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DETERMINATION OF CADMIUM IN SAUDI ARABIAN IMPORTED GREEN TEA SAMPLES BY ICP-MS

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ABSTRACT

Cadmium (Cd) in nine different Saudi Arabian imported green tea samples originated from China has been determined using inductively coupled plasma mass spectrometry (ICP-MS). Tea infusion and microwave acid digestion procedures are used for sample pre-treatment. Infusion is prepared from 2.0 g of tea samples in 100 mL boiled distilled and de-ionized water, digestion is performed with 25% (v/v) nitric acid. Samples are diluted 50 times with 1.0% (v/v) nitric acid solution which contain rhodium as an internal standard before aspiration into ICP-MS. The method showed, limit of quantification of 0.001 mg/L, 0.001 – 0.200 mg/L linearity range (r = 0.9999) and relative standard deviation (% RSD) value for reproducibility (inter-day precision) of 19%. The concentrations range of cadmium (total) in the analyzed green tea samples is 0.081 - 34.295 mg/kg. The total concentration of cadmium released 2% - 20% into tea infusions with boiling water. The calculated average daily intakes of cadmium in tea infusions was low and within the bounds of safety (< 0.001 mg/kg/day).

Key words: Inductively coupled plasma mass spectrometry (ICP-MS), Cadmium (Cd), Green tea, Infusion.

INTRODUCTION

Tea is the most popular beverage in the world. It is made from the processed leaves of the plants. The three most popular types of tea (green, oolong, and black) are distinguished on the basis of degree of fermentation. The leaves of green tea are dried and roasted but not fermented for green tea product[1]. Many studies have concluded that tea has numerous beneficial effects on health including the prevention of many diseases such as skin cancer, Parkinson's diseases, myocardial infarction and coronary artery disease[2,3].

The contamination of tea by heavy metals may pose a serious threat to human,

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because they remain in environment and pass to food chain[4]. The increasing industrial use of cadmium causes soil, air and water contamination[5]. Cadmium and its compounds have been classified as human carcinogens based on sufficient evidence of carcinogenicity in humans. Exposure to various cadmium compounds increased the risk of death due to lung cancer and it is also associated with liver tumors and toxicity[6-8]. Accordingly, cadmium content in tea is a public health issue.

The concentration of cadmium in different tea leaves and tea infusion samples have been studied, but there is still not enough information on its allowable standard limits[4,9,10]. U.S. EPA calculated an estimated daily reference dose for chronic oral exposure from food of 0.001 mg/kg/day[11]. In the present study, cadmium has been determined in nine imported green tea samples using ICP-MS. Tea infusion and microwave acid digestion procedures are used for sample pre-treatment. The data obtained will provide information on cadmium toxic levels in these samples when compared to U.S. EPA cadmium reference dose for chronic oral exposure from food.

EXPERIMENTAL

Instrumentation

Perkin-Elmer SCIEX inductively coupled plasma mass spectrometer (ICP-MS), model ELAN 9000, with S10 auto-sampler, DELL PC with Perkin-Elmer software system control, MDS SCIEX Canada, was used for the detection of cadmium. Its operating conditions given in Table 1[12,13]. Multi-wave 3000 acid digestion system, Anton Paar GmbH, Graz, Austria, Milli-Q academic, RiOs 8, water purification system (≥ 18.2 MΩ), Millipore, France, and Beckman Coulter, Allegra X-12 centrifuge, 3500 RPM, USA were used in this study.

Table 1: Instrumental operating conditions for ICP-MS

Vacuum pressure (Torr)	**2.03e-005**	**Torch**	**Quartz**
RF power (Watts)	1100	**Skimmer cone**	Nickel
Nebulizer flow rate (L/min)	0.84	**Sampler cone**	Nickel
Lens voltage (Volts)	7.5	**Nebulizer**	Cross flow tip
Detector mode	Pulse	**Alumina injector**	2.0 mm i.d.

Reagents, standards and samples

Nine different imported Chinese green tea samples were collected from Saudi Arabian markets for the analysis. Cadmium standard solution (1000 µg/mL) supplied by Perkin-Elmer was used for preparation of standard calibration curve. Rhodium standard solution (1000 µg/mL) supplied by Perkin-Elmer was used for preparation of internal standard. FAPAS soya flour quality control test material, T0770 and soya flour external proficiency testing samples, T07109, Food & Environment Research Agency (fera), York, U.K, were used for precision and accuracy of the method. Distilled and de-ionized water was obtained using Milli-Q academic (RiOs 8), Millipore, France and nitric acid 69.0-70.0 % for trace element analysis from Baker, Inc., NJ, USA, were used throughout.

Procedure

All standards were made by appropriate dilution of 1000 mg/L stock solution of cadmium with 1.0% (v/v) HNO_3. A series of cadmium standards in 1.0% (v/v) HNO_3 was prepared to contain cadmium at 0, 10, 50 and 100 µg/L. All quality control, external proficiency testing, green tea samples were digested into multi-wave digestion system using 25% (v/v) nitric acid. Infusion is prepared from 2.0 g of tea sample in 100 mL boiled distilled and de-ionized water. Matrix matched calibration standards, quality control, external proficiency testing and green tea samples were prepared by adding 200 µL of each into 10 mL diluent (diluent was prepared by adding 10 µL of Rhodium as an internal standard into1 L of 1.0% (v/v) HNO_3). Reagent blank was prepared similarly by adding 200 µL of distilled de-ionized water. Blank, standards and samples were mixed, centrifuged and carried out in the ICP-MS auto-sampler for measurements. Data acquisition parameters were, peak hopping using mass 111 amu, 50 ms dwell time, 20 sweeps per reading, 1 reading per replicate and 3 replicates. The total analysis time per solutions was 40 s.

RESULTS AND DISCUSSION

The analytical standard calibration curve obtained for cadmium gave correlation coefficient values better than 0.9999 in all cases. The linearity of the method in an aqueous matrix was obtained using twice the high working standard, (200 µg/L) and diluted serially six times and run against the analytical curve giving correlation coefficient of 0.9999 over 6.3–200.0 µg/L range. Similarly, detection limits were obtained with a serial dilution of standard one (10 µg/L) by six times (0.3–10.0 µg/L) giving a correlation coefficient of 0.9999 and limit of detection of 0.3 µg/L, limit of quantification of 1.0 µg/L and an analytical measurement range of 1.0-200.0 µg/L.

The precision of the method was tested using FAPAS Soya flour quality control sample, T0770. The Inter-day precision data for cadmium obtained from twenty determinations are in agreement with ± 19% correlation of variation (CV) value. The mean concentration of cadmium, standard deviation and satisfactory range are shown in Table 2. For accuracy, one external proficiency testing, FAPAS, Soya flour, T07109, was analyzed, and three tea samples were analyzed by both KFSHRC, Saudi Arabia and Food and Environment Research Agency (fera), UK for comparison. The percent difference values range from 7-18 as shown in Table 3.

Table 2: Inter-day precision data for cadmium

Quality control	n[a]	Mean (mg/kg)	SD (mg/kg)	Satisfactory range (mg/kg)	CV (%)
FAPAS[b]	20	0.306	0.059	0.189-0.424	19

[a] Number of reading.

[b] FAPAS Soya flour Quality control test material, T0770, Fera, U.K.

Table 3: Comparison of cadmium results obtained by KFSHRC and fera laboratories

Sample	KFSHRC[a] (mg/kg)	fera[b] (mg/kg)	% Diff. from fera
Tea 1	0.130	0.110	18
Tea 2	6.554	5.780	13
Tea 3	8.762	9.600	9
Proficiency testing[c]	0.150	0.140	7

[a] King faisal specialist hospital & research center, KSA.

[b] Food & environment research agency, UK.

[c] PT, FAPAS, soya flour, T07109, fera, UK.

The concentration range for cadmium in the nine different imported Chinese green tea samples is 0.081-34.295 mg/kg with acid digestion and 0.007-1.706 mg/kg using the infusion procedure. The total concentration of cadmium released 2%-20% into tea infusions. Concentration of cadmium in green tea products and their infusions and cadmium percentage release are shown in Table 4. The calculated average daily intakes of cadmium

from green tea infusions, based on 2 g of tea in 100 mL, three cups/day and 70 kg adult body weight was low for all cases, < 0.001 mg/kg/day and within the bounds of safety[14]. Average daily intakes of cadmium from green tea infusions and recommended reference dose are presented in Table 5.

Table 4: Concentration of cadmium in green tea products and their infusions

Sample	Mean (± RSD) mg/kg (Tea product)	Mean (± RSD) mg/kg (Tea infusion)	% Release [a]
Tea 1	0.130 (± 0.025)	0.007 (± 0.001)	5
Tea 2	6.554 (± 1.245)	0.947 (± 0.180)	15
Tea 3	8.762 (± 1.665)	1.706 (± 0.324)	20
Tea 4	0.149 (± 0.028)	0.021 (± 0.004)	14
Tea 5	0.081 (± 0.012)	0.013 (± 0.003)	16
Tea 6	0.114 (± 0.022)	0.023 (± 0.004)	20
Tea 7	0.107 (± 0.020)	0.021 (± 0.004)	20
Tea 8	0.090 (± 0.017)	0.017 (± 0.003)	19
Tea 9	34.295 (± 6.516)	0.766 (± 0.146)	2

[a] Calculated as percentage of the total cadmium amount present in tea powder

Table 5: Average daily intakes of cadmium from green tea infusions

Sample	Average daily intakes [a]	Recommended reference dose [b]
Tea 1	< 0.001 mg/kg/day	
Tea 2	< 0.001 mg/kg/day	0.001 mg/kg/day
Tea 3	< 0.001 mg/kg/day	
Tea 4	< 0.001 mg/kg/day	
Tea 5	< 0.001 mg/kg/day	0.001 mg/kg/day
Tea 6	< 0.001 mg/kg/day	

Cont…

Sample	Average daily intakes [a]	Recommended reference dose [b]
Tea 7	< 0.001 mg/kg/day	
Tea 8	< 0.001 mg/kg/day	0.001 mg/kg/day
Tea 9	< 0.001 mg/kg/day	

[a] Calculations based on 2 g of tea in 100 mL, three cups per day and 70 kg adult body weight.

[b] U.S. EPA reference dose for chronic oral exposure

CONCLUSION

The method is accurate, precise and applied successfully for monitoring cadmium content in some Saudi Arabian marketed green tea products originated from China. The calculated average daily intakes of cadmium from green tea infusions was low and within the bounds of safety.

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