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NOTE

Determination of Major and Trace Elements in Lotus Seed by Inductively Coupled Plasma-Mass Spectrometry

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The trace elements (V, Cr, Mn, Co, Ni, Zn, Sr, Cd, Pb) and major elements (Na, Mg, Al, K, Ca, Fe) in two lotus seed samples were determined by inductively coupled plasma-mass spectrometry, the contents of major elements in lotus seed are according to order of K > Mg > Ca > Na > Fe > Al and that of trace elements is Mn > Zn > Sr > Ni > Cr > Cd > V > Pb > Co. K is abundance in the lotus seed, the contents of two samples are both over 1000 μg g^{-1}. The contents of Cd, Cr and Pb are lower than 0.06 μg g^{-1} in the two samples.

Key Words: Determination, Elements, Lotus seed, ICP-MS.

Lotus seed or lotus nuts are the seed of plants in the genus *Nelumbo*, particularly the species *Nelumbo nucifera*. The seed are of great importance to East Asian cuisine and are used extensively in traditional Chinese medicine and in Chinese desserts.

The lotus seed have the function of anti-aging according to Chinese medicine[1], people consider that the elements in the lotus seed can play an important role in this function[2], otherwise as food the lotus seed consumed widely by people, thus it is necessary to understand the concentration of elements in it. The methods of flame or graphite furnace atomic absorption spectrometry[3] and inductively coupled plasma-atomic emission spectrometry (ICP-AES)[4,5] were used regularly to determine the compositions of elements in the lotus seed samples. As method of inductively coupled plasma-mass spectrometry (ICP-MS) has low detection limits, high sensitivity and more efficient than ICP-AES, FAA and GFAA. Thus it provided a powerful instrument for multi-elements analysis.

In this paper 15 elements in lotus seed samples were determined by ICP-MS, included the trace elements (V, Cr, Mn, Co, Ni, Zn, Sr, Cd, Pb,) and major elements (Na, Mg, Al, K, Ca, Fe).

Two samples of lotus seed were sampled from the markets in Hangzhou and Shaoxing city Zhejiang province, in south-east of China. The samples were homogenized by a homogenate machine and collected in plastic bottles then preserved in ice chest under -18 ºC temperature.

A microwave digest system (Mars X, CEM Company, USA) was used to prepare test solutions. For detection of trace and major elements by ICP-MS (X-series II instrument, Thermo Fisher Company, USA).

About 1 g sample was accurately weighed and transferred into vessel, followed by adding 4 mL 65-70 % HNO_3, cold digestion over night, then added 2 mL 30 % H_2O_2. The vessels were capped and placed in the microwave system and select "Ramp to temperature" control type.

The parameters of digestion program listed in Table-1. At the end of the program, the vessels were cooled to room temperature in a fume hood and the pressure inside the vessels slowly released. After cooling the digested samples were transferred to 50 mL graduated polypropylene centrifuge tubes, washed three times with double-deionized water (DDW) to a volume of 50 mL and filtered by filter paper to remove any remaining undissolved solid particles that might block the nebulizer. Repeat the process, a reagent blank in the same way.

TABLE-1
OPERATING PARAMETERS, TEMPERATURE PROGRAMS FOR MICROWAVE SYSTEM

Stage	Power (W)	Ramp (min)	Control temperature (ºC)	Hold time (min)
1	1600	5.0	120	5.0
2	1600	10.0	185	30.0
3	1600	5.0	190	0

The operating parameters for ICP-MS are shown in Table-2. In order to minimize the interference of polyatomic ions and concomitant concentrations, during the detection 10 μg L^{-1} Rh solution was used as internal standard to compensate for the matrix effects[6]. Besides that the choosing alternative isotopes of different elements were carried out, that are listed in Table-3.

TABLE-2
OPERATING PARAMETERS FOR ICP-MS

Auxiliary (L/min)	0.74
Nebuliser (L/min)	0.85
Forward power (W)	1300
Analogue detector (V)	2060
PC detector (V)	1300
Resolution	Standard/ high
Dwell (ms)	100
Number of replicates	3
Peri-pump (r/min)	20

TABLE-3
SPECTROSCOPIC DATA OF MEASURED ISOTOPES

Isotopes	Abundance (%)	Limit of detection[a]
Trace elements		
^{51}V	99.8	0.048
^{52}Cr	83.8	0.17
^{55}Mn	100	0.43
^{59}Co	100	0.0047
^{60}Ni	26.2	0.34
^{66}Zn	27.9	0.39
^{88}Sr	82.6	0.0042
^{111}Cd	12.8	0.015
^{208}Pb	52.4	0.15
Major elements		
^{23}Na	100	0.0024
^{24}Mg	79	0.00055
^{27}Al	100	0.00034
^{39}K	93.3	0.035
^{44}Ca	2.1	0.35
^{56}Fe	91.8	0.034

[a]Measured in ng mL^{-1} for trace elements and μg mL^{-1} for major elements

The limits of detection were lower than 0.43 ng mL^{-1} and 0.35 μg mL^{-1} for trace elements and major elements respectively (Table-3).

The contents of 15 elements are showed in Table-4, which indicated that the contents major elements in lotus seed are according to list of K > Mg > Ca > Na > Fe > Al and that of trace elements is Mn > Zn > Sr > Ni > Cr > Cd > V > Pb > Co. K is abundance in the lotus seed, the contents of two samples are both over 1000 μg g^{-1} and all the contents of major elements are similar in the two samples except that of Na, which is higher in sample 2# than sample 1#.

TABLE-4
CONTENTS OF 15 ELEMENTS IN TWO LOTUS SEED SAMPLES (μg g^{-1})[a]

Elements	Sample 1#	Sampe 2#
V	0.0065 ± 0.00005	0.0079 ± 0.00037
Cr	0.044 ± 0.0021	0.058 ± 0.0058
Mn	28.9 ± 0.58	27.5 ± 0.64
Co	0.0092 ± 0.0003	0.028 ± 0.0011
Ni	0.21 ± 0.0075	0.075 ± 0.0015
Zn	1.9 ± 0.047	2.1 ± 0.064
Sr	0.81 ± 0.0087	1.2 ± 0.014
Cd	0.013 ± 0.0007	0.0076 ± 0.0013
Pb	0.005 ± 0.004	0.008 ± 0.005
Na	8.1 ± 0.16	25.7 ± 1.1
Mg	313.5 ± 3.5	406.6 ± 13.1
Al	0.43 ± 0.02	0.43 ± 0.01
K	1015.5 ± 8.4	1146.0 ± 28.4
Ca	59.3 ± 0.82	52.8 ± 1.3
Fe	3.6 ± 0.13	3.6 ± 0.09

[a]Results are based on the "fresh weight" material, mean ± SD ($n = 6$)

The contents of Cd, Cr and Pb as well, heavy metals can induce chronic toxicity to human body, are lower than 0.06 μg g^{-1} in two samples, that indicated people may consume the lotus seed safely.

Conclusion

Zhang *et al.*[4] used the ICP-AES to determination multi-elements in lotus seed; there were no signal peak of elements of Cd, Pb, Sn, W and Mo in their result. That due to the detection limits of ICP-AES was not lower enough for trace elements determination in lotus seed. In this paper it showed that the detection limits of trace elements by ICP-MS were range from 0.0042 to 0.43 ng mL^{-1}, can meet with the demand of detection.

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