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Determinations of Cadmium, Lead, Arsenic and Mercury in Rice from Iran

Somayeh Sadat Fakoor Janati, Hamed Reza Beheshti, Javad Feizy*, Niloofar Khoshbakht Fahim

Testa Quality Control Laboratory, North-East food industrial technology and biotechnology park, Mashhad, Iran

*Email: feizy.j@gmail.com

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Abstract

Rice is one of the cereals that is more consumed in the human food all over the world. It is used in various forms for preparation of food, cookies, cakes, and etc. This study was applied for determination of lead (Pb), cadmium (Cd), arsenic (As) and mercury (Hg) in 100 samples of rice purchased supermarkets Khorasan province, Iran. The calibration curves exhibited good linear correlation coefficients (0.989–0.998), excellent recoveries (97.50–99.00%), and limits of detection (LODs) between 0.36–5.20 ng g⁻¹. The results showed that As was the most abundant of the trace elements in rice with average concentration 51.85 ng g⁻¹. The results obtained after complete digestion of these samples and determination using graphite furnace Atomic Absorption Spectrometry (GFAAS) and hydride generation Atomic Absorption Spectrometry (HGAAS).

Keywords: AAS; Arsenic; Cadmium; Lead; Mercury; Rice.

1. Introduction

Rice, especially white rice, *Oryza sativa* L. is the staple food in the diet of various people including Asian countries [1]. Rice is the second food in high consumption among Iranian people. It is the commonest crop grown in agricultural lands in the north of Iran [2].

Many workers have reported trace element concentrations, especially for Cd, Pb, As, Hg and the other elements in rice grains from various countries [3-9]. Al-Saleh and Shinwari [8] for example, noted that the average levels of Cd, Pb and Hg in rice grains were 0.02, 0.135 and 0.0032 mg/kg, respectively. Shimbo et al. [7] also reported that the geometric mean contents of rice produced in Japan in 1998–

2000 were 0.05 and 0.002 mg/kg based on fresh weight for Cd and Pb, respectively. According to Bennett et al. [6], the median values in wild rice seed from northern Wisconsin, USA were 0.136, 0.016 and 0.250 mg/kg for As, Cd and Pb respectively. Jung [10] also reported that Cd and Pb concentrations in rice grown in various countries were in the range of 0.01 to 0.05 and 0.001 to 0.5 mg/kg, respectively. Various studies have also focused on investigation of a dietary reference intake (DRI) or a provisional tolerable weekly intake (PTWI) value for essential and trace elements by consuming food, drinking water and nutrition [11-15].

In the analysis of heavy metals in plant materials and food samples, atomic absorption spectrometry (GFAAS, FAAS) is reported the most frequently [16-21].

The aim of presented work is estimation of the quantity of Cd, Pb, As and Hg intake in rice, that is especially sold and consumed in Khorasan province of Iran.

2. Materials and methods

2.1. Apparatus

An Analytik Jena AG AAS ZEE nit 700 AAS (Jena, Germany) equipped with a hydride generation system (HS60) and GFAAS with the Zeeman background corrector was used in the experiments. WinAAS Version 4.5.0 software was used. Cd and Pb were determined with GFAAS. The operating parameters for working elements are given in Table 1. As and Hg were determined by HGAAS and cold vapor atomic absorption spectrophotometer, respectively. The operating parameters for working these elements were set as recommended by the manufacturer.

Table 1. Operating Parameters for Working Elements

Analyte	Wavelength (nm)	Lamp intensity (mA)	Split (nm)
Cd	228.8	3	1.2
Pb	283.3	4	0.8

2.2. Reagents and solutions

All the reagents were obtained from Merck (Darmstadt, Germany). Deionized double distilled water was used throughout the experimental work. Laboratory glassware was kept overnight in 10% (v/v) nitric acid. Before use, the glassware were rinsed with deionized water and dried in a dust free environment. The Phosphate-Matrix modifier solution (Merck) added to samples and standards was prepared by diluting 1 mL of Phosphate modifier stock solution ($\text{NH}_4\text{H}_2\text{PO}_4$ 100± 2 g/L in H_2O) to 10 mL, resulting in a final concentration of 2500

mg/L Phosphate. The rice samples investigated in this study were locally available brands, collected from supermarkets of Khorasan province, Iran.

2.3. Digestion procedures

For digestion with wet ashing, 5 g of rice samples were used. Wet digestion of samples was performed by using mixtures of two acids, namely, HNO_3 -HCl. 30 mL of concentrated HNO_3 was used for 5.0 g of sample. Each mixture was heated on the hot plate. Gently boil until 3-6 mL digest remain. Then, 25 mL concentrated HCl was added. Increase heat, and boil until 10-15 mL volume remain. After cooling, the residue was filtered through blue band filter paper. Then the sample was diluted to 50 mL with distilled water. The blank digestions were also carried out in the same way [22].

2.4. Calibration Curves

Four external standard curves were constructed using reference standard to qualify the metals (Cd, Pb, As and Hg) content in all samples. Calibration curves were performed with five or six different concentrations. The square of correlation coefficient (r^2) were 0.989, 0.997, 0.996 and 0.998 for Cd, Pb, As and Hg, respectively.

3. Results and discussion

Various sources contribute to the metal composition of the rice. The sample pretreatment procedure must take into account the analyte of interest, the matrix characteristics and the minimal required time period of the analytical technique considered. In this work, Cd and Pb were analyzed by GFAAS. As was analyzed with HSAAS and Hg was analyzed with cold vapor method. The analytical characteristics of the AAS method are shown in Table 2.

Table 2. Analytical Characteristics of the AAS Method

Analyte	Limit of detection (ng/g)	Limit of quantification (ng/g)	Repeatability (%RSD, n=7)	Recovery (%)
Cd	0.36	1.15	1.16	97.50
Pb	5.2	16.5	1.68	97.97
As	0.4	1.3	6.4	99.00
Hg	0.36	1.14	5.7	98.50

Accuracy was examined by the determination of the recoveries of the Cd, Pb, As and Hg. The recoveries of Cd, Pb, As and Hg from samples spiked at levels of 10 and 100 ng/g for Cd and Pb, respectively and 2 ng/g for As and Hg were quite good (Table 2). Relative standard deviations for within laboratory repeatability (RSD_r, n=7) range from 2.03 to 2.38. These results confirm the validity of the method for determination of the investigated metals. As the Table 3 shown, Cd, Pb, As and Hg were detected in 65%, 100%, 100% and 80% of rice samples with mean value of 10.02, 26.02, 51.85 and 13.44 ng/g, respectively. Cd, Pb and As concentrations were compared with upper limits (60, 150 and 150 ng/g for Cd, Pb and As, respectively) and approved by Iranian Ministry of Health (ISIRI 2010). In the Turkish Food Codex also maximum limit values given for both Pb and Cd in the rice samples were 300 ng/g [23]. The maximum Cd level permitted in rice samples is 400 ng/g according to the Codex Alimentarius [24]. There is no health problems resulting from Khorasan rice consumption for three elements of Cd, Pb and As, because their concentrations in rice sample were lower compared to the upper limit (60, 150 and 150 ng/g for Cd, Pb and As, respectively) approved by the Iranian Ministry of Health (ISIRI 2010). There is no limit for Hg in rice in Iran. So, for these three elements there are no health problems resulting from Khorasan rice consumption.

Table 3. Heavy metal concentration (ng/g) of rice samples

Analyte	Average (±SD)	Range
Cd	10.02 (±6.18)	0.45-21
Pb	26.02 (±14.67)	8-67
As	51.85 (±29.24)	5-144
Hg	13.44 (±9.13)	1-39

4. Conclusions

In this study, 100 rice samples from Khorasan province of Iran were analyzed for 4 elements using GFAAS and HGAAS and an acid digestion method. All results for 100 samples are shown in Table 4. The acid digestion system in rice samples provides a simple and effective method of sample digestion. The calibration curve exhibit good linear correlation coefficients (0.989–0.998), excellent recoveries (97.50–99.00%), and limits of detection (LODs) between 0.36–5.20 ng/g. This study is expected to provide important insight into the disparity of the mineral element concentrations in different rice samples. Maximum contents of Cd, Pb, As and Hg in the all samples were found as 21, 67, 144 and 39 ng/g, respectively. Therefore, routinely monitor is needed for these elements as a food quality control measure. Additional investigations, currently ongoing with our samples, will provide further information on this potentially useful taxonomic tool.

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Table 4. Results of total data for 100 samples (ng/g)

No. Sample	Cadmium	lead	Arsenic	Mercury	No. Sample	Cadmium	lead	Arsenic	Mercury
1	16	16	23	16	51	ND	17	30	ND
2	11	8	56	5	52	14	12	35	14
3	9	15	51	10	53	ND	25	17	ND
4	ND	25	49	ND	54	ND	14	30	16
5	15	12	29	11	55	6	19	38	25
6	13	56	144	7	56	8	17	57	8
7	12	17	103	ND	57	ND	19	75	4
8	3	19	120	20	58	21	41	54	30
9	0.46	15	64	14	59	ND	43	68	5
10	ND	10	79	15	60	4	18	43	12
11	0.9	8	60	9	61	ND	17	35	13
12	ND	16	110	ND	62	ND	25	41	10
13	0.45	18	81	4	63	13	65	50	7
14	0.9	19	44	14	64	ND	46	50	12
15	4	11	65	23	65	0.9	27	57	3
16	2	12	65	9	66	ND	46	87	13
17	1	13	71	13	67	2	53	117	12
18	ND	22	75	3	68	ND	64	41	9
19	16	24	72	15	69	12	22	114	9
20	ND	21	59	ND	70	ND	32	60	5
21	ND	15	53	26	71	18	18	102	7
22	12	14	89	17	72	20	16	73	6
23	7	25	67	22	73	ND	17	32	5
24	ND	21	81	34	74	ND	43	33	6
25	9	26	67	14	75	15	32	37	10
26	10	46	63	ND	76	ND	32	45	27
27	11	35	59	24	77	13	16	45	22
28	13	38	123	17	78	ND	26	32	4
29	0.56	15	107	22	79	7	24	37	28
30	2	52	111	10	80	15	41	31	22
31	0.48	17	101	13	81	1	19	23	16
32	0.9	9	89	21	82	ND	26	24	3
33	ND	45	71	19	83	15	18	28	ND
34	ND	39	55	13	84	13	18	21	ND
35	16	14	59	2	85	ND	22	24	ND
36	19	16	60	2	86	15	14	30	ND
37	ND	66	81	2	87	19	19	25	39
38	10	45	31	2	88	ND	17	30	11
39	11	25	137	2	89	20	16	25	ND
40	ND	21	59	12	90	11	24	26	1
41	6	20	47	7	91	9	55	30	ND
42	5	20	23	4	92	8	46	17	ND
43	ND	23	53	4	93	18	34	30	ND
44	13	63	34	9	94	ND	67	29	ND
45	ND	18	69	28	95	5	17	29	28
46	ND	15	59	32	96	10	34	30	39
47	18	17	55	17	97	19	13	30	12
48	ND	46	50	ND	98	13	24	28	ND
49	ND	19	14	ND	99	18	24	27	ND
50	7	17	57	4	100	13	9	27	15

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