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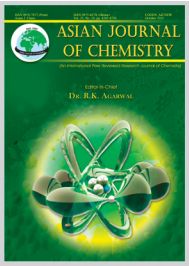

Determination of Macroelements and Microelements in Flos Sophorae by ICP-OES with Microwave Digestion

SUYING MA*, HAI XIA LV and XIAOJUN SHANG

School of Pharmacy, Xinxiang Medical University, Xinxiang 453003, Henan, P.R. China

*Corresponding author: Fax: +86 373 3029879; Tel: +86 373 3029879, E-mail: suyingma@163.com

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The contents of macroelements and microelements (Na, K, Ca, P, Mg, Mn, Fe, Zn, As, Cu, Se, Cr, Cd, Ni and Pb) in flos sophorae were studied by ICP-OES with microwave digestion. The best operation conditions of microwave digestion and ICP-OES were optimized. The results indicated that there were high contents of K, P, Ca, Mg, Na, Fe elements in flos sophorae and the sequence of metal elements contents is as follows P > Na > K > Ca > Mg > Fe > Zn > Mn > Cu > As > Cr > Se > Cd, while Ni and Pb elements were not detected. The ICP-OES with microwave digestion method is simple and accurate enough for determination of macroelements and microelements in flos sophorae.

Key Words: Macroelements, Microelements, Flos sophorae, ICP-OES, Microwave digestion.

INTRODUCTION

Fols sophorae (Huai hua in Chinese), the dried buds of *Sophora japonica* L, is a commonly used as traditional Chinese medicine herb. It is mainly distributed in the north China plain and loess plateau elevation 1000 metres high zone. It has been used for thousands of years in treating having blood in shit, urine and hemorrhoids, diarrhea, bleeding from the nose, and so on according to "quan guo zhong cao yao hui bian". It can also serve in beneficial role in improvement of blood circulation, prevention of blood vessel sclerosis, reduction in blood pressure. Now It has been confirmed that the flos sophorae contains flavonoids, plant steroid, tannic acid, 17 kinds of amino acids, protein and trace elements, *etc.*[1]. Many researches have proved that there was a close relationship between trace elements and efficacy of herbal medicine, human health, disease prevention. A few of methods have been developed to detect macroelements and microelements in flos sophorae. But the results were not ideal for equipment constrains[2-4].

High-pressure microwave digestion vessel using microwave warming and pressurized digestion sample is a significant improvement than classical chemical pretreatment technology. Inductively coupled plasma atomic emission spectrometry (ICP-OES) method has more advantages than traditional atomic absorption spectrometry and atomic fluorescence spectrometry, such as high sensitivity, good stability, wide linear range, small matrix effects, rapid analysis and multi-element determination and so on. It is now widely used in elemental analysis[5].

The objective of this research was to measure the macroelements and micro-elements in flos sophorae with a new method.

EXPERIMENTAL

Nitric acid and hydrogen peroxide of guaranteed reagent were purchased from Bafang Chemicals Co. Ltd., (Zhengzhou, P.R. China). Double deionized water was used throughout the analysis. All glassware's were soaked in nitric acid 10 % for 0.5 h and rinsed with deionized water before use.

Prepration of solutions: The combined standard solutions of Cu, Fe, Zn, Mn, Na, K, Ca, Mg is 1000 mg/L, (Merck). The combined standard solutions of As, Cr, Ni, Pb, Se, Cd is 100 mg/L. The standard solutions of P was prepared with 0.219 5 g KH_2PO_4 dissolved in 1000 mL water consisting of 5 mL H_2SO_4 and the final concentration is 50.0 mg/L.

Working solution: The combined standard solutions of Cu, Fe, Zn, Mn, Na, K, Ca, Mg were diluted with 2 % nitric acid, make the final solution to be 0.00, 0.50, 1.00, 2.00, 4.00, 8.00 mg/L. The concentrations of combined As, Cr, Ni, Pb, Se, Cd standard solutions was 0.00, 0.25, 0.50, 1.00, 2.00, 4.00 mg/L respectively; the concentrations of P standard solutions was 0.00, 5.00, 10.00, 15.00, 20.00, 25.00 mg/L, respectively.

An optima 2100 DV ICP-OES (Perkin-Elmer Instruments, Shelton, CT, USA) was used for the determination of elements

in this study. The operation conditions are summarized in Table-1.

TABLE-1
INSTRUMENTAL AND OPERATING CONDITIONS FOR ICP-OES MEASUREMENTS

Parameters	
RF power (watts)	1300
Nebulizer gas flow rate (L/min)	0.8
Plasma gas flow rate (L/min)	15.0
Auxiliary gas flow rate (L/min)	0.2
Sample flow rate (mL/min)	1.5

Samples: The flos sophorae was obtained in the campus of Xinxiang Medical University, Henan province, P. R. China.

Procedure: The flos sophorae sample was washed with deionized water 3-5 times to remove the dust and then it was filtered with pledget and dried in oven at 80 ºC. Finally it was grinded with stainless steel grinder and passed through a 100 mesh sieve.

An accurately weighed quantity of 0.5 g flos sophorae sample was digested by 5 mL nitric acid and 1 mL hydrogen peroxide in microwave. The translucent digested liquid was diluted with 2 % nitric acid. The final samples were analyzed with ICP-OES. The absorption measurements of the elements were performed under the conditions recommended according Table-1. The digestion parameters (temperature, time, power) were compared and the best operation conditions are summarized in Table-2.

TABLE-2
INSTRUMENTAL AND OPERATING CONDITIONS FOR MICROWAVE DIGESTION

No	Programmed power/W	Programmed time (min)	Held temperature (ºC)	Held time (min)
1	800	5	120	3
2	800	3	150	5
3	800	5	210	15

RESULTS AND DISCUSSION

Selection of wavelength: It is possible to choose 2-3 wavelengths for every element in ICP-OES method, one best wavelength with background correction and high precision was selected. The wavelength for Cu, Fe, Zn, Mn, Na, K, Ca, Mg, P, Se, Ni, Pb, Cd, As, Cr was 324.78, 259.96, 213.87, 257.61, 589.62, 766.52, 317.95, 279.10, 213.63, 1 96.04, 231.62, 220.37, 226.52, 193.71, 267.73 nm, respectively.

Standard calibration curve: Serial working solutions were analyzed with ICP-OES. The certain value was regressed with the certain concentration to calculate the calibration equation. The response fitted a linear regression model; the specific results were presented in Table-3.

Limit of quantification (LOQ): In this work, LOQ were determined by an empirical method that consisted of analyzing series of working solutions. Limit of quantification was defined as a signal-to- noise ratio 10. Furthermore, these values were in acceptable precision and accuracy values. The LOQ of K, Na, Ca, Mg, Fe, Mn, Zn, Cu, P, Se, Ni, Cr, Pb, Cd, As was 42.85, 0.0690, 0.0100, 0.0300, 0.0062, 0.0014, 0.0018, 0.0054, 0.0 760, 0.0061, 0.0150, 0.2400, 0.0420, 0.0034, 1.790 mg/L, respectively.

TABLE-3
CALIBRATION PLOTS FOR ICP-OES MEASUREMENTS

Elements	Regression equation	Correlation coefficient
K	$y = 10.140x + 506.0$	0.9999
Na	$y = 43.720x + 18815.4$	0.9988
Ca	$y = 31.030x + 6027.2$	0.9989
Mg	$y = 2.788x + 121.1$	0.9999
Fe	$y = 44.580x - 402.6$	0.9999
Mn	$y = 183.000 + 5066.1$	0.9999
Zn	$y = 28.710x + 233.5$	0.9999
Cu	$y = 47.620x + 1682.5$	0.9999
P	$y = 353.1x - 104.1$	0.9986
Se	$y = 1.192x + 61.9$	0.9992
Ni	$y = 27.250x + 160.2$	0.9999
Cr	$y = 4.993\,000x - 7\,0433.9$	0.9999
Pb	$y = 2\,231x$-13.9	0.9999
Cd	$y = 61\,800x + 2\,116.6$	0.9999
As	$y = 573.8x + 221.1$	0.9963

Recovery: The recovery of flos sophorae was determined by comparing the data obtained by the direct injection of standard solutions to those obtained after the extraction procedure. 0.5 g flos sophorae sample was accurately weighed; a standard addition of standard working solutions to each sample and the values was measured using the above procedure. Three reduplicate samples were made for every element. The results are shown in Table-4.

TABLE-4
RESULTS OF RECOVERY EXPERIMENT (n = 3)

Elements	Detecting value (μg)	Recovery (%)	RSD (%)
K	415.3	99.50	0.28
Na	450.5	99.18	0.33
Ca	380.5	100.23	0.46
Mg	352.6	99.35	0.86
Fe	303.5	99.24	1.85
Mn	23.24	98.38	0.89
Zn	39.4	98.98	0.68
Cu	18.8	99.32	1.02
P	550.24	99.53	0.98
Se	1.52	99.12	1.24
Ni	0.15	98.63	1.45
Cr	0.75	100.41	2.03
Pb	0.15	99.18	1.54
Cd	0.15	98.20	1.78
As	1.55	101.56	1.24

The recovery of developed method was between 98.2-101.56 %. These results showed that the method was precise, accurate and selective.

Accuracy experiment: The control solutions were measured five times using the above procedure to estimate the accuracy of the method. The results showed that the RSDs were lower than 2.03 %. The RSDs showed that the method was precise.

Determination the elements content of flos sophorae: The well-handled flos sophorae was analyzed using the above procedure and the values were calculate according to the regression equation. The results are shown in Table-5.

TABLE-5
DETECTION RESULTS OF FLOS FOPHORAE (mg/Kg)

Cu	Fe	Zn	Mn	Na	K	Ca	Mg	P	Se	Ni	Pb	Cd	As	Cr
13.5	269.3	28.7	15.38	432.2	389.3	303.8	287.6	537.48	0.18	-	-	0.11	0.74	0.34

The results showed that flos sophorae was abundant in macroelements and micro-elements, the contents of K, P, Ca, Mg, Na, Fe was 389.3, 537.48, 303.8, 287.6, 432.2, 269.3 mg/Kg, respectively.

Conclusion

The ICP-OES with microwave digestion method is simple, reliable, high sensitivity and good accuracy in determination of the macroelements and microelements in flos sophorae. The results indicate that flos sophorae contain massive K, P, Ca, Mg, Na, Fe, but also includes rich Mn, Cu, Zn and include a few of As, Se, Cr and Cd element, while Ni and Pb elements were not detected. All these elements have high complexing capacity, but the relationship of efficacy and the contents of these elements require further research.

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