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Hindawi
Journal of Food Quality
Volume 2018, Article ID 7926768, 7 pages
https://doi.org/10.1155/2018/7926768

Research Article

Detection of Cyanuric Acid and Melamine in Infant Formula Powders by Mid-FTIR Spectroscopy and Multivariate Analysis

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Received 29 June 2017; Revised 9 January 2018; Accepted 19 February 2018; Published 22 March 2018

Academic Editor: Marina Carcea

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Chemometric methods using mid-FTIR spectroscopy were developed in order to reduce the time of study of melamine and cyanuric acid in infant formulas. Chemometric models were constructed using the algorithms Partial Least Squares (PLS1, PLS2) and Principal Component Regression (PCR) in order to correlate the IR signal with the levels of melamine or cyanuric acid in the infant formula samples. Results showed that the best correlations were obtained using PLS1 (R2: 0.9998, SEC: 0.0793, and SEP: 0.5545 for melamine and R2: 0.9997, SEC: 0.1074, and SEP: 0.5021 for cyanuric acid). Also, the SIMCA model was studied to distinguish between adulterated formulas and nonadulterated samples, giving optimum discrimination and good interclass distances between samples. Results showed that chemometric models demonstrated a good predictive ability of melamine and cyanuric acid concentrations in infant formulas, showing that this is a rapid and accurate technique to be used in the identification and quantification of these adulterants in infant formulas.

1. Introduction

Many foods have the potential for being adulterated naturally or deliberately. Food safety concerns have become a major concern due to a string of incidents involving food poisoning [1].

Melamine (2,4,6-triamino-1,3,5-triazine) is used for the production of multipurpose melamine-formaldehyde resins and has high nitrogen content (≈66% by mass). Consequently, melamine has been dishonestly added to dairy products (such as milk (liquid or powder) and infant formula) by dishonest milk producers to gain an incorrectly higher readout of apparent protein content than that determined by the conventional standard Kjeldahl test [2]. This action has been a cause of severe illnesses and numerous infants have been intoxicated because the addition of melamine into food products can cause death [3]. Besides melamine, its structural analogue, cyanuric acid (2,4,6-trihydroxy-1,3,5-triazine), was also found to increase apparent protein content in milk products and it has been added purposely to give high protein content [4, 5].

The problems related to melamine and cyanuric acid contamination have pointed out the need for rapid and reliable techniques capable of detecting these analytes. To analyze the banned use of melamine and cyanuric acid, various analytical methods have been developed. The techniques are liquid chromatography [6–8], liquid chromatography-mass spectroscopy (LC-MS-MS) [9], gas chromatography (GC) with mass spectroscopy (MS), photodiode detector (DAD), ultraviolet (UV), enzyme-linked immunosorbent assay (ELISA) [10], and capillary zone electrophoresis [11]. These methods are costly and demanding.

Midinfrared spectroscopy (mid-FTIR) is a rapid, reliable, and user-friendly technique, which requires insignificant or no sample preparation and reduces the use of expensive reagents. Coupled with multivariate analysis, mid-FTIR has been useful for adulterant detection in various food products. Midinfrared (mid-FTIR) [12, 13] and near-infrared (NIR-FTIR) spectroscopy have been used to quantify melamine in milk and infant formula powders [13–15]; however, mid-FTIR coupled with multivariate analysis has not been used to quantify cyanuric acid in infant formula powders.

The objective of this work is to evaluate the ability of mid-FTIR combined with chemometrics to detect and quantify melamine and cyanuric acid in infant formula powders. Notably, the Soft Independent Modeling of Class Analogy (SIMCA) has never been used to discriminate melamine and cyanuric acid in infant formula powders from vibrational spectral data.

2. Materials and Methods

2.1. Reagents. Melamine (99%) and cyanuric acid (98%) were purchased from Sigma-Aldrich (St. Louis, Missouri, USA). All chemicals and reagents used were of analytical grade.

2.2. Samples. Infant formula powder samples were acquired from marketplaces in Mexico City. The quality grade of all the samples was guaranteed by the sellers. However, to identify melamine in the samples, they were analyzed by immunosorbent assay (ELISA).

2.3. Immunosorbent Assay (ELISA). The melamine test kit (Microtiter Plate Abraxis®, Abraxis, Warminster, Pennsylvania, USA) was used. The test is based on the recognition of melamine by antibodies. The calibrators, sample extracts, and melamine conjugate are pipetted into test wells coated with melamine antibody to initiate the reaction. During the 30-minute incubation period, melamine from the sample and melamine conjugate compete for binding to melamine antibody. Following this 30-minute incubation period, the contents of the well are removed and the wells are washed to remove any unbound melamine and melamine conjugate. After washing with the diluted wash solution, a clear substrate is then added to the wells and any bound enzyme conjugate causes the conversion to a blue color. Following 20-minute incubation, the reaction is stopped and the amount of color in each well is read using an ELISA reader. The color of the unknown samples is compared to the color of the calibrators and the melamine concentration of the samples is derived. The concentrations of the samples are determined by interpolation using the standard curve constructed with each run.

2.4. Adulterated Samples. Infant formula powder was spiked with solutions containing different concentrations of the corresponding adulterant (melamine or cyanuric acid). Forty samples of infant formula powder with melamine or cyanuric acid in concentrations ranging from 0.5 μg/L to 20 μg/L with increments of 0.5 μg/L were prepared. Thirty-five of these samples were selected for the calibration set and five samples were chosen for the validation set.

2.5. Mid-FTIR Spectra Acquisition. The mid-FTIR spectra were obtained with a PerkinElmer Spectrum GX spectrophotometer (Norfolk, CT, USA) equipped with a deuterated triglycine sulphate detector. The spectra were obtained using an ATR accessory (Pike Technologies, Madison, WI, USA) with a crystal ZnSe. The spectra were scanned in a wavenumber interval of 4000–650 cm^{-1}, 64 scans, and a resolution of 4 cm^{-1}. Spectra were managed with the Spectrum software version 3.01.00 (PerkinElmer, Inc.) and acquired in triplicate.

2.6. Multivariate Analysis

2.6.1. Quantitative Multivariate Analysis. The algorithms Principal Component Regression (PCR) and Partial Least Squares (PLS, two versions: PLS1 and PLS2) were used. The software Spectrum Quant+ version 4.51.02 (PerkinElmer, Inc.) was used.

Various pretreatments were performed on the spectral data. The pretreatments were Savitzky-Golay filter (9 points), first derivative (Savitzky-Golay method), and Standard Normal Variate (SNV).

The spectral area designated for building the models covered 3600 to 2800 and 1750 to 650 cm^{-1} because this range showed the highest association between the spectral data and sample concentrations. R^2 and SEC (Standard Error of Calibration) were used to select the best model.

Five samples were used for the validation model. The best model was selected according to the higher R^2 and SEP (Standard Error of Prediction).

2.6.2. Identification by SIMCA. The model SIMCA was developed with the software AssureID version 4.0.2.0175 (PerkinElmer, Inc.). The classes were infant formula powder, infant formula with melamine, and infant formula with cyanuric acid. The SIMCA model was modeled with 35 spectra of each class (infant formula, infant formula with melamine, and infant formula with cyanuric acid). 105 mid-FTIR spectra were used. Five samples of each class were used for the validation model. The best model was selected according to the interclass distance (≥3) and the scores plot of principal components.

Various pretreatments were performed on the spectral data: ambient filters, Savitzky-Golay filter (13 points), derivative, Standard Normal Variate (SNV), and Detrending (DT).

3. Results and Discussion

3.1. Quantification of Melamine by ELISA. R^2 = 0.9958 was obtained on the calibration curve for the ELISA determinations. In the infant formula samples, no melamine residues were quantified, so these samples were used to construct the prediction models.

3.2. Mid-FTIR Spectra. Figure 1 illustrates the mid-FTIR spectrum of pure infant formula powder with the corresponding bands. An N-H stretching band from 3500 to 3400 cm^{-1} corresponds to the primary amino acids, the region at 3400–3200 cm^{-1} is due to O-H stretching modes of water, and that at 3000–2800 cm^{-1} is due to milk's fatty

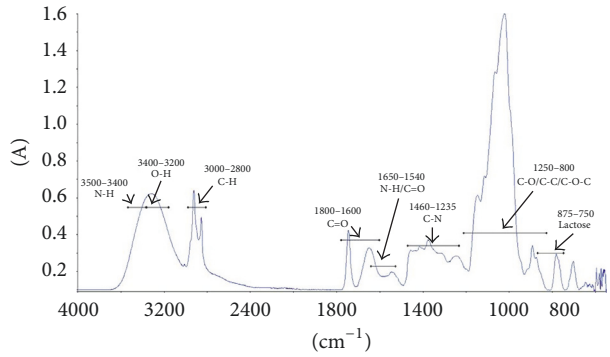

Figure 1: Mid-FTIR spectrum of infant formula powder.

acid CH_2 stretching. The peak at 1800–1600 cm^{-1} is due to triglyceride functional groups (C=O stretching). The peak at 1650 cm^{-1} is due to amide I and C=O stretching; the band at 1540 cm^{-1} corresponds to N-H bending with C-N stretching. At 1460–1235 cm^{-1}, it corresponds to C-N stretch vibrations (amides I, II). The mono- and polysaccharides dominate the region at 1250–800 cm^{-1} (C-O-C stretch related to sugar as lactose) [16, 17].

3.3. Spectra of Infant Formula Powder Adulterated with Melamine and Cyanuric Acid. Figures 2(a) and 2(b) show the spectra from samples with melamine and cyanuric acid. The changes in the bands are observed in Figures 2(a) and 2(b) which depicted spectra of infant formula powder with different levels of melamine and cyanuric acid (0.5–20 μg/L). A spectrum for a sample with a higher contaminant (in this case, melamine and cyanuric acid) content has higher absorbance. Hence, the spectra of Figures 2(a) and 2(b) show changes in intensity across mid-FTIR due to the modification in concentration of the functional groups.

3.4. Calibration Models. The spectra for each calibration set containing infant formula powder adulterated with melamine and cyanuric acid and the corresponding concentration data were subjected to PCR and PLS regression. Table 1 shows the number of factors, R^2 (coefficient of determination), and SEC (Standard Error of Calibration) for the quantitative models used to predict melamine and cyanuric acid. The number of factors represents the new variables from which the useful information was extracted from the infrared spectrum. There are two types of risk: the infra-adjustment that indicates that the model does not have enough information to predict samples and the overadjustment that indicates that the model has a lot of information that is not useful. To avoid these risks, the factors were designated based on low SEC and SEP [18].

The R^2 values developed with the PLS1 to predict melamine and cyanuric acid concentrations in infant formula powder exceeded 0.99 (0.998 and 0.997 for melamine and cyanuric acid, resp.) (Table 1). The models show a lower relationship when using the PCR for the set of samples contaminated with melamine and cyanuric acid (Table 1). R^2 evaluates the data set. R^2 is based on the percentage of variability (concentration of melamine and cyanuric acid) that can be explained by a certain set of variables. Therefore, $R^2 = 1$ means perfect fit (concentration of melamine and cyanuric acid is fully explained by the regression model). Otherwise, $R^2 = 0$ indicates that the model does not explain the variation of the concentration of melamine and cyanuric acid.

The SEC values evaluate the goodness of fit of the regression during calibration. The SEC fluctuated between 0.0793 and 0.1074 for the PLS1 model, between 2.8840 and 3.1960 for the PCR model, and between 0.1496 and 0.3459 for the PLS2 model. The PLS1 model has lower SEC values and higher R^2 than the PCR and PLS2 models did, indicating that the PLS1 model had better calibration power than the PCR and PLS2 models. Thus, the best calibration models were obtained with the PLS1 algorithm (Table 1).

Figures 3(a) and 3(b) show the graphs demonstrating the relationships between the values presented in the samples and the values expected by the models found with PLS1 (melamine and cyanuric acid, resp.). Figures 3(a) and 3(b) show that R^2 is close to 1 for both adulterants, confirming that the prediction of adulterants is completely explained by the model. Also, this indicates a high correlation between the real and predicted values; this indicates good predictive capacity of the developed models.

The PLS1 algorithm was used for the validation set (ten external samples: five of each contaminant). Table 1 shows R^2 and SEP between the predicted and the actual values of the validation set; these statistical data were chosen to evaluate the models. R^2 exceeded 0.99 for PLS1 models (melamine, $R^2 = 0.996$; cyanuric acid, $R^2 = 0.997$) (Table 1). According to Tamaki and Mazza [19], R^2 exceeding 0.90 is considered an excellent prediction. Also, the SEP ranged from 0.5021 to 0.5545 (Table 1). SEP indicates the prediction error of the model. SEP presented a low value which indicates good future predictions. R^2 and low SEC indicate appropriate regression models [20].

Figures 4(a) and 4(b) show predicted values by models against actual values of melamine and cyanuric acid, respectively, in the validation set; R^2 was 0.996 and 0.997 for melamine and cyanuric acid, respectively. According to data from Figures 4(a) and 4(b), good correlations were obtained for melamine and cyanuric acid values showing good predictive ability.

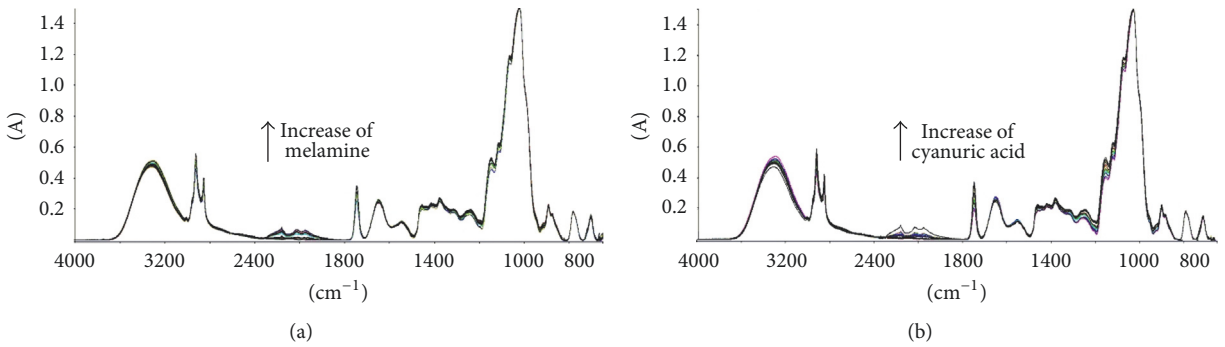

Figure 2: Mid-FTIR spectra of infant formula powder with different concentrations of (a) melamine (0.5–20 μg/L) and (b) cyanuric acid (0.5–20 μg/L).

Table 1: Calibration and validation data of the models developed with PCR, PLS1, and PLS2 algorithms to predict melamine and cyanuric acid concentration in infant formula powder.

Calibration set*	Algorithm	Calibration (n = 35)			Validation (n = 5)	
		Number of factors[a]	R^{2}[b]	SEC[c]	R^{2}[b]	SEP[d]
Infant formula-melamine	PCR	9	0.9401	2.8840	–	—
	PLS1	***10***	***0.9998***	***0.0793***	0.9996	0.5545
	PLS2	14	0.9969	0.3459	–	—
Infant formula-cyanuric acid	PCR	12	0.7960	3.1960	—	—
	PLS1	***6***	***0.9997***	***0.1074***	0.9997	0.5021
	PLS2	14	0.9993	0.1496	–	—

*Calibration set was prepared ranging from 0.5 to 20 μg/L. [a]Optimal number of factors. [b]Coefficient of determination (R^2) should be as close to 1 as possible. [c]Standard Error of Calibration (SEC) should be as low as possible. [d]Standard Error of Prediction (SEP) should be as low as possible. *Note*. The best model is shown in italics and bold type.

Table 2 illustrates the validation set of the PLS1 algorithm. Table 2 confirms the good performance of each developed model. Both models (melamine and cyanuric acid) have good predictive capacity, since the concentrations predicted by both models are very close to the actual concentration of the adulterants in the validation samples. In both models, Mahalanobis distance was less than 1, indicating the similarity of spectral characteristics between the calibration samples and the external validation samples, and the residual error is below the allowed limit (3), indicating that the external samples of the validation are modeled correctly by the developed models.

According to the results, models created with PLS1 have a good predictive capacity to predict melamine and cyanuric acid in unknown samples.

3.4.1. Detection Limit of Melamine and Cyanuric Acid in Infant Formula. To determine the detection limit (LOD) of the chemometric models developed, three samples of infant formula powder containing 0.5 μg/L, 0.4 μg/L, and 0.3 μg/L of melamine or cyanuric acid were prepared. The PLS1 models for melamine and cyanuric acid were used to quantify the amount of melamine and cyanuric acid in the samples. Table 3 shows the detection limits of melamine and cyanuric acid in infant formula by PLS1 models. Table 3 shows that infant formula samples containing less than 0.5 μg/L (0.4 μg/L and 0.3 μg/L) have highly statistical parameters (italics and bold type in Table 3) (Mahalanobis distance: 1.49–1.15 for melamine and 1.15–1.11 for cyanuric acid, resp.) and residual error (1.99–2.74 for melamine and 1.81–2.94 for cyanuric acid, resp.). Results show that the LOD of the calibration models to predict melamine and cyanuric acid concentrations in infant formula is above 0.5 μg/L (LOD > 0.5 μg/L) (Table 3). The LOD of the PLS1 models are acceptable because they are below the MRLs established by the FDA (1 mg/kg) [21].

3.5. Identification by SIMCA. Figure 5 shows the three-dimensional projection of the classes from SIMCA (infant formula, infant formula-melamine, and infant formula-cyanuric acid) obtained with three PCs. Figure 5 shows excellent cluster separation which indicates the correct separation of classes, without overlapping classes. Table 4 shows the distances between classes (infant formula, infant formula-melamine, and infant formula-cyanuric acid). The interclass distances between the classes (infant formula and infant formula-melamine; infant formula and infant formula-cyanuric acid; infant formula-melamine and infant formula-cyanuric acid) are 14.8, 13.4, and 3.2, respectively. Distances above 3 are considered appropriate for good separation between classes [22].

The prediction of the SIMCA was analyzed by external validation samples (five additional spectra of each class)

Table 2: External validation data of the PLS1 chemometric models.

Validation set (n = 5)	Actual value (μg/L)	Predicted value (μg/L)	Mahalanobis distance[a]	Residual error[b]
Infant formula-melamine	3.0	3.02	0.22	1.22
	7.0	6.93	0.28	1.47
	7.5	7.42	0.08	1.35
	11.5	11.68	0.27	0.99
	16.0	16.05	0.23	1.08
Infant formula-cyanuric acid	2.0	2.00	0.25	1.10
	5.0	5.04	0.30	1.17
	10.0	10.02	0.03	1.07
	10.5	10.31	0.02	0.99
	14.5	14.46	0.08	1.39

[a]Mahalanobis distance should be as low as possible, not to exceed 1. [b]Residual error should be as low as possible, not to exceed 3.

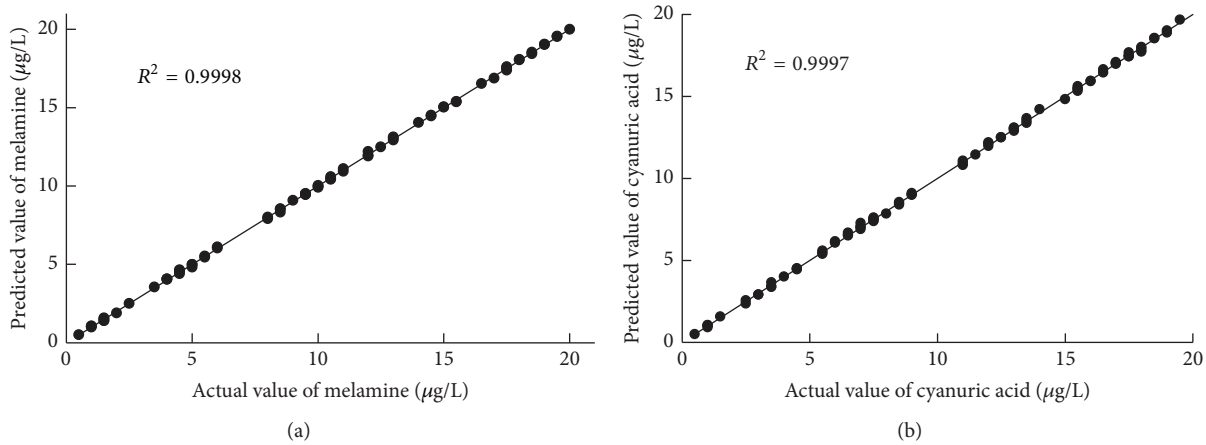

Figure 3: Plots of predicted values versus actual values of infant formula powder with (a) melamine and (b) cyanuric acid of *calibration* samples determined by the PLS1 algorithm.

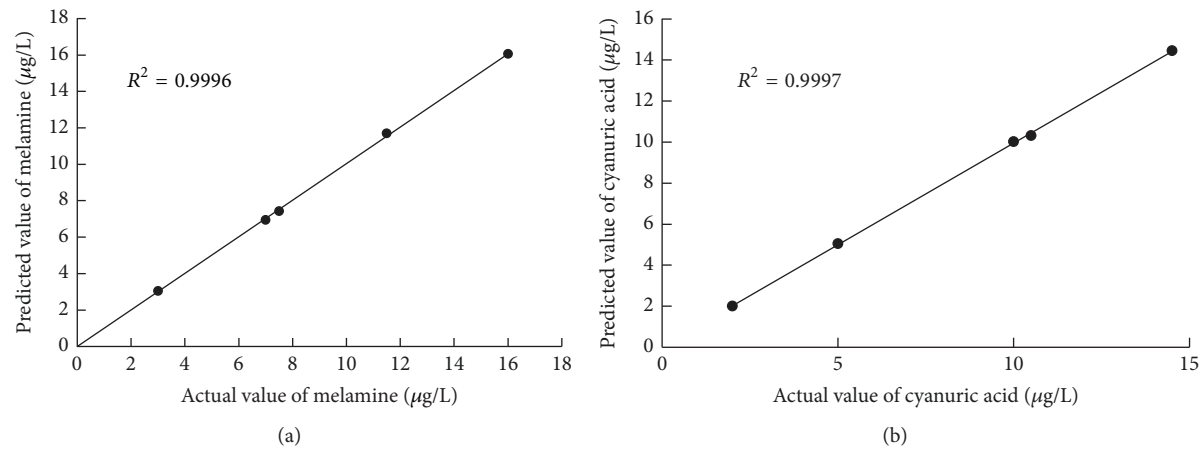

Figure 4: Plots of predicted values versus actual values of infant formula powder with (a) melamine and (b) cyanuric acid in the *external validation* determined by the PLS1 algorithm.

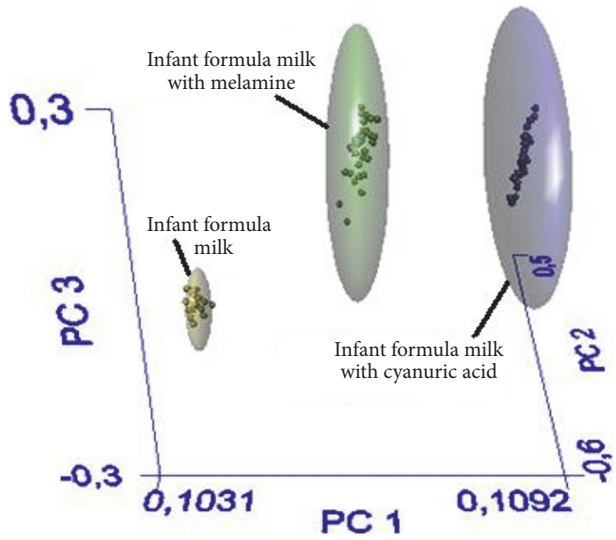

Figure 5: Three-dimensional principal component analysis scores plot of the populations derived from SIMCA: infant formula powder, infant formula powder with melamine, and infant formula powder with cyanuric acid.

Table 3: Detection limits of melamine and cyanuric acid in infant formula powder by PLS1 models.

Set (n = 3)	Actual value (μg/L)	Predicted value (μg/L)	Mahalanobis distance[a]	Residual error[b]
Infant formula-melamine	0.5	0.50	0.42	1.24
	0.4	***11.41***	***3.15***	***5.99***
	0.3	***18.10***	***4.49***	***6.74***
Infant formula-cyanuric acid	0.5	0.50	0.58	0.85
	0.4	***8.28***	***2.15***	***4.81***
	0.3	***6.66***	***4.11***	***3.94***

[a]Mahalanobis distance should be as low as possible, not to exceed 1. [b]Residual error should be as low as possible, not to exceed 3.

Table 4: Interclass distances of the different populations derived from SIMCA.

Class	Infant formula	Infant formula-melamine	Infant formula-cyanuric acid
Infant formula	0	14.8	13.4
Infant formula-melamine		0	3.2
Infant formula-cyanuric acid			0

Note. Interclass distances should be as high as possible, minimum 3.

Table 5: External validation data of the SIMCA model.

Samples	Specified material[a]	Identified material[b]	Result[c]	Total distance ratio[d]	Residual distance[e]
1–5	Infant formula	Infant formula	Identified	0.42–0.92	0.63–1.38
1–5	Infant formula-melamine	Infant formula-melamine	Identified	0.68–0.90	0.91–1.20
1–5	Infant formula-cyanuric acid	Infant formula-cyanuric acid	Identified	0.67–0.86	0.89–1.14

[a]Specified material, the material indicated during validation. [b]Identified material, the material identified during validation. [c]Result, the material identified or not as the specified material. [d]Total distance ratio must be less than 1.0 for a sample to be classified. [e]Residual distance should be as low as possible.

that had not been used at any time to generate the model. The 15 samples to validate the SIMCA model were correctly identified and classified. Statistical parameters were also good (Table 5). Based on the results, the SIMCA model can identify and classify the samples unadulterated and adulterated with melamine and cyanuric acid (99% confidence limit).

4. Conclusion

The results obtained in this work demonstrate a very good ability of MIR spectroscopy to predict melamine and cyanuric acid concentrations in infant formula powder in a low concentration (LOD > 0.5 μg/L). The results show the

possibility of using this analysis to monitor the banned use of melamine and cyanuric acid and confirm the safety of the infant formula powder quickly and reliably.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

Financial support from the Instituto Politécnico Nacional de México (IPN) and the Consejo Nacional de Ciencia y Tecnología (CONACyT) is greatly appreciated.

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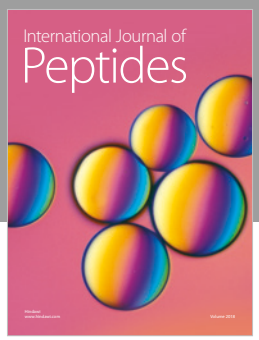

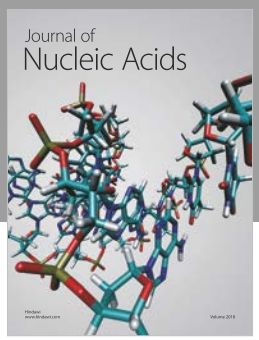

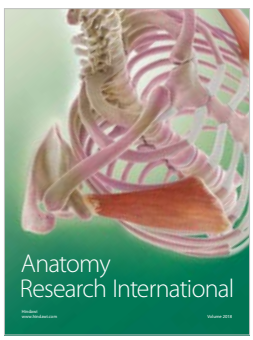

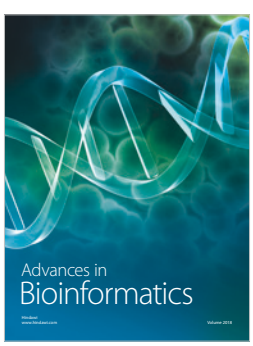

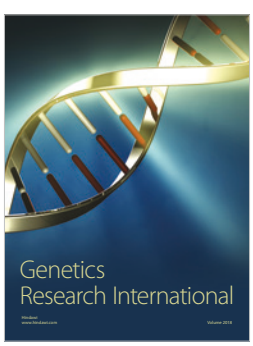

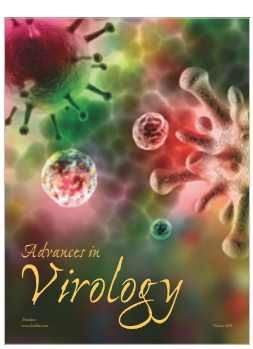

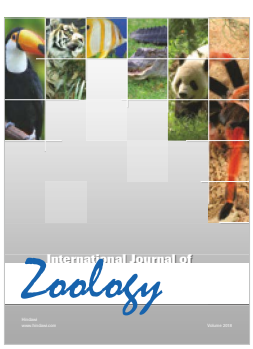

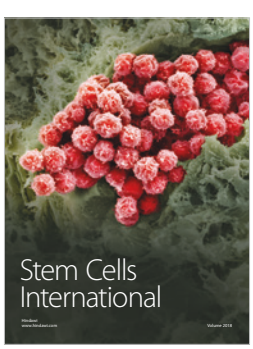

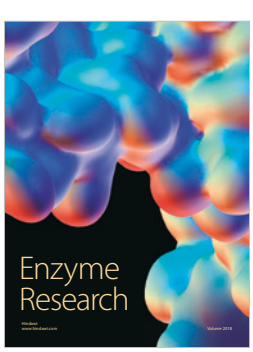

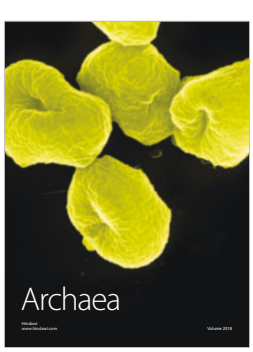


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