Method Development for Determination of Pb(II) Ions in Thai Herbs Using Flame Atomic Absorption Spectrometry

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Abstract

An accurate and precise quantitation method for the determination of Pb(II) ions in Thai herbs has been developed by using wet digestion method followed by atomic absorption spectrometry (AAS). The developed method has been fully validated in various parameters including linearity range, accuracy, precision, limit of detection (LOD), and limit of quantitation (LOQ). The results exhibited that the calibration graph was linear in the range of 1.0-10.0 mg/L, with a detection limit (LOD) and limit of quantitation (LOQ) of 0.03 mg/L and 1.2 mg/L, respectively. The relative standard deviation (%RSD) for 6 replicate measurements of 1.0 mg/L Pb(II) was ±2.5%. The method was applied to the analysis of 5 types of Thai herb samples. Percentage recoveries were in the range 90.4-110.6%. It was found that Pb(II) concentrations in all Thai herb samples were less than the maximum residue level.

Keywords: Lead; Thai herbs; Atomic Absorption Spectrometry

Introduction

Herbal products are traditionally used in developing countries. Nowadays, interest in natural therapies has also become popular, resulting in the rapidly increasing worldwide consumption of these herbal. Therefore, a critical evaluation of their safety and quality is important. The World Health Organization[1] has established standards for the quality control of medicinal plants including the classification, botanical identification, determination of active principles, and identification of contaminants. One of the most frequent contaminants likely to be found in herbal products is Pb(II) ions[2]. Pb(II)is known to be not only high toxic elements to

human beings but also may cause critical environment problems[3].The World Health Organization sets the maximum permissible levels of Pb(II) in medicinal herbs[4] at 10 mg/kg. And also, in Thailand, the Ministry of Public Health sets the maximum permissible levels of Pb(II) in medicinal plant materials and herbal products[5] at 10 mg/kg. In this work, an accurate and precise quantitation method for the determination of Pb(II) ions in Thai herbs has been developed by using wet digestion method followed by atomic absorption spectrometry (AAS). The developed method has been fully validated in various parameters including linearity range, accuracy, precision, limit of detection (LOD), and limit of quantitation (LOQ).

Experimental

Sample collection

Five samples of herbal medicines in the form of powder were selected and bought directly from herbal drug stores in Nakhon Si Thammarat Provinces, Thailand between April 2019 and June 2019. All samples were from domestic cultivated plants and produced in Thailand. After delivery to the laboratory, coarse particles like powders were ground to fine particles using mortar and pestle. The fine powdered form samples were dried in an oven at 60 °C for 4 hours and stored in a desiccator at room temperature for about 10 days until it reached a constant mass. The dried samples were then individually packaged in clean poly-ethylene bags and stored in a desiccator. Each sample was analyzed in triplicate.

Sample preparation

Approximately 0.25 g of all herbal samples were accurately weighed into glass test tubes with a cap (50 mL), and 2.0 mL of 2:1 acid-oxidant mixture (conc. $HNO_3:H_2O_2$) was added. The glass tube cap was tightly closed. All tubes were allowed to stand for 10 minutes at room temperature. And then, the tubes were immersed in the ultrasonic water bath for 10 minutes. After 10 minutes of the first digestion step, 10 mL of 2:1 acid-oxidant mixture was added and the tubes were digested under the same condition for a further 10 minutes. After sonication, the sample digestion was made up to 10 mL in volumetric flasks with deionized water and then was filtered through filter paper. The final volume was stored in polyethylene bottles at 4 °C for analysis. Blanks were also treated in the same manner without samples for each experiment.

Results and Discussion

Analytical features of the proposed system

The analytical characteristics of the proposed method were investigated. Using the optimum conditions, the standard calibration in the range of 1-10 mg/L was constructed by plotting the absorbance against concentrations. The limit of detection (LOD) ($3\sigma/s$) and limit of quantification (LOQ) ($10\sigma/s$) [where σ is SD of digestion blank (n = 11) and s is the slope of calibration curve] were obtained at LOD 0.03 mg/L and LOQ 1.20 mg/L, respectively. The relative SDs for 6 replicate determinations of 1 mg/L Pb(II) was ±2.5%. The reproducibility for six determinations of 1 mg/L Pb(II) was 3.2%.

Analysis of herbal medicine samples

The proposed procedure was used for flame atomic absorption spectrometry (FAAS) determination of Pb(II) in herb samples. The analysis results of Pb(II) in all samples are presented in Table 1. To perform the recovery study, all samples were spiked with Pb(II) at 1 mg/L and 10 mg/L, respectively. Satisfactory results for the concentration levels studied were obtained for Pb(II), with percentage recoveries of 90.4-110.6%. As presented in Table 1, most of all studied samples were not contaminated with Pb(II). The maximum permitted level (MPL) of Pb(II) in medicinal plant materials and finished herbal products[6] is 10 mg/kg. Therefore, It was found that Pb(II) concentrations in all Thai herb samples were less than the maximum permitted level.

Table 1 Analytical features of merit for Pb(II) using wet digestion-FAAS method

Linear equation	Y = 0.0275X + 0.0015
Linear range (mg/L)	1.0 - 10.0
Correlation coefficient (R^2)	0.997
Precision (%RSD) Intra-day (n = 6)	2.5
	3.2
Inter-day (n = 6)	
Limit of detection (mg/L)	0.03
Limit of quantitation (mg/L)	1.20

Table 2.   Analysis of Pb(II) in herbal samples by wet digestion-FAAS method

Common name	Scientific name	Amount of Pb(II)(mg/kg)	Recovery (%)
Soap pod (leaf)	*Acacia concinna* (Wild.) DC	ND	98.7
Mangosteen (peel)	*Garcinia mangostana* Linn.	ND	90.4
Sappan wood	*Caesalpinia sappan* Linn.	ND	105.4
Horseradish tree (leaf)	*Moringa oleifera* Lam.	ND	110.6
Turmeric	*Curcuma longa* Linn.	ND	103.9

Conclusion

High efficiency sample preparation procedure based on wet digestion was successfully applied for the acid digestion of the herbal samples. Determination of Pb(II) was performed by FAAS with sensitive, precise, and accurate results. The recommended method offered fast, convenient, and high sample throughput for herbal samples. The amount of Pb(II) contaminants in the herbal plants were not found for all samples. And also, all herbal samples were less than the maximum permitted level. This study provides significant data on the safety and quality of herbal medicine consumed in Thailand. In addition, the proposed method has potential as a good alternative for analysis of Pb(II) contaminants in various biological samples.

References

1**.**   World Health Organization. WHO Quality control methods for medicinal plant materials. Geneva: WHO; 1998.

2. J. Zhang, B. Wider, H, Shang, X. Li, E. Ernst, Quality of herbal medicines: challenges and   solutions. *Complement Ther Med.* 2012, 20, 100-6.

3. V. Tripathy, B.B. Basak, T.S. Varghese, A. Saha, Residues and contaminants in medicinal herbs   a review. Phytochem Lett. 2015, 14, 67-78.

4. M.C.V. Mamani, L.M. Aleixo, M.F. De Abreu, S. Rath, Simultaneous determination of cadmium   and lead in medicinal plants by anodic striping voltammetry. *J. Pharmaceut. Biomed.* 2005, 37,   709-13.

5. S. Nookabkaew, N. Rangkadilok, J. Satayavivad, Determinationof trace elements in herbal tea   products and their infusions consumed in Thailand. *J. Agr. Food. Chem.* 2006, 54, 39-44.

6. World Health Organization. WHO guidelines for assessing quality of herbal medicines with   reference to contaminants and residues. Geneva: WHO; 2007.