Determination of Four Metal Elements in Chicken Feed by Microwave Digestion-HR-CS FAAS

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Abstract. A new application of high resolution continuum source flame atomic absorption spectrometry has been developed for the determination of Ca, Fe, Zn and Mn in chicken feed. Under the selected determination conditions, the correlation coefficients better than 0.998, the relative standard deviations less than 2.6% and the recoveries between 96.3% and 103.1% were obtained for the four metal elements. The characteristic concentrations were 0.092 mg/L (Ca), 0.17 mg/L (Fe), 0.033 mg/L (Zn) and 0.15 mg/L (Mn), respectively. The results showed that the contents in chicken feed were 6.75±0.12 mg/g (Ca), 1.30±0.03 mg/g (Fe), 0.174±0.05 mg/g (Zn) and 0.313±0.04 mg/g (Mn), respectively. Therefore, the proposed method was accurate and stable with a high practical value. It provided scientific basis for determination of metal elements in food.

Introduction

Several sample preparation techniques are used for the determination of metal elements in food, such as inductively wet digestion, dry digestion, incomplete digestion and microwave digestion. Wet digestion and dry digestion are two traditional ways, but both of them have obvious disadvantages, for instance, some dense mass of strong acids are used in wet digestion, while dry digestion is a time-consuming process[1,2]. The objective of incomplete digestion is uniform and transparent digestive solutions,which do not need of complete destruction and colorless liquors. As a consequence of that, the sample-process time just need less than 20 min[3]. Nevertheless, incomplete digestion also requires a good deal of strong acids and the microemulsification stability after that is an important parameter for accurate analysis. Closed-vessel microwave-assisted digestion need only 5 mL of HNO_3 and 4 mL H_2O_2 in this study, and its main advantages are the high relative speed, good reproducibility, low blank, low possibility of contamination and the minimum loss of volatile elements[4].

The objective of this study was to develop a simple and robust method for the fast sequential multi-element determination of Ca[5], Fe[6], Zn[2] and Mn in chicken feed by high resolution continuum source flame atomic absorption spectrometry (HR-CS FAAS)[7-10] using new features.

Experimental

Instrumentation

An Analytik Jena ContrAA 700 High Resolution Continuum Source Atomic Absorption Spectrometer (Analytik Jena, Berlin,Germany) had been used for all measurements in this work. This spectrometer consists of a high-intensity xenon short-arc lamp, a high-resolution double echelle monochromator (DEMON) and a chargecoupled device (CCD) array detector[11]. All absorption lines in the range from 185 nm to 900 nm are provided by the high-intensity xenon short-arc lamp as the radiation source. The highest resolution of about 2 pm is carried out by DEMON, including a pre-dispersing prism monochromator and a high-resolution echelle grating monochromator. 200 pixels of the linear CCD array detector are used for monitoring all spectral informations on both sides of center wavelength. The flame type was C_2H_2-air and the burner type was 100 mm in the process of determination. The fixed air flow was 470 L/h for determination. The optimized determination conditions were shown in Table 1.

Table 1 Determination conditions of HR-CS FAAS

Element	Wavelength (nm)	Spectr.range	Fuel flow (L/h)	Burner height (mm)
Ca	422.6728	200	70	6
Fe	248.3270	200	80	5
Zn	213.8570	200	90	6
Mn	279.4817	200	100	6

Reagents and standards

The reagents were of guaranteed reagent. Ultrapure water with a resistivity of 18.2 MΩ·cm was obtained from a Milli-Q system (Millipore, Billerica, USA). Calibration solutions of Fe, Zn and Mn were prepared in the ultrapure water with 0.5% (v/v) HNO_3 and 0.1% (m/m) KCl by serial dilution of the stock solutions with 100 mg/L respectively, that of Ca were prepared in the ultrapure water with 0.5% (v/v) HNO_3, 1% (m/m) KCl and 0.5% (m/m) $La(NO_3)_3$ by serial dilution of the stock solutions with 100 mg/L (National Chemical Reagent Company, Beijing, China). All glasswares were previously soaked overnight in dilute HNO_3 (5% v/v) for cleaning and were rinsed with abundant ultrapure water prior to avoid contamination.

Microwave digestion

The chicken feed were purchased from a feed store where farmers bought grain for their animals (Xuzhou, China) during 2015 and analyzed of their Ca, Fe, Zn and Mn contents. They were crushed to powder. Approximately a 0.5 g of the crushed sample was preprocessed with 5 mL of HNO_3 and 2 mL H_2O_2 in the PTFE jar, which was the process of heating to 120 ℃ for 60 min in an air-ventilated oven. The compound (sample and acids) was digested in the intelligent microwave digestion system (Xin-tuo, Shanghai,China) after adding again 2 mL H_2O_2. A five stage program (Table 2) with a maximum pressure of 2.0 Mpa was chosen for achieving complete digestion of the crushed sample within the shorter time. The digestive liquor was diluted to 25 mL with the ultrapure water with 0.5% (v/v) HNO_3 and 0.1% (m/m) KCl when its volume was less than 3.0 mL for determination of Fe, Zn and Mn. A 1.00 mL of that sample solution was diluted to 25 mL with the ultrapure water with 0.5% (v/v) HNO_3, 1% (m/m) KCl and 0.5% (m/m) $La(NO_3)_3$ for determination of Ca. A blank digest was carried out in the same way. Three independent aboved treatments of the sample were performed for obtaining the average of repetitive determinations of Ca, Fe, Zn and Mn.

Table 2 Microwave digestion program

Stages	Pressure (Mpa)	Hold (min.)	Power (W)
1	0.2	60	500
2	0.5	60	1000
3	1.0	120	1000
4	1.5	120	1000
5	2.0	60	1000

Results and discussion

The analytical characteristic data of HR-CS FAAS were shown in Table 3. The calibration curves used to determine of Ca, Fe, Zn and Mn with HR-CS FAAS were built-up by measuring the absorbance of the calibration solutions in the selected determination conditions, as shown in Fig.1.. The correlation coefficients were 0.9992 (Ca), 0.9996 (Fe), 0.9989 (Zn) and 0.9989 (Mn), respectively. The characteristic concentrations were 0.092 mg/L (Ca), 0.17 mg/L (Fe), 0.033 mg/L (Zn) and 0.15 mg/L (Mn), respectively. The relative standard deviations were 2.3% (Ca), 1.9% (Fe), 2.6% (Zn) and 2.1% (Mn), respectively. As can be seen, the proposed method represented one of more sensitive and more reproducibility methodologies for determination of the four metal elements.

The proposed means, using microwave digestion-HR-CS FAAS, had been applied to determination of the four metal elements in chicken feed and was verified through spike recovery tests[12]. The results, obtained as the average of three replicates of each element, were shown in Table 4. The contents in chicken feed were 6.75±0.12 mg/g (Ca), 1.30±0.03 mg/g (Fe), 0.174±0.05

mg/g (Zn) and 0.313±0.04 mg/g (Mn), respectively. The recoveries were 103.1±1.9% (Ca), 98.4±2.3% (Fe), 96.3±2.9% (Zn) and 97.7±1.8% (Mn), respectively. Therefore, the proposed method represented one of more accurate methodologies for the determination of the four metal elements.

Table 3 Analytical characteristic data of HR-CS FAAS

Element	Calibration function (C in mg/L)	Correlation coefficient (R^2)	Characteristic concentration (mg/L)	Concentration range (mg/L)	Precision (RSD, n=6)
Ca	A=(0.0029893＋0.0472545×c) /(1＋0.0206515×c)	0.9992	0.092	0～10	2.3%
Fe	A=(－0.0013854＋0.0256013×c) /(1＋0.0194315×c)	0.9996	0.17	0～5	1.9%
Zn	A=(－0.0018482＋0.1311885×c) /(1＋0.1288429×c)	0.9989	0.033	0～4	2.6%
Mn	A=(0.0003392＋0.0285501×c) /(1＋0.0148045×c)	0.9989	0.15	0～5	2.1%

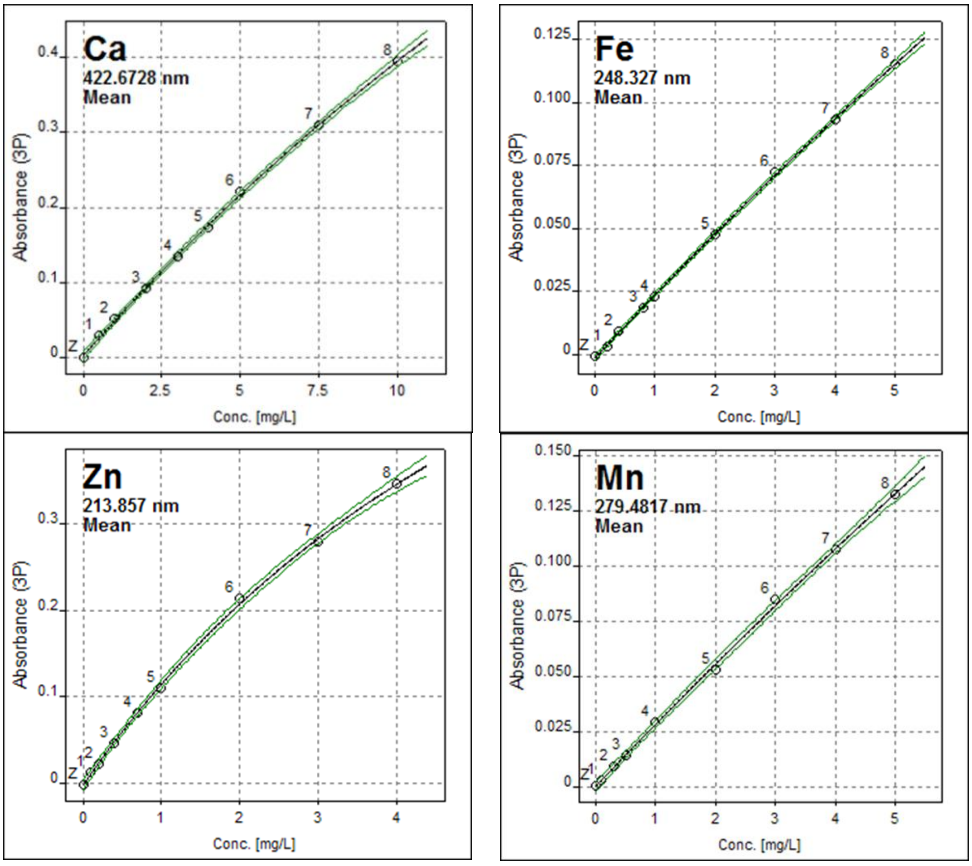

Fig.1. Calibration curve.

Table 4 Determination of Ca, Fe, Zn and Mn in chicken feed and recoveries (Avg.±SD of three trials)

Element	Content (mg/g)	Spiked (mg/g)	Recoveries (%)
Ca	6.75±0.12	5.0	103.1±1.9
Fe	1.30±0.03	2.0	98.4±2.3
Zn	0.174±0.05	0.2	96.3±2.9
Mn	0.313±0.04	0.5	97.7±1.8

Conclusions

The developed means provided a exact and sensitive procedure for the determination of Ca, Fe, Zn and Mn in chicken feed by HR-CS FAAS after microwave digestion. Under the selected determination conditions, the correlation coefficients better than 0.998, the relative standard deviations less than 2.6% and the recoveries between 96.3% and 103.1% were obtained for the four metal elements. The characteristic concentrations were 0.092 mg/L (Ca), 0.17 mg/L (Fe), 0.033 mg/L (Zn) and 0.15 mg/L (Mn), respectively. The results showed that the contents in chicken feed were 6.75±0.12 mg/g (Ca), 1.30±0.03 mg/g (Fe), 0.174±0.05 mg/g (Zn) and 0.313±0.04 mg/g (Mn), respectively. Low characteristic concentrations, good correlation coefficients and precisions, and high recoveries showed that the proposed method is accuracy, reliability and stability.

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