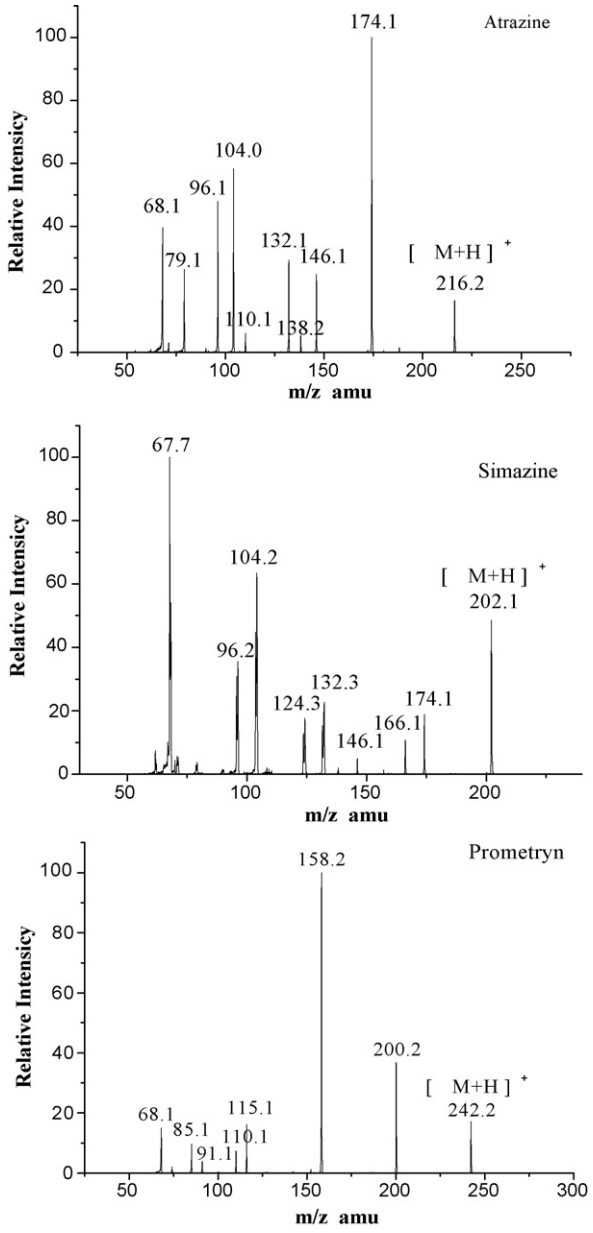

Fig. 2. Mass spectra of three triazine herbicides.

2.2.2. AMAE

The modified household microwave oven with the 30% output maximum power of 800 W and a distilling flask (100 mL) fitted with a water-cooling condenser tube were used. Two grams of dried sample was weighed and transferred into the distilling flask and dissolved by 40 mL of methanol. The mixture was refluxed for 20 min. When the extraction was completed, the

extract was cooled down to room temperature. Then the extract was filtrated and transferred into a 100 mL flask. The distilling flask and the sediment were rinsed three times with methanol and the rinsed solvent was also transferred into the flask. Then 1 mL of acetonitrile was employed to reconstitute the residue and the mixture was centrifugated prior to analysis.

2.2.3. UE and SE

UE was performed on the Ultrasonic Cleaners and 2 g of spiked sample was exactly weighted and placed into 100 mL flask, into which 40 mL of methanol was added. This flask was immersed in a water bath and sonicated for 90 min. After completing extraction, the extract was filtered through ashless filter paper. The flask was rinsed three times with methanol. Both collected extracts and rinsed solvent were transferred into 100 mL flask and dried under rotatory evaporator at 45 °C. The residue was redissolved by 1 mL acetonitrile and then centrifugated.

For Soxhlet extraction (SE), 2 g of spiked dried sample was placed in a glass Soxhlet thimble and 40 mL methanol was added into a 100 mL distilling flask. The flask was fitted with a water-cooling condenser tube and was immersed in a water bath. SE was carried out for 2 h at 100 °C. When the extraction was finished, the extract was filtrated and transferred into a 100 mL flask. The distilling flask and the sediment were rinsed three times with extraction solvent, and then the rinsed solvent was added into the flask followed by drying under rotatory evaporator at 45 °C and the residue was reconstituted by 1 mL acetonitrile and then centrifugated prior to analysis.

3. Results and discussion

3.1. Selection of PMAE conditions

3.1.1. Selection of extraction solvent

The effect of different extraction solvents on target compound recoveries was studied. The methanol, acetonitrile, acetone, acetone–*n*-hexane (1/1, v/v), methanol–dichloromethane (1/1, v/v) and water were used as the extraction solvents. When water was used as extraction solvent, the experimental results indicated that water was not suitable for extraction because of emulsification of extract and low recovery. The required microwave radiation time to reach a temperature of 85 °C for the extraction solvents was tested and the radiation time was increased in the order: methanol (241 s) < dichloromethane–methanol (1/1, v/v) (250 s) < acetonitrile (279 s) < acetone (289 s) < acetone–*n*-hexane (1/1, v/v) (310 s) < water (404 s) < dichloromethane (426 s). The rate of temperature increase seemed to be related to the solvent's ability to absorb the microwave energy and its specific heat. The influence of the solvent on the recovery of triazines is shown in Fig. 3. The results indicated that the highest mean extraction yields for the three triazine herbicides were obtained by using 10 mL methanol as extraction solvent.

The effect of volume of extraction solvent on extraction yields was also studied using methanol as solvent. It can be seen from Fig. 4 that 20 mL methanol offers the highest extraction yields.

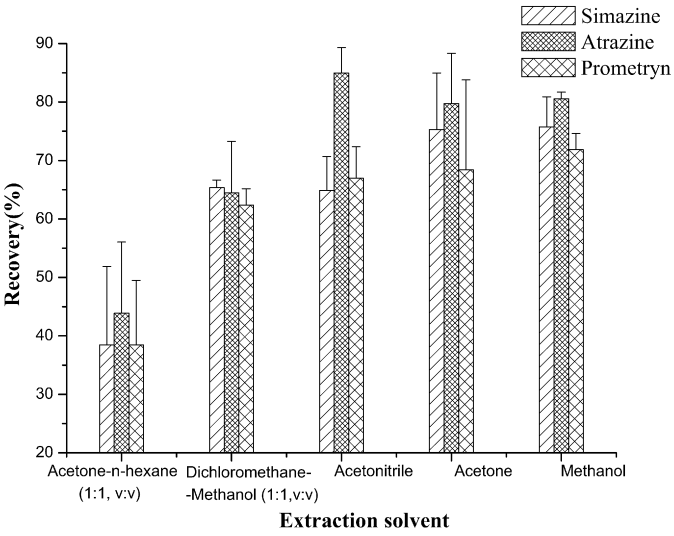

Fig. 3. Influence of the extraction solvent of PMAE on the recoveries extraction temperature 85 °C, extraction time 10 min, solvent volume 10 mL.

Therefore, 20 mL methanol was used as the extraction solvent for the further experiments.

3.1.2. Extraction temperature

Temperature is very important to ensure an efficient extraction and Fig. 5 shows recoveries obtained at different temperatures (65–135 °C). Simazine and atrazine have the highest extraction yields at 105 °C and prometryn has the highest yield at 95 °C. Too high temperature was not useful to increase the extraction yield and may result in the degradation of the analyte. Thus, 105 °C was selected as extraction temperature in the further experiments of PMAE.

3.1.3. Extraction time

The effect of extraction time was examined by extracting the spiked samples for 1–20 min with the other parameters kept con-

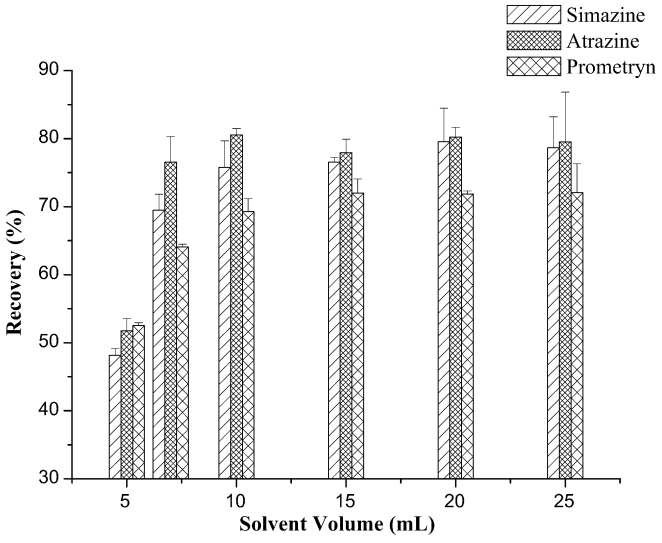

Fig. 4. Influence of the volume of extraction solvent of PMAE on the recoveries extraction temperature 85 °C, extraction time 10 min.

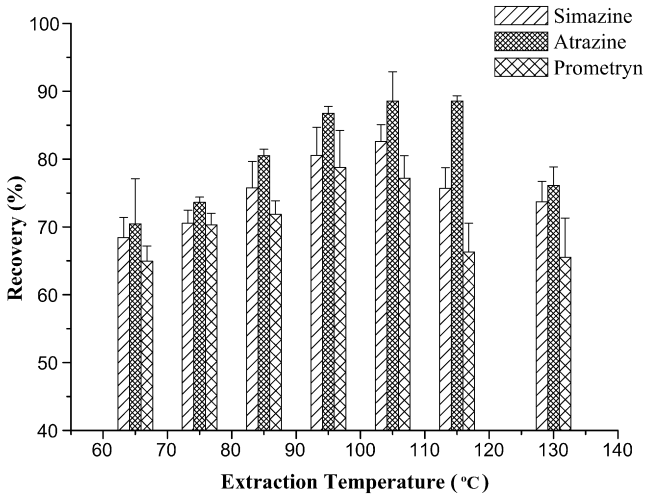

Fig. 5. Influence of extraction temperature of PMAE on the recoveries extraction time 10 min, methanol volume 20 mL.

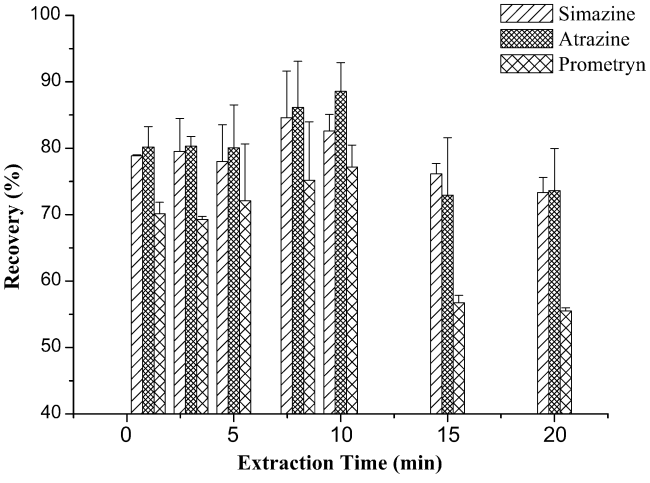

Fig. 6. Influence of the radiation time of PMAE on the recoveries extraction temperature 105 °C, methanol volume 20 mL.

stant. As shown in Fig. 6, the highest recoveries were obtained in the time ranging from 8 to 10 min, while low recoveries were obtained when the extraction time is shorter than 8 min and longer than 10 min. On the one hand, the low recoveries at short irradiation time might result from insufficient microwave energy, which can be available to attain the temperature of phase change and hence enable the breaking of the analyte–matrix bonds or might result from the strong adsorption of the analytes on the sample particle surface. On the other hand, according to the literature, long extraction time can cause degradation of the thermolabile compounds [17]. Although triazines are not the labile compounds at high temperatures, the results in Figs. 5 and 6 show that degradation of the analytes probably can take place to some extent under MAE conditions.

3.2. Evaluation of the method

3.2.1. Standard curve and limit of quantification

As shown in Table 1, standard curves based on matrix-matched standards from 1000 to 2 μg kg^{-1} were constructed and used to determine the three kinds of triazines. The limits of quantification (LOQs) of the proposed procedures, indicated in Table 1, were determined as the lowest pesticide concentration injected that yielded a signal-to-noise (S/N) ratio of 10. The LOQs are better than the results described in the literature [18] for determination of triazines in cereals, celery and apples (limits of detection, 20–1000 μg kg^{-1}) and similar to the ones found by Aramendía et al. [19] for determination of triazines in olive oil. The observed differences can be a result of different equipment used, different method of separation and detection as well as different sample matrix.

3.2.2. Analysis of spiked samples

In this part, validation of the proposed method for spiked samples was studied. Firstly, the proposed method was applied to fresh spiked samples at different concentrations. The results summarized in Table 2 indicate that the present method provides good recoveries (71.9–88.56%) and acceptable precisions (≤10.63%) for triazines in the range of 50–500 μg/kg. It can also be seen that relatively poor R.S.D. values and low recoveries were given by the samples spiked at 10 and 5 μg/kg. Secondly, the proposed method was applied to aged spiked samples. The results given in Table 2 indicate that, compared with fresh spiked samples, when the aged spiked samples were analysed, low recoveries are obtained and the results still satisfactory for the quantitative analysis of practical samples. The low recovery may be related to strong adsorption for the analytes by the cereal active sites for a long duration or to the degradation during this period. On the contrary, Molins et al. [20] reported that the recovery of the analytes triazines in aged spiked soil sample was similar to that of the analytes in fresh samples. In their study, MAE was applied to samples and dichloromethane–methanol was used as extractant. Helling et al. [21] reported that only about 2% losses were estimated for atrazine and simazine under real environmental conditions for 20 days. To decide which fac-

Table 1
Calibration data

Determination method	Compound	Slope	Intercept	r^2	LOQ (μg kg^{-1})
HPLC–DAD	Simazine	4.66×10^{-1}	0.09×10^{2}	0.9999	47.1
	Atrazine	3.62×10^{-1}	-0.49×10^{2}	0.9998	48.7
	Prometryn	4.20×10^{-1}	-0.02×10^{2}	0.9999	39.1
HPLC–ESI/MS	Simazine	5.24×10^{3}	7.85×10^{2}	0.9999	2.0
	Atrazine	1.03×10^{4}	3.28×10^{3}	0.9999	1.3
	Prometryn	3.48×10^{4}	8.57×10^{3}	0.9999	0.9

Table 2
Recoveries and R.S.D.s of fresh and aged samples spiked at different concentration levels ($n = 3$)

Sample	Concentration ($\mu g\,kg^{-1}$)	Simazine		Atrazine		Prometryn	
		Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)
Fresh	500	82.6	3.03	88.6	4.87	77.2	4.31
	100	76.3	5.31	83.7	7.61	71.9	10.63
	50	78.1	3.61	80.7	3.35	78.0	10.05
	10	66.9	12.62	81.9	4.49	71.1	11.58
	5	66.2	10.30	79.9	8.28	68.5	9.07
Aged	100	61.3	9.22	68.1	8.81	56.8	1.47
	50	51.1	1.49	64.0	7.19	59.5	5.40

Table 3
Analytical results for aged samples obtained by proposed method at different conditions of PMAE

Extraction conditions	Simazine		Atrazine		Prometryn	
	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)
105 °C, 10 min	61.3	9.22	68.1	8.81	56.8	1.47
115 °C, 10 min	52.5	2.64	60.2	2.23	57.1	3.69
105 °C, 12 min	42.0	7.85	46.9	6.67	52.0	6.64
105 °C, 15 min	36.9	7.56	44.3	5.59	47.8	9.91

Table 4
Analytical results for different infant cereal-based foods ($n = 3$)

Sample (500 $\mu g\,kg^{-1}$)	Simazine		Atrazine		Prometryn	
	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)
Sample 1	86.1	8.21	75.1	7.92	77.0	5.88
Sample 2	78.4	8.05	70.2	7.67	74.5	6.88
Sample 3	82.6	3.03	88.6	4.87	77.2	4.31
Sample 4	70.2	4.51	87.1	8.38	72.7	5.70
Sample 5	82.1	5.51	89.2	6.35	73.0	1.59
Sample 6	70.7	5.89	60.8	6.06	73.7	1.24

tor was important, the proposed method was then applied to the aged sample (100 μg/kg) at 105 and 115 °C. The results in Table 3 indicate that the highest recoveries were still obtained at 105 °C. In the further experiment, when extraction temperature is 105 °C and the extraction time is 10, 15 and 20 min, the recoveries cannot be improved. Because the recoveries cannot be improved at longer extraction time or higher extraction temperature, degradation of aged samples may play a key role.

3.2.3. Repeatability of the method

The repeatability of the method was estimated for determination of triazines in six kinds of samples. The detailed data are represented in Table 4, and from the table, we can see that good repeatability was obtained for all six kinds of cereals except for Sample 5.

3.2.4. Comparison of PMAE, APMAE, UE and SE

In order to evaluate the proposed method, other extraction methods were also applied. Firstly, the recoveries obtained by PMAE, AMAE, UE and SE were compared. All experiments for the comparison were performed with Sample 3. Table 5 shows the results obtained by four extraction methods. The recoveries of triazines obtained by the four methods were significantly different. The recoveries obtained by PMAE are higher than

Table 5
Analytical results of samples spiked at 100 $\mu g\,kg^{-1}$ obtained by PMAE, AMAE, SE and UE ($n = 3$)

Method	Simazine		Atrazine		Prometryn	
	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)	Recovery (%)	R.S.D. (%)
PMAE	76.3	5.31	83.7	7.61	71.9	10.63
AMAE	76.7	6.89	71.5	12.9	80.6	9.02
SE	77.4	3.83	38.9	9.40	42.3	4.89
UE	65.5	4.76	74.2	3.33	64.5	5.59

those obtained by SE and UE. The results obtained by PMAE and AMAE are similar. Compared with the other three methods, especially SE and UE, it can be concluded that PMAE is a labour-saving and time-saving procedure. All the experiment results indicate that PMAE is suitable for extracting triazines from nutrient cereal-based foods.

4. Conclusions

A method was developed to isolate and determine three triazines in some representative infant cereal-based foods. The results indicated that the proposed method has more advantages in respect of efficiency, extraction time and labour consumption compared with AMAE, SE and UE. Although the precision was not too high, it was still satisfactory. The results suggested that degradation may take place in period of sample stocking (45 days). The proposed method, in which the PMAE is coupled with HPLC–ESI/MS, can be sensitive and selective. So it seems possible to extend this method to the extraction and determination of triazines in other infant foods samples by varying the extraction conditions.

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