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Determination of Inorganic Elements Content and Distribution in Bamboo Shoots by Microwave Digestion and ICP-MS

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Abstract

Microwave digestion and inductively coupled plasma mass spectrometry (ICP-MS) method was used to determine the contents of 25 inorganic elements in basal part, meat and shell of bamboo shoots. It could be concluded that the method could be applied to determine 25 inorganic elements in bamboo shoots. The elements with a dry basis content higher than 10 mg/kg were listed in the order of content decrease as follows, basal part of bamboo shoots: K > Ca > Mg > Mn > P > Al > Fe > Zn > Na; bamboo shoots meat: K > P > Ca > Mg > Mn > Fe > Al > Zn > Na > Ba > Sr; bamboo shoots shell: K > P > Mg > Ca > Mn > Al > Fe > Na > Zn; B, Pb, Cu, Cr ranged from 1.0 - 10 mg/kg; Ga, As, Se, Cd, Sn, Sb, V, Co, Ni were lower than 0.80 mg/kg. The harmful elements Cd, As, Cr, Hg, Pb were commonly monitored in feeds. The wet basis content (mg/kg) of Cd, As, Cr was lower than: 0.028, 0.022 and 0.42 respectively; no Hg was found; the content of Pb(mg/kg) in basal part, meat and shell of bamboo shoots was 0.82, 0.35 and 0.41 respectively. The results provide basic data for the development of bamboo shoots and its byproduct.

Keywords

Bamboo Shoots, Basal Part of Bamboo Shoots, Bamboo Shoots Meat, Bamboo Shoots Shell, Inorganic Element

1. Introduction

China is the world's main bamboo producing country, and the production of bamboo shoots is leading in the world. The production in Fujian, Zhejiang, Jiangxi and Hunan Province is the highest. About 40% of bamboo

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shoots are sold fresh. Others are made into boiled canned bamboo shoots, dried bamboo shoots, sour bamboo shoots and various soft-packing or canned condiments [1]. Processing by-products of bamboo shoots are also continuously developed and utilized. The bamboo shoot shell is rich in phytosterol, amino acids, polysaccharide, flavonoids and other bioactive substances [2]. Cellulose, hemicelluloses, lignin and others can also be separated from them [3] [4]. Bamboo shoot shells can be also made into the "nutritious diets" [5]. Sun Jingya *et al.* [6] extracted the brown pigment with the special odor of bamboo shoots from bamboo shoot shells and used as additives for the food industry. The application prospect is broad. The basal part accounts for 31% of the total weight of fresh bamboo shoots. It is rich in dietary fiber, β-sitosterol, etc. Now there are a lot of studies related to the extraction [7] [8]. Many inorganic mineral elements are beneficial to human health. Most of them exist in the human body in the form of complex, convey various substances necessary to life, and adjusting human body's metabolism [9]. A few toxic metal elements, such as chromium, nickel, arsenic, lead, cadmium and mercury, have carcinogenicity. The intake of some elements has a safe and suitable scope. Outside this scope, they will be harmful to the body [10]. Take Se as an example. Lack of Se may cause epidermal keratinization of human body and cancer, but excessive intake can cause poisoning [11]. So understanding the content of inorganic elements in bamboo shoots and its by-products provides the basis for development of food, medicine, etc.

At present, a number of studies on the organic components of bamboo shoots and its by-products from processing have been reported [12]-[15]. Because of the limitation of detection means, the attention to inorganic element is not comprehensive enough [16]. ICP-MS is a kind of analysis and test technology developed in the 1980s. Through the unique interface, it combines the high temperature ionization of ICP and sensitive and rapid scanning of MS, and forms a new kind of multi-element and isotope analysis method. The method can test elements with the mass number of 6 - 260 at the same time. The linear dynamic range of concentration has nine orders of magnitudes, and various elements with big content difference can be measured at the same time.

Thus, it quickly became an important tool of elemental analysis [17] [18]. This experiment adopts the method of microwave digestion and ICP_MS, and determines the content of 25 kinds of inorganic elements in basal part, meat and shell of bamboo shoots at the same time.

2. Materials and Methods

2.1. Instrument and Equipment

ICP-MS (ThermoFisher Scientific X series II, USA), microwave digestion instrument (CEM MARS, USA), ultrapure water generator (Milli-Q Element, Millipore, France), digestion instrument (Annan DV4000, Beijing).

2.2. Test Materials and Reagents

Bamboo shoots: Longyan in Fujian Province.

Guaranteed reagent of HNO_3 (65%), Germany Merck; guaranteed reagent of H_2O_2 (30%), Germany Merck; high purity argon with the purity of 99.999%; mixture of hydrogen and helium (high-purity, containing 7.12% of hydrogen); ultrapure water as the experiment water (18.2 MΩ); mixed tuned liquid of Li, Co, In and U of 10 mg/L, O2si of the United States; 1000 mg/L of Re, National Center of Analysis and Testing for Nonferrous Metals and Electronic Materials; B, Na, Mg, Al, Ca, P, K, Ca, V, Cr, Mn, Co, Ni, Cu, Zn, Ga, As, Sr, Cd, Ba, Hg, Pb, Fe, Se, Ge, In and Rh of 1000 mg/L, Iron & Steel Research Institute of China National Center for Quality Supervision and Testing of Iron and Steel; Sn and Sb of 500 mg/L, Iron & Steel Research Institute of China National Center for Quality Supervision and Testing of Iron and Steel; Au of 100 μg/g, National Institute of Metrology, China; and Tea Leaves GBW10016, Geophysical and Geochemical Prospecting Institute.

2.3. Method

2.3.1. Sample Treatment Method

Each part of the bamboo shoot was taken. 0.25 kg of wet material was taken from each part with the cross quartering method, dried at 60˚C, crushed and sieved with 40-mesh screen. 0.50 g of sieved sample was accurately weighed and placed into the digestion tank. 6.0 mL of HNO_3, 2.0 mL of H_2O_2 and 0.02 mL of Au standard liquid was added. After the same treatment of standard substance, 0.200 ml of Ga, Sn and Hg intermediate liquid (1000 ug/L) was added, respectively. It was digested for 2 h at 90˚C, cooled to room temperature, capped, sealed, and digested with microwave. The digestion conditions are shown in **Table 1**. Six parallel samples and blank

samples were made for every part sample, standard substance and adding standard sample.

2.3.2. ICP-MS Work Conditions

In the startup optimization, the signal value of Li, Co, In and U was adjusted to the maximum. Double charge and oxide interference was reduced to the value below 3.0%. The sampling depth was 150 mm. It was 16 mm horizontally and 285 mm vertically. The rate of cooling air was 13.02 L/min, that of auxiliary gas was 0.8 L/min, and that of atomization gas was 0.93 L/min. When the CCTS technology was used, the mixed gas was hydrogen + helium with the rate of 7:93. The gas flow rate was 4.5 mL/min. Float mode was used for high-content and high-sensitivity elements. Each element was scanned for 100 times. The hold time was 10 ms. There were 3 channels. The channel spacing was 0.02 AMU.

3. Results and Analysis

3.1. The Selection of Isotope and Interference Correction

In ICP-MS analysis, by the selection of appropriate isotope of element to be tested, equation correction or CCTS technology, mass spectrum interference can be prevented to the greatest extent. The isotope of each element to be tested includes ^{11}B, ^{23}Na, ^{24}Mg, ^{27}Al, ^{31}P, ^{39}K, ^{44}Ca, ^{451}V, ^{52}Cr, ^{55}Mn, ^{56}Fe, ^{59}Co, ^{60}Ni, ^{65}Cu, ^{66}Zn, ^{69}Ga, ^{75}As, ^{80}Se, ^{88}Sr, ^{111}Cd, ^{118}Sn, ^{121}Sb, ^{137}Ba, ^{202}Hg and ^{208}Pb. ^{80}Se is corrected with CCTS technology [19]. Co and Ni are corrected with the following equation. Co = ^{59}Co-[0.001 × ^{43}Ca], Ni = ^{60}Ni-[0.003 × ^{43}Ca] [17].

In order to overcome the sample matrix effect and other influences, the mixed internal standard of Ge, Rh, In and Re was added for correction of results.

Hg^{2+} has strong adsorption ability. To reduce the effect of memory effect on the results, when ICP-MS was used to measure the content of Hg, 0.02 mL of Au was added into the sample [20].

To protect the detector, according to the content of elements and sensitivity, Ca, Mg, AL, Na, P, K, V, Cr, Mn, Sr and Fe was measured with float mode and high resolution.

3.2. Work Curve and the Detection Limit

Ca, Mg, Al, Na, P, K, V, Cr, Mn, Sr and Fe were used to prepare a set of mixed standard solution. B, Co, Ni, Cu, Zn, Ga, As, Cd, Sn, Sb, Ba, Hg and Pb were used to prepare a set of mixed standard solution. Se was used to prepare a set of standard solution alone. Work curve of each element consists of evenly spaced 6 concentration values from 0 to the highest concentration. The highest concentration is shown in **Table 2**.

Blank solution was determined for 10 times. The concentration corresponding to the three times of standard deviation is the detection limit of the method. Linear range, correlation coefficient and detection limit of the method are shown in **Table 2**.

3.3. Accuracy and Precision of the Method

To verify the accuracy and reliability of this method, according to the experimental steps and operating conditions selected, Tea Leaves GBW10016, the national standard substance, was determined. The results are shown in **Table 3**. The measured values are within the scope of the standard value. The recovery rate of Ga, Sn and Hg is between 95.0% and 106.5%. RSD is between 1.1% and 6.3%. It proves that the method meets the requirements.

3.4. Test Results are Shown in Table 4

To detect bamboo shoot, bamboo shoots meat, bamboo in the content of inorganic elements using this method, the results are shown in **Table 4**.

Table 1. Working procedure of microwave digestion.

Step	Power (W)	Emission rate (%)	Heating up time (min)	Temperature (˚C)	Hold time (min)
1	1600	100	5	120	3
2	1600	100	3	150	3
3	1600	100	3	180	15

Table 2. Linear range, correlation coefficient and detection limit of each element.

Element	Linear range (μg/L)	Correlation coefficient	Detection limit (μg/L)	Element	Linear range (μg/L)	Correlation coefficient	Detection limit (μg/L)
B	0 - 100	0.9997	0.23	Cu	0 - 100	0.9997	0.40
Na	0 - 5000	0.9978	4.3	Zn	0 - 100	0.9998	1.6
Mg	0 - 2500	0.9988	1.0	Ga	0 - 10	1.0000	0.0081
Al	0 - 2000	0.9983	1.2	As	0 - 100	1.0000	0.030
P	0 - 10000	0.9999	12	Se	0 - 10	0.9996	0.20
K	0 - 2500	0.9998	1.6×10^2	Sr	0 - 100	0.9998	0.015
Ca	0 - 10,000	0.9993	40	Cd	0 - 10	0.9999	0.0023
V	0 - 100	0.9994	0.019	Sn	0 - 10	0.9999	0.026
Cr	0 - 100	0.9996	0.095	Sb	0 - 10	1.0000	0.0065
Mn	0 - 1000	0.9994	0.26	Ba	0 - 100	0.9996	0.069
Fe	0 - 1000	0.9994	1.9	Hg	0 - 10	0.9996	0.088
Co	0 - 10	1.0000	0.0033	Pb	0 - 100	1.0000	0.028
Ni	0 - 100	0.9996	0.20				

Table 3. Accuracy and precision of the method (n = 6).

Element	Standard value (mg/kg)	Measured value (mg/kg)	RSD (%)	Element	Standard value (mg/kg)	Measured value (mg/kg)	RSD (%)
B	14 ± 1	13.5	3.2	Cu	18.6 ± 0.7	18.9	4.0
Na	90 ± 10	85.0	5.2	Zn	51 ± 2	52.9	2.1
Mg	1860 ± 110	1728	4.8	As	0.09 ± 0.01	0.098	5.1
Al	940 ± 90	970	6.3	Se	0.098 ± 0.008	0.102	3.9
P	4500 ± 300	4720	4.4	Sr	9.1 ± 1.2	10.0	1.1
K	16,300 ± 700	16,900	3.1	Cd	0.062 ± 0.004	0.062	1.1
Ca	3260 ± 80	3310	3.7	Sb	0.022 ± 0.006	0.019	3.1
V	0.17 ± 0.03	0.20	2.4	Ba	9.6 ± 0.5	9.2	4.7
Cr	0.45 ± 0.10	0.53	1.9	Pb	1.5 ± 0.2	1.7	3.2
Mn	500 ± 20	485	2.1	Element	Standard adding value (mg/kg)	Recovery rate (%)	RSD (%)
Fe	242 ± 18	243	2.4	Ga	0.410	103.3	3.0
Co	0.22 ± 0.02	0.23	1.2	Sn	0.410	106.5	4.4
Ni	3.4 ± 0.3	3.11	2.7	Hg	0.410	95.0	4.7

Table 4. Content of inorganic element in each part of bamboo shoots.

Element	Basal part	Meat	Shell	Element	Basal part	Meat	Shell
K (g/kg)	27	35	9.9	B (mg/kg)	1.8	2.1	0.92
Ca (g/kg)	0.83	2.9	0.39	Cr (mg/kg)	1.8	1.1	2.0
Mg (g/kg)	0.73	1.3	0.43	Sb (ng/kg)	0.73	1.8	1.5
Mn (g/kg)	0.35	0.34	0.065	Ni (mg/kg)	0.52	0.80	0.22
P (g/kg)	0.22	4.4	0.75	Ga (mg/kg)	0.39	0.73	0.084
Al (g/kg)	0.19	0.080	0.057	V (mg/kg)	0.25	0.14	0.065
Fe (g/kg)	0.13	0.092	0.050	Cd (mg/kg)	0.12	0.14	0.019
Zn (mg/kg)	44	60	10	Se (mg/kg)	0.11	0.21	0.032
Na (mg/kg)	22	26	22	As (mg/kg)	0.095	0.13	0.039
Ba (mg/kg)	9.9	20	1.8	Co (mg/kg)	0.056	0.040	0.055
Sr (mg/kg)	5.9	16	1.2	Sn (mg/kg)	—	0.058	0.025
Cu (mg/kg)	5.0	6.9	2.3	Hg (mg/kg)	—	—	—
Pb (mg/kg)	5.0	4.2	2.9				

"—": not detected. Detection limit of Hg: 0.0088 mg/kg, detection limit of Sn: 0.003 mg/kg.

Table 5. Wet base content of harmful element (mg/kg).

	Pb	Cd	As	Cr	Hg
Basal part	0.82	0.028	0.022	0.42	—
Meat	0.35	0.017	0.015	0.13	—
Shell	0.41	0.0039	0.0079	0.41	—

The method can meet the testing requirements; the ranking of content of inorganic element above 10 mg/kg is as follows. Basal part: K > Ca > Mg > Mn > P > Al > Fe > Zn > Na; meat: K > P > Ca > Mg > Mn > Fe > Al > Zn > Na > Ba > Sr; shell: K > P > Mg > Ca > Mn > Al > Fe > Na > Zn; the elements whose content in each part is 1.0 - 10 mg/kg include B, Pb, Cu, Cr, etc. The content of Ga, As, Se, Cd, Sn, Sb, V, Co and Ni are below 0.80 mg/kg.

Rich Na, K and other elements which can maintain water in human body and constant pH of body fluid are contained. Ca content in meat of bamboo shoots is as high as 2.9×10^3 mg/kg. The content of Fe, Zn, Mn and other essential microelements of human body is $(10 - 3.5) \times 10^2$ mg/kg. The basal part and shall of bamboo shoots can be selectively developed into main feed of livestock. Se is the element drawing the most attention in the environment and health research field. Its content in each part of the bamboo shoots is above 0.032 mg/kg. Se content in the meat of bamboo shoots meat is 0.21 mg/kg.

Water content of basal part, meat and shell of bamboo shoots is 76.46%, 88.12% and 79.72% respectively. The calculated wet base content of Pb, Cd, As, Cr and Hg are shown in **Table 5**.

Harmful elements often monitored in food and feed include Cd, As, Cr, Hg, Pb, etc. The wet base content of Cd, As and Cr (mg/kg) is below 0.028, 0.022 and 0.42, respectively. Hg was not detected. The wet base content of Pb in meat, basal part and shell of bamboo shoots (mg/kg) is 0.82, 0.35 and 0.41, respectively. According to the standard in the strict GB 2762-2012 Maximum Limit of Contaminants in Food, the content of Pb is a little higher. When it is developed into food, this should be considered. According to GB13078-2001 Feed Hygiene Standard, all raw materials for feed are safe.

4. Conclusion

In this paper, microwave digestion and ICP-MS method is used to measure the content of 25 kinds of inorganic elements, namely Ca, Mg, Al, Na, P, K, V, Cr, Mn, Sr, Co, Ni, Cu, Fe, B, zinc, Ga, As, Cd, Sn, Sb, Ba, Hg, Pb and Se. By measuring the national standard substance and the standard addition recovery test, the results show that the method can meet the testing requirements. Meanwhile, it tests the distribution 25 kinds of elements in bamboo shoot, bamboo shoots meat, bamboo and provides the basic data for the comprehensive utilization.

Fund

Dissimilarity Evaluation of the characteristics of the nutrient components of the plant derived agricultural products from different producing area, different growing period, different storage and fresh keeping and different consumption patterns (GJFP201601503).

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