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ORIGINAL PAPER

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Monitoring of Total Pb, As, Cd, Se, and Cr as Trace Pollutants in Sewage Sludge of Riyadh Second Industrial City by ICP-MS

Abstract. The concentration levels of Pb, As, Cd, Se, and Cr in sewage sludge of the Riyadh second industrial city environment were monitored using ICP-MS. Samples were collected from five different reservoirs three times a day for a period of four weeks during the summer season. The microwave acid digestion method was developed for sample preparation. Sample reference material was analyzed ten times to determine the precision and accuracy of the method. Better results were obtained using external standard calibration with the addition of Yttrium (Y) as an internal standard. Relative standard deviation ranged between 1-10% for all elements, with percentage recovery values ranging between 99%-106%. The concentrations of the elements obtained with the proposed method were in good agreement with the given certified values.

Keywords: Sewage sludge, Riyadh, industrial city, pollutants, monitoring, ICP-MS, deviation range.

مراقبة المحتوى الكلي للرصاص والزرنيخ والكادميوم والسلينيوم والكروم كملوثات في مياه الصرف الصحي بالمدينة الصناعية الثانية بمنطقة الرياض بطريقة البلازما مزدوجة الحث مطياف الكتلة.

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المستخلص: تهتم هذه الدراسة بمراقبة مستويات تراكيز عناصر الرصاص والزرنيخ والكادميوم والسلينيوم والكروم في المخلفات الصلبة لمياه الصرف الصحي للمدينة الصناعية الثانية بمنطقة الرياض. جمعت العينات من خمس خزانات مختلفة (نقاط تجمع) ثلاث مرات في اليوم ولمدة أربعة أسابيع خلال فصل الصيف. و تم تحليل عينة قياسية مرجعية عشر مرات لتحديد دقة ومصداقية الطريقة، مع إضافة عنصر اليتريوم كعنصر تقييس داخلي. أعطت النتائج المتحصل عليها، قيما للانحراف المعياري النسبي المئوي بين 1 – 10% لكل العناصر وفي كل العينات، وقيما لنسب الاسترجاع المئوية بين 99%–106%. وقد وجد أن تراكيز العناصر المحللة متوافقة إحصائياً مع القيم القياسية.

كلمات مدخلية: مياه الصرف الصحي، الرياض، المدينة الصناعية، ملوثات، مراقبة، البلازما، إنحراف معياري.

Introduction

Riyadh's second industrial city is one of the biggest industrial cities in the Kingdom of Saudi Arabia. It is located on an area of 16 million square meters, southeast of metropolitan Riyadh, and about 18 kilometers from the city center. The city houses more than 500 factories producing food, textiles, furniture, printed paper, chemical products, plastic molding, steel, metals, buses, car frames, electrical systems, etc. The industrial city has been designed to fulfil infrastructure requirements of the industries, such as a water supply system, storm water drainage system, sewage sludge treatment plant and sewage sludge collection system (RED 1998).

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Determinations of toxic elements such as cadmium, lead, arsenic, selenium and chromium in sewage sludge is necessary due to their serious threat to human health and environment (Dezuane 1980). Cadmium is considered to be a toxic element. The source of cadmium in sewage sludge may be from metallurgical alloying, textile printing and plastic stabilizer or pigment industries (Christensen *et al.* 1982). The level of lead in blood is a toxicological parameter. When the level reaches 100-120μg/dl, lead may cause irreversible brain damage (Dezuane, 1980). Lead is widely used in industry as a raw material for storage batteries, matches, pigments, photographic material, leaded glass, among others, that can contaminate the sewage sludge (Katz *et al.* 1981). Arsenic has a poisonous effect on humans at levels of 100mg or more, and is considered lethal at levels of 130mg or more (Dezuane, 1980). The toxicity of arsenic varies widely, depending on the compound. Lethal doses for some arsenic compounds are 1.5, 5, 50 and 500mg/kg for arsenite, arsenate, monomethlyarsonate and dimethylarsinate, respectively (Patterson, 1985). Many poisoning accidents have been reported due to arsenic contamination of food (18,500 people were infected and there were more than 200 fatalities in England and Japan) (Mezey, 1979, Johnson and Thompson *et al.* 1980). Sewage sludge can be contaminated by arsenic from the contribution of waste from metallurgical industries or wood and metal furniture industries (Christensen *et al.* 1982). Selenium is toxic at high levels, while it is essential to the human body at some levels (animal studies have confirmed its effectiveness in prevention of certain endemic disease) (Dezuane, 1980). Sewage sludge streams of paint factories, paper manufacturing, and pigment and dye formulating industries could contain selenium . Trivalent chromium may be nutritionally essential with a safe and relative innocuous level of 0.20 mg/day (Dezuane, 1980). Hexavalent chromium has an adverse effect on the liver, kidney and respiratory organs with symptoms such as bleeding effects, dermatitis, and ulceration of the skin from chronic and subchronic exposure (Dezuane, 1980). A toxic dose for a human adult was reported at about 0.5g of potassium dichromate ($K_2Cr_2O_7$). Chromium can be present in industrial waste streams due to chromium compounds used in industrial cooling water as corrosion inhibitors. Also it may be present in the waste stream of ink, paint pigment, as well as in metal-plating industries where chromic acid rinse water is used (Christensen *et al.* 1982).

Various sample digestion procedures for sewage were evaluated (Thompson *et al.* 1980, Christensen *et al.* 1982 and Katz *et al.* 1981). Microwave technique has been used for sewage sludge sample preparation (Vela *et al.* 1993, Morales *et al.* 1989, Jallan *et al.* 1989, Bettinelli *et al.* 1990 and Thomaidis *et al.* 1995). Some work has been done to compare the conventional method with the microwave digestion method (Moral *et al.* 1996, Fournier *et al.* 1997 and Florian *et al.* 1998). Also some authors have reported the use of ultrasonic bath for the digestion of sewage sludge using aqua regia (Sanchez *et al.* 1994).

Experimental

• Instrumentation

The inductively coupled plasma mass spectrometer model ELAN 6000 from PE SCIEX was used. A Pentium 133 MHz digital computer was used to control the instrument control and data acquisition, manipulation and storage. Results were printed using an HP LaserJet printer. A peristaltic pump was used for sample introduction and an auto sampler model AS 90 from Perkin Elmer was used for auto sample transport. Samples were nebulized using a PE cross flow nebulizer. The operating conditions for the ICP-MS were set as listed in Table 1.

A Fisher Scientific centrifuge apparatus was used for sample centrifuging. The microwave sample preparation system that was used for sewage sludge sample preparation was CEM Corporation model MDS-2100, USA. Teflon-lined digestion vessels were used in the microwave digestion system. A Fisher Scientific isotemp oven model 655G, USA, was used for the drying process.

Table 1. Operating conditions of ELAN 6000 ICP-MS

Nebulizer gas flow	0.8 L/min	Sweeps/readings	10
Lens voltage	9.0V	Integrated time	2000 ms
Analog stage voltage	1000V	Interface pressure	1-2 torr
Pulse stage voltage	-2100V	Mass spec pressure	1.06×10^{-5}
Discriminator threshold	70	Nebulizer	Cross flow
AC rod offse	-5	Sampler	nickel
Number of replicates	3	Skimmer	nickel
Readings/replicates	2	Dwell time	100ms
Scan mode	Peak hopping		

• Reagents

Nitric acid, Aristar grade 69%-71% from BDH Laboratory supplies, England was used for liquid sample preservation. Nitric acid, Certified A.C.S plus grade from Fisher was used for glassware and plastic bottle cleaning. Certified standard (Claritas ppt) from SPEX certiprep, Inc., NJ, USA, lot No. 13-01AS, containing 10mg/L in 5% (v/v) HNO_3 was used for standard preparation containing the following elements: As, Cd, Cr, Pb, Se. 10mg/L in 2% (v/v) HNO_3 Yttrium single element Certified standard, from (Claritas ppt), SPEX Certiprep, Inc., NJ, USA, lot No. 9-92AS, was used as an internal standard. Four standards, S1, S2, S3 and S4, containing 5, 50, 75 and 100mg/L respectively were prepared using the dilution procedure from the stock solution. For recovery, reproducibility, accuracy and precision studies, one sewage sludge sample was taken and spiked with 20mg/L of As, Pb, Cd, Se and Cr standard. The same sample was prepared and analyzed without any standard spiking. This step was repeated ten times to get representative data. The average concentration, standard deviation, relative standard deviation and recovery were calculated and tabulated. Deionized water obtained from compact Milli-Q UV plus system, Millipore, USA, with an indicated outlet conductivity of 18M/ohm was used.

• Samples

Four batches (B_1-B_4) of sewage sludge samples were collected every week for one month on different days, the first week on Saturday, the second week on Monday, the third week on Wednesday and the fourth week on Friday, from five reservoirs (R_1-R_5) located in different areas. Each batch was collected at different times of the day; the first sampling time at 09:00 hours, the second sampling time at 16:00 hours and the third sampling time at 23:00 hours. Samples were collected using polyethylene bottles. For cleaning, bottles were soaked in 20% (v/v) nitric acid for at least 24 hours then rinsed with deionized water for three times prior to use; moreover, bottles were rinsed several times with the sample prior to the sampling process.

• Procedure

Sewage sludge samples were transferred immediately after collection to the laboratory. Samples were filtered using a vacuum, buchner funnel and Whatmann filter paper No. 542. Filters including samples were then transferred to an oven and dried at 110°C for 2 hours. 50±10 mgs of sample were taken in a Teflon vessel and 10ml nitric acid (50% v/v) was added. Samples were then placed in the microwave digestion system. Table 2 illustrates the program used for the microwave digestion method. The solution was then transferred to a 50ml polypropylene centrifuge and stored in a refrigerator at 4°C prior to analysis (according to the standard method for the examination of water and sewage sludge) (APHA, 1992). A blank was treated the same as the sample. Internal standard (Yttrium) was added prior to the analysis. Samples were diluted several times if dilution was required.

Table 2. The microwave oven program.

Stage	1	2	3	4
Power (%)	60	80	80	90
Pressure (PSI)	40	80	120	150
Time (min.)	10	10	20	20
TAP[a]	3	10	10	10
Fan Speed	100	100	100	100

For recovery, reproducibility, accuracy and precision studies, sewage sludge certified reference material was analyzed ten times. The average concentration, standard deviation, relative standard deviation and recovery data were calculated, tabulated and compared to the certified data. Standards were prepared directly from the stock solution using the dilution method.

• Calibration

Mass calibration and resolution checks were conducted for the ICP-MS using a tuning solution contain 20mm/L of Mg, Pb, Rh, Ce and Ba obtained from Perken Elemer, USA. The instrument was calibrated using blank (deionized water) and four standards, S1, S2, S3 and S4, containing 5, 50, 75 and 100mg/L respectively. Table 3 illustrates the isotopes used in this methods, mass interferences and elemental equations for data calculations.

Table 3. Element isotopes and elemental equation for interference correction

Element	Mass	Interferences	Elemental equation	Note
As	75	ArCl	mass 75-(3.127)[(mass77)–(0.815)(mass 82)]	(1)
Se	82	Kr	mass 82 – 1.0082 x mass 83	(2)
Pb	207		mass 207	
Cd	111	MoO	mass111–(1.073)[(mass108)-(0.712)(mass106)]	(3)
Cr	52		mass 52	

Note:
(1) Correction for chloride interference with adjustment for Se^{77}.
(2) Correction for Kr^{82} interference.
(3) Correction for MoO interference.

Results and Discussion

The calibration curves of all elements obtained by the instrument using five points 0, 5, 50, 75 and 100mg/L showed an excellent linearity for all elements. Correlation coefficient values for all elements were within the 0.9987 to 0.9999 range. Table 4 illustrates the percentage recovery, standard deviation and relative standard deviation.

Table 4. Statistical data for all elements

Elements	Sample (mg/L)			Sample + 20 mg/L Spike			%Recovery
	AVG[a]	SD[b]	RSD[c]	AVG[a]	SD[b]	RSD[c]	
Cr	1.35	0.15	11.1%	21.47	0.44	2.1%	100.6%
As	1.68	0.16	9.8%	20.67	0.44	2.1%	94.9%
Cd	0.00	0.00	0.0%	18.74	0.27	1.4%	93.7%
Pb	6.14	0.60	9.8%	26.47	0.93	3.5%	101.6%
Se	6.09	0.57	9.3%	24.90	0.60	2.4%	94.1%

[a]Ten replicates.
[b]Standard deviation.
[c]Relative standard deviation.

The results obtained for sewage sludge reference material in comparison to the certified values indicate that the microwave digestion with nitric acid procedure is a good method. The relative standard deviation calculated for 10 replicates rangeed between 15% to 10% for all elements, except for selenium, which had a higher relative standard deviation. This may be due to the low concentration level of selenium and the high detection limit of the instrument. On the other hand, and disregarding the selenium value, percentage recovery, which ranged between 99% to 106% indicates the very good precision of this method.

Table 5 illustrates the chromium concentration levels in the sewage sludge during the study periods.

Table 5. Distribution of arsenic concentration in sewage sludge at different reservoirs

	[a]Mean conc. ± [b]S.D. (mg/L)				
Period	R1	R2	R3	R4	R5
[c]B1-1[d]	< 0.09	12.0 ± 1	10.7 ± 0.8	18.9 ± 0.3	5.7 ± 0.1
B1-2[d]	2.3 ± 0.2	16.4 ± 0.6	7.7 ± 0.2	28.9 ± 0.3	14.9 ± 1.1
B1-3[d]	2.0 ± 0.3	9.6 ± 1.0	8.9 ± 0.2	20.4 ± 0.2	19.5 ± 0.6
[c]B2-1	2.2 ± 0.01	11.7 ± 0.3	10.1 ± 0.4	20.1 ± 0.5	20.2 ± 0.7
B2-2	5.0 ± 0.1	12.0 ± 0.3	10.2 ± 0.3	16.9 ± 0.7	17.9 ± 0.4
B2-3	5.2 ± 0.1	10.7 ± 0.7	11.4 ± 0.2	15.8 ± 0.6	17.5 ± 0.3
[c]B3-1	2.2 ± 0.1	9.3 ± 0.2	11.1 ± 0.2	15.2 ± 0.5	17.9 ± 1.0
B3-2	4.3 ± 0.1	9.6 ± 0.3	11.2 ± 0.2	13.2 ± 0.8	14.3 ± 0.3
B3-3	5.8 ± 0.2	8.9 ± 0.6	10.9 ± 0.5	13.7 ± 0.3	16.8 ± 0.4
[c]B4-1	7.3 ± 0.4	38.9 ± 1.9	10.4 ± 0.1	15.7 ± 0.4	11.8 ± 0.4
B4-2	8.5 ± 0.2	17.0 ± 0.2	11.0 ± 0.3	13.4 ± 0.9	31.3 ± 0.6
B4-3	9.5 ± 0.1	11.1 ± 0.5	11.1 ± 0.4	13.5 ± 0.5	29.3 ± 0.7

[a]Three replicates
[b]Standard deviation
[c]B_1 first week on Sat., B_2 2[nd] week on Mon., B_3 3[rd] week on Wed, B_4 4[th] week on Fri.
[d](1) at 9:00 hours, (2) at 16:00 hours, (3) at 23:00 hours.

The concentration levels, in general, were below 2% (w/w) except for two occasions of the last week, when the level of chromium exceeded 11% (w/w) (B_4-R_2-1 and 2). Moreover, in the third and fourth week of reservoir R_5 the concentration level was around 6% (w/w). These high concentration levels may be due to the high number of metal manufacturing factories located in that area. Due to many glass factories, arsenic concentration levels were less than 5mg/g most of the time; however, the level of arsenic exceeded 30mg/g at one time (B_1-R_2-3) (Table 6).

Table 6. Distribution of chromium concentration in sewage sludge at different reservoirs

	[a]Mean conc. ± [b]S.D. (mg/L)				
Period	R1	R2	R3	R4	R5
[c]B1-1[d]	2.7 ± 0.3	6.3 ± 0.2	3.9 ± 0.3	341.0 ± 6.0	37.9 ± 0.7
B1-2[d]	1929.0 ± 20.0	17.2 ± 0.2	14.1 ± 0.8	260.0 ± 4.0	341.0 ± 4.0
B1-3[d]	1275.0 ± 21.0	64.1 ± 1.1	23.4 ± 0.6	379.0 ± 8.0	403.0 ± 3.0
[c]B2-1	11.4 ± 0.3	29.3 ± 0.1	25.4 ± 0.8	124.0 ± 1.1	130.0 ± 0.9
B2-2	15.4 ± 0.3	51.1 ± 0.6	46.5 ± 0.4	96.5 ± 2.2	101.0 ± 0.9
B2-3	57.4 ± 0.2	59.1 ± 0.5	131.0 ± 1.9	92.8 ± 1.6	141.0 ± 0.8
[c]B3-1	634.0 ± 7 3	6.6 ± 0.3	11.1 ± 0.2	153.0 ± 1.3	362.0 ± 1.6
B3-2	419.0 ± 0.7	57.6 ± 1.7	22.4 ± 0.3	103.0 ± 1.6	138.0 ± 1.0
B3-3	343.0 ± 0.5	76.9 ± 0.7	27.6 ± 0.2	138.0 ± 1.4	272.0 ± 6.0
[c]B4-1	9.2 ± 0.2	203.0 ± 1.4	21.3 ± 0.1	152.0 ± 1.3	13.6 ± 0.2
B4-2	9.2 ± 0.2	10.3 ± 0.2	22.2 ± 0.2	94.6 ± 1.0	1249.0 ± 7.0
B4-3	9.6 ± 0.3	8.8 ± 0.2	18.2 ± 0.2	131.0 ± 1.0	883.0 ± 3.0

[a]Three replicates.
[b]Standard deviation.
[c]B_1 first week on Sat., B_2 2[nd] week on Mon., B_3 3[rd] week on Wed, B_4 4[th] week on Fri.
[d](1) at 9:00 hours, (2) at 16:00 hours, (3) at 23:00 hours.

Many metallurgical alloying industries as well as textile printing industries can lead to high cadmium levels in the waste stream leading to a high accumulation in the sewage sludge. As illustrated in Table 7, cadmium concentration levels were not stable; in three reservoirs (R_1, R_3, R_5) the levels were less than 5mg/g on average but sometimes the 15 and 20mg/g concentration levels were reached in reservoir R_3. The concentration levels in R_4 were 15mg/g on average, while R_2 had the highest concentration level, sometimes reaching 30mg/g.

Table 7. Distribution of lead concentration in sewage sludge at different reservoirs

Period	[a]Mean conc. ± [b]S.D. (mg/L)				
	R1	R2	R3	R4	R5
[c]B1-1[d]	< 0.4	4.9 ± 0.3	< 0.4	< 0.4	< 0.4
B1-2[d]	12.8 ± 0.2	9.3 ± 0.1	< 0.4	6.1 ± 0.10	< 0.4
B1-3[d]	4.7 ± 0.4	81.2 ± 1.1	7.2 ± 0.2	15.0 ± 0.10	< 0.4
[c]B2-1	< 0.4	104.0 ± 1.0	71.2 ± 1.6	350.0 ± 5.00	< 0.4
B2-2	< 0.4	16.9 ± 0.1	13.9 ± 0.2	157.0 ± 3.30	< 0.4
B2-3	< 0.4	37.8 ± 0.7	10.5 ± 0.3	98.5 ± 1.40	0.6 ± 0.1
[c]B3-1	< 0.4	273.0 ± 4.0	15.3 ± 0.1	1.0 ± 0.10	< 0.4
B3-2	< 0.4	257.0 ± 3.0	19.3 ± 0.2	0.1 ± 0.02	< 0.4
B3-3	1.4 ± 0.1	225.0 ± 3.2	15.8 ± 0.2	4.8 ± 0.20	< 0.4
[c]B4-1	< 0.4	< 0.4	32.4 ± 0.5	< 0.4	30.7 ± 0.3
B4-2	< 0.4	1.9 ± 0.1	12.5 ± 0.7	< 0.4	< 0.4
B4-3	< 0.4	< 0.4	16.3 ± 0.3	< 0.4	< 0.4

[a]Three replicates.
[b]Standard deviation.
[c]B_1 first week on Sat., B_2 2[nd] week on Mon., B_3 3[rd] week on Wed, B_4 4[th] week on Fri.
[d](1) at 9:00 hours, (2) at 16:00 hours, (3) at 23:00 hours.

Table 8 illustrates the concentration levels in the sludge, which were below 1000mg/g in all reservoirs and generally averaged between 200 to 800mg/g.

Table 8. Distribution of cadmium concentration in sewage sludge at different reservoirs

Period	[a]Mean conc. ± [b]S.D. (mg/L)				
	R1	R2	R3	R4	R5
[c]B1-1[d]	<0.80 ± 0.10	0.50 ± 0.02	< 0.03	< 0.03	< 0.03
B1-2[d]	5.70 ± 0.10	< 0.03	< 0.03	< 0.03	< 0.03
B1-3[d]	< 0.03	< 0.03	< 0.03	<0.03	< 0.03
[c]B2-1	1.80 ± 0.01	8.20 ± 0.10	1.40 ± 0.1	< 0.03	< 0.03
B2-2	< 0.03	1.80 ± 0.10	0.20 ± 0.01	0.10 ± 0.06	< 0.03
B2-3	< 0.80 ± 0.10	4.30 ± 0.03	<0.03	0.10 ± 0.02	0.10 ± 0.03
[c]B3-1	28.30 ± 0.50	< 0.03	< 0.03	< 0.03	< 0.03
B3-2	16.00 ± 0.30	< 0.03	< 0.03	< 0.03	< 0.03
B3-3	9.90 ± 0.20	< 0.03	< 0.03	< 0.03	< 0.03
[c]B4-1	2.70 ± 0.02	1.20 ± 0.01	< 0.03	< 0.03	< 0.03
B4-2	1.30 ± 0.02	1.30 ± 0.03	< 0.03	< 0.03	< 0.03
B4-3	1.80 ± 0.01	< 0.03	< 0.03	< 0.03	< 0.03

[a]Three replicates.
[b]Standard deviation.
[c]B_1 first week on Sat., B_2 2[nd] week on Mon., B_3 3[rd] week on Wed, B_4 4[th] week on Fri.
[d](1) at 9:00 hours, (2) at 16:00 hours, (3) at 23:00 hours.

Lead concentration could be from battery industries, metallurgical manufacturing or from photographic material manufacturing. Selenium results, as illustrated in Table 9, were not reproducible and the standard deviations were in most cases slightly high, indicating poor sensitivity and stability. However, the average concentration levels of Selenium in the sludge were below 15mg/g, except for reservoir R_4, where the level was near 27mg/g in some periods. Selenium is an important element in paper and pulp, as well as manufacturing, and it could be a water pollutant, which would explain this accumulation in the sewage sludge.

Table 9. Distribution of selenium concentration in sewage sludge at different reservoirs

	[a]Mean conc. ± [b]S.D. (mg/L)				
Period	R1	R2	R3	R4	R5
[c]B1-1[d]	<0.5	2.9 ± 0.4	1.0 ± 0.2	12.6 ± 0.7	0.6 ± 0.1
B1-2[d]	0.7 ± 0.2	4.2 ± 1.6	< 0.5	12.5 ± 2.9	2.6 ± 1.6
B1-3[d]	< 0.5	1.7 ± 0.7	< 0.5	11.1 ± 0.6	< 0.5
[c]B2-1	< 0.5	5.1 ± 0.8	4.8 ± 0.3	11.6 ± 1.2	5.2 ± 1.2
B2-2	2.9 ± 1.1	4.4 ± 1.2	4.8 ± 1.1	10.6 ± 1.2	6.3 ± 0.6
B2-3	1.5 ± 0.6	6.2 ± 0.6	5.2 ± 0.9	9.3 ± 0.4	7.0 ± 0.9
[c]B3-1	< 0.5	5.4 ± 1.0	8.3 ± 0.9	16.9 ± 0.5	1.8 ± 0.5
B3-2	2.6 ± 0.6	5.0 ± 1.0	6.1 ± 1.7	10.9 ± 0.5	3.1 ± 0.4
B3-3	2.1 ± 0.9	5.6 ± 0.6	6.4 ± 0.6	8.2 ± 0.1	2.3 ± 0.5
[c]B4-1	4.9 ± 1.1	< 0.5	1.7 ± 1.1	4.7 ± 0.9	4.8 ± 1.2
B4-2	6.0 ± 1.3	7.7 ± 1.1	3.9 ± 1.3	6.4 ± 0.9	< 0.5
B4-3	6.9 ± 0.7	3.7 ± 1.1	2.6 ± 1.9	5.8 ± 0.9	< 0.5

[a]Three replicates.
[b]Standard deviation.
[c]B_1 first week on Sat., B_2 2nd week on Mon., B_3 3rd week on Wed, B_4 4th week on Fri.
[d](1) at 9:00 hours, (2) at 16:00 hours, (3) at 23:00 hours.

Table 10. The maximum allowable limits of element concentration in the sewage sludge controlled by the Royal Commission of Jubail and Yanbu (RCJY)

Elements	As	Cr	Pb	Cd	Se
Concentration limits (mg/L)	1250	5000	500	500	500

Conclusion and Recommendations

The microwave digestion method with the addition of Yttrium as an internal standard for the matrix effect correction, followed by ICP-MS analysis, proves to be a very powerful, advanced, rapid and precise technique for digestion and analysis of elements in industrial sewage sludge. Results obtained from the certified reference material of the sludge using 50% (w/v) nitric acid and the proposed microwave digestion method shows very good recoveries (99% to 106%) of several metals of environmental interest and can easily be applied as routine in laboratories. It is concluded that the suspended elements are accumulating in those reservoirs by time. Therefore, it is highly recommended to clean those reservoirs in a periodic manner and dispose of the collected sludge in a proper way.

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