DETERMINATION OF IRON IN MILK POWDER BY MICROWAVE DIGESTION AND FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY

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Abstract: To investigate the conditions of microwave digestion for determining Iron in milk powder by flame atomic absorption spectrophotometry(FAAS), the content of iron in milk powder was determined by flame atomic absorption spectrophotometry after the samples were microwavely digested under different conditions. The optimum parameters for microwave digestion were obtained by the orthogonal test at last. The best optimum parameters for microwave digestion was that, the volume of digestion solution was 8mL, the reagent proportion for HNO3 and H2O2 was 4:1, the digestion time was 8min, the digestion pressure was 2.6 Mpa and the digestion power was 1000 W. The content of Iron in assayed milk powder was 0.0560mg/g. Microwave digestion was a time-saving and practical pretreatment of samples.

Key words: microwave digestion; flame atomic absorption spectrophotometry(FAAS); milk powder

1. INTRODUCTION

Due to its superior sensoury and nutritional properties, the milk powder has a considerable market potential and people pay more and more attention

to the nutritional properties of it such as the content of trace element. So, the determination of trace element in milk powder is more important. Flame atomic absorption spectrophotometry (FAAS) is a preferred method for the derermination of trace element (Wang et al., 2004). The advantages of FAAS are that it is highly sensitive and quick. The traditional dry ashing and acid dissolution standard methord for the samples of FAAS is time-consuming and carries risk of loss and contaminative. Microwave digestion with acid mixtures in closed teflon vessels avods these problems and ensures that the total mental content can be analysed(Li., 2007). Chen Wenting et al determined the metal elements in milk powder byultrasonic extraction(Chen et al., 2007). Zhang Limin and Zhou Xuemei et al determined the metal elements in milk powder by flame atomic absorption spectrophotometry after the samples were microwavely digested(Zhang., 2007; Zhou et al., 2005), but they did not investigate the optimum parameters for microwave digestion.

Iron is a essential trace element for people because it could inactivate the enzyme in people body. The objective of the present work was to investigate the optimum working condition for microwave digestion used in assay the amount of Iron in milk powder by FAAS, and this results also could be used in other trace element in milk powder by FAAS.

2. EXPERIMENTAL

2.1 Reagents and standard solutions

HNO_3 used was purified by quartz sub-boling distillation. Stock standard of Fe was prepared by dissolving 9.99%pure metal in HNO_3. Working standard was prepared daily by subsequent dilution.

2.2 Apparatus

AA140/240 atomic absorption spectrophotometry（VARIAN,USA）; MDS-200 microwave digestion equipment（CEM,USA）

2.3 Decomposition of sample by conventional heating

About 2.5 g of samples were taken in PFA containers and 15 mL HNO_3 was added. Once the initial reaction ceased, the screw cap was tightened and the sample was digested on the hot plate for 1 h at 80-90℃. The sample solution was evaporated near to dryness and made up to 50 mL using ultrapure water. The time required was about 3 h.

2.4 Decomposition of sample by microwave digestion

About 2.5 g of samples were taken in PFA containers and digestion solution (contained HNO_3 and H_2O_2) was added. The sample was pre-digested on the hot plate at 100℃ some time until white smoke rised. When the temperature of containers decreased, they were put into the digestion vessel and the microwave digestion was producted according to the prosedures designed before. After microwave digestion the sample solution was made up to 50 mL using ultrapure water.

2.5 Standard curve and samples measurements

Working standard was prepared daily by diluting stock standard of Fe to some solution levels of 0.020mg/L，0.040mg/L, 0.060mg/L, 0.080mg/Land 0.100 mg/L.

The absorption value of working standard solution were obtained according to the conditions shown in table 1 by FAAS and the standard curve equation (drawn automaticly by the computer) was

Abs=0.03734×C+0.00143(r^2=0.9995). Then, the samples and the blank were assayed in the conditions shown in table 1 by FAAS .

Table1. Instrument working conditions

Element	Wave/nm	Electricity mA	Slit nm	Highth of burning nm	Flow of acetylene $L.min^{-1}$	Flow of air $L.min^{-1}$
Iron	248.3	5.0	0.2	7.5	2.00	13.50

3. RESULT AND DISCUSSION

3.1 Effect of microwave digestion power on the determination of Iron in milk powder

When other factors were fixed, only the microwave digestion power was a variation to determine Iron in milk powder by flame atomic absorption spectrophotometry(FAAS) and the results were shown in fig1.

The content of Iron in milk powder increased as the microwave digestion power increased, and when the microwave digestion power was 1000W the content of Iron in milk powder was the most (Fig 1). So, the the microwave digestion power was fixed at 1000 W in the following experiments.

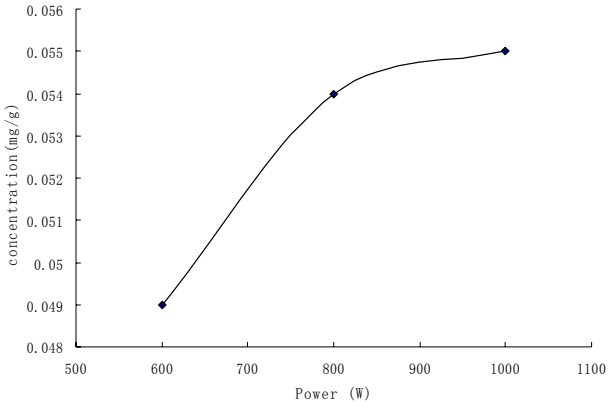

Fig 1 Effect of microwave digestion power on the determination of Iron in milk power

3.2 Effect of the reagent proportion on the determination of Iron in milk powder

When the reagent proportion(HNO_3:H_2O_2) was 4:1, the content of Iron in milk powder was 0.552 mg/g and was the most(Fig 2). So, a proportion of 4:1 was the best reagent proportion(HNO_3:H_2O_2).

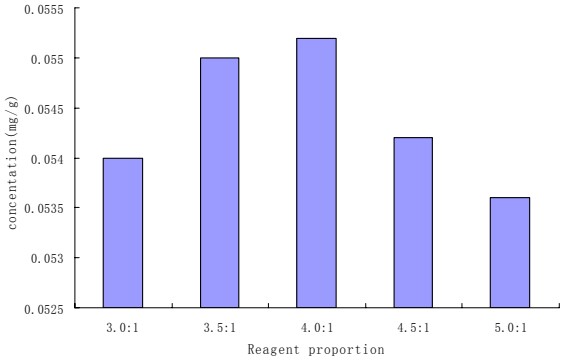

Fig 2Effect of reagent proportion on determination of Iron in milk power

3.3 Effect of the digestion pressure on the determination of Iron in milk powder

The content of Iron in milk powder increased as the microwave digestion pressure increased from 2.1 to 2.4 MPa, and when the microwave digestion

pressure was 2.4MPa the content of Iron in milk powder was the most(Fig 3). Then, the content of Iron in milk powder decreased as the microwave digestion pressure decreased. If the microwave digestion pressure was lower, not only decreased the digestion efficacy, but also cost more digestion time.

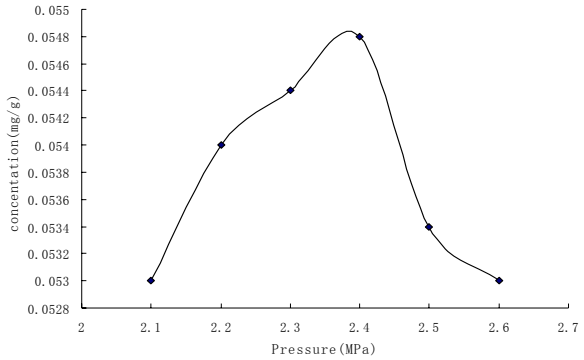

Fig 3 Effect of digestion pressure on the determination of Iron in milk power

3.4 Effect of the digestion time on the determination of Iron in milk powder

The content of Iron in milk powder increased as the microwave digestion time increased from 6 to 8 min, and when the microwave digestion time was 8 min the content of Iron in milk powder was the most and was0.0550mg/g (Fig 4). Then, the content of Iron in milk powder decreased as the microwave digestion time decreased. The microwave digestion could destroy the surface of the samples and this could provide more new surface of samples to touch with the HNO_3 to increase the speed of reaction. If the microwave digestion time was too long, the microwave digestion pressure would be decreased to low the the determination of Iron in milk powder.

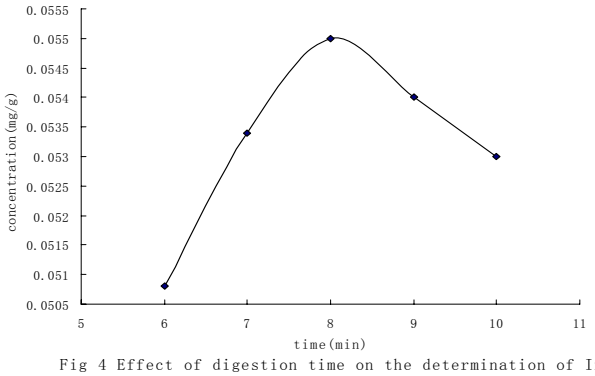

Fig 4 Effect of digestion time on the determination of Iron in milk power

3.5 Effect of the digestion solution volume on the determination of Iron in milk powder

The content of Iron in milk powder increased at first and then decreased when the volume of digestion solution increased from 8 to 12 mL. The content of Iron in milk powder was the most (0.0548mg/g, Fig 5) when the volume of digestion solution was 10 mL. The samples surface could not touch with the acid enough if the digestion solution was less, and more acid left after digestion would effect the accuracy of the dermination of Iron in milk powder. So, the digestion solution volume should be control at a optimum level.

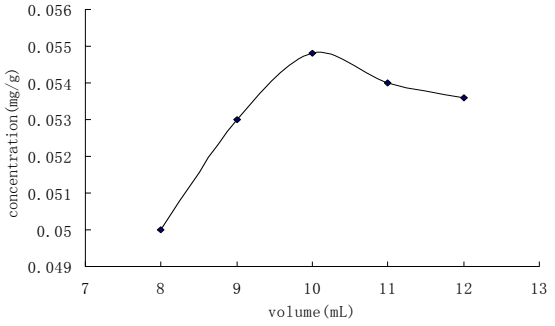

Fig 5 Effect of solution volume on the determination of Iron in milk power

3.6 Results of orthogonal test of microwave digestion

A orthogonal test for microwave digestion of four factors(time, solution volume, pressure and power) and three levels was designed according to the single factor experimenta above, and the results were shown in table 2.

Table 2 Results and analysis of orthogonal test of microwave digestion

Number	Factors				Concentration (mg/g)
	(A) Time (min)	(B) Solution volume (mL)	(C) Pressure(Mpa)	(D) Power(W)	
1	7	8	2.2	600	0.0476
2	7	10	2.4	800	0.0474
3	7	12	2.6	1000	0.0512
4	8	8	2.4	1000	0.0552
5	8	10	2.6	600	0.0504
6	8	12	2.2	800	0.0532
7	9	8	2.6	800	0.0550
8	9	10	2.2	1000	0.0540
9	9	12	2.4	600	0.0486
K1	0.1462	0.1578	0.1548	0.1466	
K2	0.1598	0.1518	0.1512	0.1556	
K3	0.1576	0.1530	0.1566	0.1604	
R	0.0126	0.0060	0.0054	0.0138	

The power was the most important and the pressure was the lest important for the microwave digestion of milk powder(Tbble 2). The optimum parameters for microwave digestion were $D_3A_2B_1C_3$ and that were, the volume of digestion solution was 8mL, the reagent proportion for HNO_3 and H_2O_2 was 4:1, the digestion time was 8min, the digestion pressure was 2.6 Mpa and the digestion power was 1000 W. The content of Iron in assayed milk powder was 0.0560mg/g according to these parameters for microwave digestion.

3.7 Comparison of decomposition of sample by microwave digestion and by conventional heating

For the same milk powder sample assayed by FAAS, the content of Iron was more in sample decomposed by microwave digestion than it by conventional heating. So, for FAAS, decomposition by microwave digestion was time-saving and more accurate than by by conventional heating.

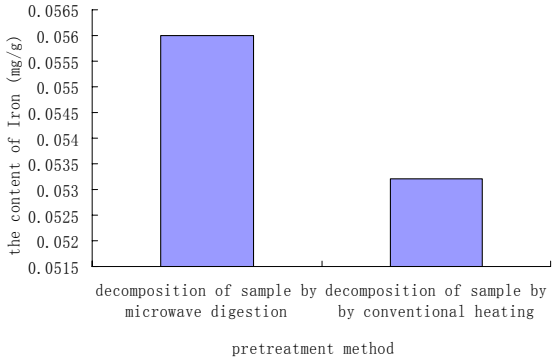

Fig 6 Comparision of the result by different pretreatment methods

4. CONCLUSION

The best optimum parameters for microwave digestion was that, the digestion power was 1000 W, the digestion time was 8min, the volume of digestion solution was 8mL and the digestion pressure was 2.6 Mpa. The content of Iron in assayed milk powder was 0.0560mg/g assayed by FAAS. Microwave digestion was a mordern ,advantageous and future pretreatment of samples.

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