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Monitoring of Some Trace Pollutants in Wastewater of Riyadh Second Industrial City by ICP-MS

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Abstract. The concentration levels of Pb, As, Cd, Se, and Cr in wastewater of 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. Precision and accuracy study for the analysis of industrial wastewater were obtained by spiking the samples with the elements of interest and percentage recoveries for those elements were calculated. Results were obtained using external standard calibration with the addition of Yttrium (Y) as internal standard. The percentage recovery for all elements are ranging between 93% to 102% and the relative standard deviation for almost all elements in the samples were ranging between 0 to 12%. Results were calculated and compared with the maximum allowable concentrations ruled by the Royal Commission of Jubail and Yanbo (RCJY) at Saudi Arabia.

Keywords: Wastewater, trace elements, pollutants, ICP-MS

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 at the southeast of Riyadh metropolitan, about 18 kilometers from the city center. There are more than 500 factories producing food, textiles, furniture, paper printing, chemical products, plastic molding, steel, metals, buses, car frames, electrical systems, etc. The industrial city has been designed to fulfil the requirements of the industrial services such as water supply system, storm water drainage, wastewater treatment plant and wastewater collection system [1, p. 17].

The determinations of toxic elements such as cadmium, lead, arsenic, selenium and

chromium in wastewater is necessary due to their serious threat to human health and environment [2, p. 19]. Cadmium is considered as a toxic element, the source of cadmium in wastewater may be from metallurgical alloying, textile printing and plastic stabilizers or pigment industries [3, p. 9]. Lead level in blood is a toxicological parameter, when the level reaches 100-120 μ g/dl it will cause irreversible brain damage [2, p. 19]. Lead is widely used in industry as a raw material for storage batteries, matches, pigments, photographic material, leaded glass and many other uses, that can contaminate the wastewater [4]. Arsenic has a poisonous effect in human at 100 mg levels or more, and considered lethal at 130 mg levels [2, p. 25]. The toxicity of arsenic varies widely, depending on the compound, lethal doses for some arsenic compounds are 1.5, 5, 50 and 500 (mg/kg) for arsenite, arsenate, monomethylarsonate and dimethylarsinate respectively [4]. Many poisoning accidents were reported due to arsenic contamination of food (18,500 people were infected and more than 200 fatalities in England and Japan) [5-7]. Wastewater can be contaminated by arsenic from the contribution of waste from metallurgical industry or wood and metal furniture industry [3, p. 30]. 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) [2, 32]. Wastewater stream of paint factories, paper manufacturing, pigments and dye formulating industries could contain Selenium [8, p. 4]. Trivalent chromium may be nutritionally essential with a safe and relative innocuous level of 0.20 mg/day [2, p. 40]. 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 for chronic and sub-chronic exposure [2, p. 77]. A toxic dose for a man was reported at about 0.5g of potassium dichromate ($K_2Cr_2O_7$) [2, p. 79]. Chromium can be present in industrial waste stream due to chromium compounds used in industrial cooling water as corrosion inhibitors and also present in the waste stream of ink, paint pigments, as well as in metal-plating industries where chromic acid rinse water is used [3, p. 80]. A number of studies have been done to measure toxic elements concentration in either natural water [8-10], river water [11,12] or wastewater [12-14].

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 a 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 samples transport. Samples were nebulized using 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.

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Table 1. Operating conditions	OI ELAN 0000 ICF-MG		
Nebulizer Gas Flow	0.8 L/min	Sweeps/Readings	10
Lens Voltage	9.0 V	Integrated Time	2000 ms
Analog Stage Voltage	1000 V	Interface pressure	1-2 torr
Puls Stage Voltage	-2100 V	Mass spec. pressure	1.06×10 ⁻⁵
Discriminator Threshold	70	Nebulizer	Cross flow
AC Rod Offset	-5	Sampler	nickel
Number of Replicates	3	Skimmer	nickel
Readings/Replicates	2	Dwell Time	100 ms
Scan Mode	Peak Hopping		

Table 1. Operating conditions of ELAN 6000 ICP-MS

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 bottles cleaning. Certified standard (Claritas ppt) from SPEX certiprep, Inc., NJ, USA lot No. 13-01AS containing 10 mg/L in 5% (v/v) HNO₃ was used for standard preparation containing the following elements: As, Cd, Cr, Pb, Se. 10 mg/L in 2% (v/v) HNO₃. 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 contain 5, 50, 75 and 100 ug/L, respectively were prepared using the dilution procedure from the stock solution. For recovery, reproducibility, accuracy and precision studies, one wastewater sample was taken and spiked with 20 µg/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 a 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 18 M/ohm was used.

Samples

Four batches (B_1 - B_4) wastewater samples were collected every week for one month on different days, first week on Saturday, second week on Monday, third week on Wednesday and fourth week on Friday, from five reservoirs (R_1 - R_5) located in different areas. Each batch was collected at different time periods of a day, first sampling time at 09:00 o'clock, second sampling time at 16:00 o'clock and third sampling time at 23:00 o'clock. 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 the sampling process.

Procedure

Samples were transferred immediately after the collection to the laboratory,

centrifuged at speed of 6000 RPM for 10 minutes, filtered using vacuum, buchner funnel and Whatmann filters paper No. 542. Furthermore, samples were filtered through a 0.45 μ m cellulose acetate membrane prior to the addition of preserving acid (69% (v/v) HNO₃ 5ml/1L). Samples were then stored in a 50-ml polypropylene in a refrigerator at 4°C prior to analysis (according to the standard method for the examination of water and wastewater) [15]. A blank was treated same as the sample.

Calibration

Mass calibration and resolution checks were conducted for the ICP-MS using a tuning solution contain 20 μ m/L of Mg, Pb, Rh, Ce and Ba obtained from Perkin Elemer, USA. The Instrument was calibrated using blank (deionized water) and four standards S1, S2, S3 and S4 containing 5, 50, 75 and 100 μ g/L respectively. Table 2 illustrates the isotopes used in this method, mass interferences and elemental equations for data calculations.

	Table 2. Elements isoto	pe and elemental	equation for	r interference	correction
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Element	Mass	Interferences	Elemental equation	Note
As	75	ArCl	mass75-(3.127)[(mass77)-(0.815)(mass82)]	(1)
Se	82	Kr	mass82 - 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 Kr⁸² 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 100 μ g/L shows an excellent linearity for all elements. Correlation coefficient values for all elements were within 0.9987 to 0.9999 range. Table 3 illustrates the percentage recovery, standard deviation and relative standard deviation. It is observed that the relative standard deviation for almost all elements in the samples is ranging between 0 to 12%. This is strong evidence that this method is precise and reproducible. Furthermore, the percentage recovery for all elements are ranging between 93% to 102% which is an indication that this method is accurate (accepted limits for known addition to water and wastewater (%recovery) for metals is ranging between 80 to 120% [15, p. 80]).

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Elements	Sample (µg/L)			Samp	Sample + 20 µg/L Spike			
	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%	

Table 3. Statistical data for all elements

^aTen replicates.

^bStandard deviation ^cRelative standard deviation

Relative standard deviation

Table 4 illustrates the concentration levels of arsenic which is ranging between 2 to 30 μ g/L except for (B₄- R₂-1) which is higher (around 40 μ g/L) due to many glass factories. Table 5 illustrates the chromium concentration levels in the wastewater were below 500 μ g/L except for four cases where the level of chromium exceeded 2000 $\mu g/L$ at the morning period of the first week (B₁-R₁-1) then at the same day during the afternoon period, the level was down to less than $1500 \ \mu g/L$ then back to normal level $[< 500 \ \mu g/L]$ during the night period. The same behavior was observed in (B₄-R₅-2) and (B₄-R₅-3). These high concentration levels may be due to high number of metal manufacturing factories located in that area. Lead concentration levels in wastewater are below 100 μ g/L and generally close to zero (Table 6). However, high levels were observed (over 300 μ g/L) in the third week at reservoir four in the morning period (B₃- R_{4} -1) then went down in the afternoon and night periods due to the continuous wastewater flow passing from this reservoir to the wastewater treatment plant. The same behavior was observed at the second reservoir during the third week (B_3-R_2) . The same situation was observed in case of cadmium in the third week and during the morning period in the first reservoir (B_3-R_1-1) , a high amount of cadmium, over 25 μ g/L, was detected then the level went down during the afternoon and night periods. But the average concentration levels in the wastewater were generally less than 5 μ g/L as illustrated in Table 7. Lead and cadmium contamination could be from battery industries, metallurgical manufacturing or from photographic materials manufacturing located in that area. Selenium results, as illustrated in Table 8 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 wastewater were below 8 μ g/L, except for reservoir 4 (R₄) the level was near 15 μ g/L. Selenium is an important element in paper and pulp processes as well as in glass manufacturing.

Table 4. Distribution of arsenic concentration in wastewater at different reservoirs									
		^a Mean conc. ± ^b S.D. (µg/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	8.9 ± 0.2	20.4 ± 0.2	19.5 ± 0.6				
°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				
°B3-1	2.2 ± 0.1	9.3 ± 0.2	11.1 ± 0.2	15.2 ± 0.5	17.9 ± 1				
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				
°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				

^aThree replicates ^bStandard deviation

^cB₁ first week on Sat., B₂ 2nd week on Mon., B₃ 3rd week on Wed, B₄ 4th week on Fri. ^d(1) at 9:00 o'clock, (2) at 16:00 o'clock, (3) at 23:00 o'clock.

	^a Mean conc. \pm ^b S.D. (µg/L)						
Period	R1	R2	R3	R4	R5		
^c B1-1 ^d	2.7 ± 0.3	6.3 ± 0.2	3.9 ± 0.3	341 ± 6	37.9 ± 0.7		
B1-2 ^d	1929 ± 20	17.2 ± 0.2	14.1 ± 0.8	260 ± 4	341 ± 4		
B1-3 ^d	1275 ± 21	64.1 ± 1.1	23.4 ± 0.6	379 ± 8	403 ± 3		
°B2-1	11.4 ± 0.3	29.3 ± 0.1	25.4 ± 0.8	124 ± 1.1	130 ± 0.9		
B2-2	15.4 ± 0.3	51.1 ± 0.6	46.5 ± 0.4	96.5 ± 2.2	101 ± 0.9		
B2-3	57.4 ± 0.2	59.1 ± 0.5	131 ± 1.9	$92.8\ \pm 1.6$	141 ± 0.8		
°B3-1	634 ± 7	36.6 ± 0.3	11.1 ± 0.2	153 ± 1.3	362 ± 1.6		
B3-2	419 ± 0.7	57.6 ± 1.7	22.4 ± 0.3	103 ± 1.6	138 ± 1		
B3-3	343 ± 0.5	76.9 ± 0.7	27.6 ± 0.2	138 ± 1.4	272 ± 6		
°B4-1	9.2 ± 0.2	203 ± 1.4	21.3 ± 0.1	152 ± 1.3	13.6 ± 0.2		
B4-2	9.2 ± 0.2	10.3 ± 0.2	22.2 ± 0.2	94.6 ± 1	1249 ± 7		
B4-3	9.6 ± 0.3	8.8 ± 0.2	18.2 ± 0.2	131 ± 1	883 ± 3		

^aThree replicates ^bStandard deviation ^cB₁ first week on Sat., B₂ 2nd week on Mon., B₃ 3rd week on Wed, B₄ 4th week on Fri. ^d(1) at 9:00 o'clock, (2) at 16:00 o'clock, (3) at 23:00 o'clock.

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Table 6. Distribution of lead concentration in wastewater at different reservoirs								
	^a Mean conc. \pm ^b S.D. (µg/L)							
Period	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 ± 0.1	< 0.4			
B1-3 ^d	4.7 ± 0.4	81.2 ± 1.1	7.2 ± 0.2	15.0 ± 0.1	< 0.4			
°B2-1	< 0.4	104 ± 1	71.2 ± 1.6	350 ± 5	< 0.4			
B2-2	< 0.4	16.9 ± 0.1	13.9 ± 0.2	157 ± 3.3	< 0.4			
B2-3	< 0.4	37.8 ± 0.7	10.5 ± 0.3	98.5 ± 1.4	0.6 ± 0.1			
°B3-1	< 0.4	273 ± 4	15.3 ± 0.1	1.0 ± 0.1	< 0.4			
B3-2	< 0.4	257 ± 3	19.3 ± 0.2	0.1 ± 0.02	< 0.4			
B3-3	1.4 ± 0.1	225 ± 3.2	15.8 ± 0.2	4.8 ± 0.2	< 0.4			
°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			

^aThree replicates

^bStandard deviation

^cB₁ first week on Sat., B₂ 2nd week on Mon., B₃ 3rd week on Wed, B₄ 4th week on Fri. ^d(1) at 9:00 o'clock, (2) at 16:00 o'clock, (3) at 23:00 o'clock.

Table 7. Distribution of cadmium concentration in wastewater at different res	ervoirs

	^a Mean conc. ± ^b S.D. (µg/L)							
Period	R1	R2	R3	R4	R5			
°B1-1°	0.8 ± 0.1	0.5 ± 0.02	< 0.03	< 0.03	< 0.03			
B1-2 ^c	5.7 ± 0.1	< 0.03	< 0.03	< 0.03	< 0.03			
B1-3 ^c	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03			
°B2-1	1.8 ± 0.01	8.2 ± 0.1	1.4 ± 0.1	< 0.03	< 0.03			
B2-2	< 0.03	1.8 ± 0.1	0.2 ± 0.01	0.1 ± 0.06	< 0.03			
B2-3	0.8 ± 0.1	4.3 ± 0.03	< 0.03	0.1 ± 0.02	0.1 ± 0.03			
°B3-1	28.3 ± 0.5	< 0.03	< 0.03	< 0.03	< 0.03			
B3-2	16.0 ± 0.3	< 0.03	< 0.03	< 0.03	< 0.03			
B3-3	9.9 ± 0.2	< 0.03	< 0.03	< 0.03	< 0.03			
°B4-1	2.7 ± 0.02	1.2 ± 0.01	< 0.03	< 0.03	< 0.03			
B4-2	1.3 ± 0.02	1.3 ± 0.03	< 0.03	< 0.03	< 0.03			
B4-3	1.8 ± 0.01	< 0.03	< 0.03	< 0.03	< 0.03			

^aThree replicates

^bStandard deviation

^cB₁ first week on Sat., B₂ 2^{nd} week on Mon., B₃ 3^{rd} week on Wed, B₄ 4^{th} week on Fri. ^d(1) at 9:00 o'clock, (2) at 16:00 o'clock, (3) at 23:00 o'clock.

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		^a Mean conc. ± ^b S.D. (µg/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				
°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				
°B3-1	< 0.5	5.4 ± 1	8.3 ± 0.9	16.9 ± 0.5	1.8 ± 0.5				
B3-2	2.6 ± 0.6	5.0 ± 1	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				
°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				

^aThree replicates ^bStandard deviation

^cB₁ first week on Sat., B₂ 2nd week on Mon., B₃ 3rd week on Wed, B₄ 4th week on Fri.

^d(1) at 9:00 o'clock, (2) at 16:00 o'clock, (3) at 23:00 o'clock.

Conclusion and Recommendations

The internal standard (yttrium) addition method using ICP-MS instrument proved to be precise and reproducible for the determination of elements in wastewater. Furthermore, ICP-MS with its multi-element analysis capabilities and lower detection limits is time saving and ideal for monitoring the concentration levels of trace elements in wastewater. Moreover the concentration levels of elements of interest that have been monitored were conforming to the Royal Commission of Jubail and Yanbo (RCJY) in Saudi Arabia for the concentration levels of trace elements at the point of discharge of industrial wastewater and before the treatment. The maximum concentration levels found in the wastewater of As, Cr, Pb, Cd and Se are 40, 2000, 400, 30 and 20 μ g/L respectively and those levels considered to be less than the harmful levels as they were below the maximum allowable limits ruled by the Royal Commission of Jubail and Yanbo (RCJY) as listed in Table 9 [16, p. 119].

Table 9. The maximum allowable limits of the elements concentration in the wastewater ruled by the Royal Commission of Jubail and Yanbo (RCJY)

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

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مراقبة تراكيز بعض الملوثات في مياه الصرف الصحي بالمدينة الصناعية الثانية بمنطقة الرياض بطريقة البلازما مزدوجة الحث ومطياف الكتلة

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ملحص المحث.تمت خلال هذه الدراسة مراقبة مستويات تراكيز الرصاص، الزرنيخ، الكادميوم، السلينيوم والكروم في مياه الصرف الصحي في المدينة الصناعية الثانية بمنطقة الرياض بطريقة البلازما مزدوجة الحث ومطياف الكتلة. وتمّ جمع العينات من خمس خزانات مختلفة خلال ثلاث فترات في اليوم ولمدة أربعة أسابيع خلال فصل الصيف. ودرست دقة ومصداقية الطريقة باضافة كميات معلومة من العناصر المراد تحليلها مع وجود عنصر اليتريوم كعنصر تقييس داخلي، وكذلك تمّ إيجاد نسب الاسترجاع المئوية لكل عنصر . وقد تراوحت قيم نسب الاسترجاع المئوية في كل الحالات من ٩٣% - ١٠٢% وقيم الانحراف النسبي المئوي من صفر - ١٢%. كذلك تمت مقارنة النتائج المتحصل عليها مع التراكيز القصوى المسموح بها من قبل الهيئة الملكية للجبيل وينبع في المملكة العربية السعودية.