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Assessing sediment pollution from the Julian Adame-Alatorre dam by instrumental neutron activation analysis

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ABSTRACT

The rapid industrial development in regions of Mexico during recent years has had the side effect of introducing toxic metals, fertilizers, and pesticides into the ecosystem. Sediment cores were collected from eight locations around the Julian Adame-Alatorre dam located in the Municipality of Villanueva in the State of Zacatecas, México. The cores were analyzed for 32 major, trace, and rare earth elements (As, La, Lu, Nd, Sm, U, Yb, Ce, Co, Cr, Cs, Eu, Fe, Hf, Rb, Sb, Sc, Sr, Ta, Tb, Th, Zn, Zr, Al, Ba, Ca, Dy, K, Mn, Na, Ti, and V) in order to estimate the health risk. The samples were analyzed by instrument neutron activation analysis (INAA) using thermal neutron fluxes of 8×10^{13} and 5×10^{13} n cm^{-2} s^{-1} for short and long irradiations, respectively. The results of the contamination levels for elements such as As, Ba, Cr, Fe, Mn, Ta, V, and Zn were compared with the Mexican regulations and the guidelines of USEPA. Enrichment factors for quantified elements identified high As, Sb, Hf, and Cs contents using Fe as a crustal reference.

1. Introduction

The rapid industrial development in regions of México during recent years has had the side effect of introducing toxic metals, fertilizers, and pesticides into the ecosystem. Also, the demand for rare-earth elements (REEs) used in industrial applications is increasing rapidly; and concurrently the environmental contamination by REEs is occurring. The general public has great concerns about the environmental quality of the regions in which they are living and want to maintain a healthy life style [1,2]. When these pollutants are released into the environment, they enter the atmosphere, undergo hydrological circulation, and are finally deposited in riverbeds, reservoirs, and river deltas. Sediment is known as a sink for trace elements like heavy metals produced from anthropogenic sources such as the discharges of municipal, residential and industrial waste materials. Thus, sediments must be considered one of the environmental indicators. Much research has been conducted to investigate contamination levels of toxic metals in environmental sediment samples.

Individual countries have issued their own regulations stating the limits of concentrations of heavy metals or other pollutants that can be considered acceptable or dangerous. In México, these limits have been established by the Minister of the Environmental Protection and published in the Mexican Official Norm NOM-147-SEMARNAT/SSA1-2004. The expressed limits refer to soils and sediments used for agricultural, residential, commercial and industrial purposes [1,3]. They include both soils and sediments; but between these categories there are no differences. In this way, to be considered as a pollutant, any potentially harmful compound must satisfy simultaneously two criteria: (1) to present an increased concentration near the sediment surface and (2) to exceed the legal limits. In the present work, the results are compared to the limits of concentrations established by the Mexican Official Norm; and as well as to the concentration guidelines of USEPA in this matter [4].

The high-precision, nuclear quantitative analytical technique of instrumental neutron activation analysis (INAA) is currently used in the investigation of environmental samples such as sediments or soils due to the very high sensitivity. In the present work, this analytical technique was used to study the concentrations of major, trace, and rare-earth elements (REEs) in eight sedimentary cores that were collected from the Julian Adame-Alatorre dam, located in the Municipality of Villanueva in the State of Zacatecas, México. This study aimed at applying INAA for the analysis of eight sediment cores to uncover the existence of any pollution in Julian Adame-Alatorre dam for the elements in question [2,5]. The enrichment factors' (EF) values were calculated using the mean of element concentrations found in the eight sediment samples and the continental crust average to confirm the existence of pollution in sediments [6–8].

2. Experimental

2.1. Description of field area

The Unit of The Chique–Tayahua counts of two storage dams, the Chique, with 140×10^6 m^3 capacity, and the Julian Adame-Alatorre,

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with 38×10^6 m^3 capacity, are the main sources of water supply for the river of Juchipila. This unit is located in the southeastern part of the state of Zacatecas approximately 130 km from the capital city of Zacatecas. The geographic location of Julian Alatorre dam is at latitude 22° 07′ 12″ N and longitude 102° 51′ 49″ W and altitude 1722 amsl (Fig. 1). The Julian Adame-Alatorre dam is considerably influenced by mining industry. It is also affected by traditional and intensive agriculture. Water from the dam is used to satisfy the needs of intensive agriculture. Also, there is an aquaculture center that produces Golden tilapia (*Oreochromis aureus*) and Rocky Mountain Tilapia (*Oreochromis niloticus*×*O. aureus*).

2.2. Sampling and sample preparation

Sediment samples were collected from eight locations of Julian Adame-Alatorre dam during the spring season of 2003. The location of Julian Adame-Alatorre dam site is indicated in Fig. 1. Two samples were collected from each quadrant, in order to get representative samples. Eight samples at the sampling points were collected in accordance with the depths, which were taken from the upper 10 cm of the dam bottom by using a piston corer. The resulting cores with a diameter of 3.81 cm were stored in polyethylene bags [9,10]. The sediment samples were prepared into a finely ground homogenous material and the powdered sediment samples were dried for 24 h in an oven at 100 °C to remove any water present.

2.3. Instrumental neutron activation analysis

For INAA, two aliquots of powdered sediment samples weighing 150 mg each were placed in separate polyethylene vials and high-purity quartz vials used for short and long irradiations, respectively, at University of Missouri Research Reactor (MURR) of the University of Missouri-Columbia, USA. The irradiation and counting parameters utilized for the element analyses are reported in Table 1. The short-irradiation samples in polyvials were sequentially irradiated in a thermal neutron flux of 8.0×10^{13} n cm^{-2} s^{-1} for five seconds, allowed to decay for 25 min, and then counted for 12 min each on a high-purity intrinsic germanium (HP Ge) detector with 25% relative efficiency and FWHM resolution of 1.85 keV for the 1332 keV gamma ray of ^{60}Co [1,2,9,11]. Qualitative and quantitative analysis can be achieved by analysis of gamma-lines in the gamma-spectrum detected and registered by HP Ge detector and its associated electronic circuit.

Fig. 1. Map showing the location of Julian Adame-Alatorre dam site.

Table 1
Irradiation and counting conditions.

Elements and half-life	Irradiation flux (n cm^{-2} s^{-1})	Irradiation time	Decay time	Counting time
Short-lived elements: Al, Ba, Ca, Dy, K, Mn, Na, Ti, V	8×10^{13}	5 s	25 min	12 min
Medium-lived elements: As, La, Lu, Nd, Sm, U, Yb	5×10^{13}	24 h	7 days	30 min
Long-lived elements: Ce, Co, Cr, Cs, Eu, Fe, Hf, Rb, Sb, Sc, Sr, Ta, Tb, Th, Zn, Zr	5×10^{13}	24 h	21 days	2.5 h

2.4. INAA measurements

In this work, the analytical technique employed was INAA using the MURR reactor for the activation and the HP Ge detector was connected to a CANBERRA system for gamma-ray spectroscopy. Elements measured from the short-irradiation samples were the following: Al, Ba, Ca, Dy, K, Mn, Na, Ti, and V. The long-irradiation samples in quartz vials were irradiated as a single bundle by a thermal neutron flux of 5.0×10^{13} n cm^{-2} s^{-1} for a period of 24 h. Two counts were made on the samples after long irradiation also using a HP Ge detector. The first count for 30 min per sample was made after the samples decayed for 7 days and enabled successful measurement of the following medium-lived elements: As, La, Lu, Nd, Sm, U, and Yb. The second count for 2.5 h per sample was made after the samples decayed for an additional 14 days and enabled measurement of the following long-lived elements: Ce, Co, Cr, Cs, Eu, Fe, Hf, Rb, Sb, Sc, Sr, Ta, Tb, Th, Zn, and Zr. The REE concentrations in our samples were measured for the elements La, Sm, Ce, Nd, Eu, Yb and Lu. Concentrations in the unknown samples were determined relative to the NIST standards SRM-1633a Coal Flyash and SRM-278 Obsidian Rock which were prepared and irradiated under identical conditions with the sediment samples. Measurement uncertainties for almost all of the elements are under 3%.

In this work, optimum conditions were used to obtain interference free photo-peaks of the desired elements. Irradiation, cooling and counting time were optimized depending on the half-lives of elements analyzed, in order to improve the ratio between the relative activity of each element and the bulk activity of the matrix. Table 2 shows the estimated detection limits for INAA using decay gamma rays. Assuming irradiation in a reactor thermal neutron flux of 1×10^{13} n cm^{-2} s^{-1}, at University of Missouri Research Reactor (MURR), University of Missouri-Columbia, USA [11].

3. Results and discussion

The analytical results of 32 elements from the eight sediment samples are presented in Tables 3, 4, and 5 according to their respective radioisotope half-life: short, medium and long. The elements

Table 2
Estimated detection limits for INAA using decay gamma rays. Assuming irradiation in a reactor thermal neutron flux of 1×10^{13} n cm^{-2} s^{-1}.

Sensitivity (picograms)	Elements
1	Dy, Eu
1–10	In, Lu, Mn
10–100	Au, Ho, Ir, Re, Sm, W
100–1E3	Ag, Ar, As, Br, Cl, Co, Cs, Cu, Er, Ga, Hf, I, La, Sb, Sc, Se, Ta, Tb, Th, Tm, U, V, Yb
1E3–1E4	Al, Ba, Cd, Ce, Cr, Hg, Kr, Gd, Ge, Mo, Na, Nd, Ni, Os, Pd, Rb, Rh, Ru, Sr, Te, Zn, Zr
1E4–1E5	Bi, Ca, K, Mg, P, Pt, Si, Sn, Ti, Tl, Xe, Y
1E5–1E6	F, Fe, Nb, Ne
1E7	Pb, S

Table 3
Concentrations of major and trace elements in sediments of Julian Adame-Alatorre dam for short-lived elements (in mg kg^{-1}).

Element	JA01	JA02	JA03	JA04	JA05	JA06	JA07	JA08	Range	Mean
Al (%)	7.16	7.67	7.71	7.69	6.85	6.65	6.76	7.88	6.65–7.88	7.30
Ba	670	980	921	840	1139	1112	607	885	607–1139	894
Ca (%)	0.83	0.83	0.97	0.92	0.68	0.60	0.77	1.32	0.60–1.32	0.87
Dy	7.72	8.09	6.89	6.97	4.37	4.39	7.15	7.42	4.37–8.09	6.63
K (%)	2.43	3.00	2.66	2.77	3.39	3.24	2.66	2.28	2.28–3.39	2.80
Mn	290	752	334	404	348	328	397	247	247–752	387
Na (%)	1.17	1.15	1.31	1.24	1.90	1.87	1.22	1.00	1.00–1.90	1.36
Ti (%)	0.31	0.30	0.31	0.34	0.35	0.37	0.30	0.35	0.30–0.37	0.33
V	46.9	47.6	43.6	44.7	28.8	25.7	30.5	51.2	25.7–51.2	39.9

Table 4
Concentrations of major and trace elements in sediments of Julian Adame-Alatorre dam for medium-lived elements (in mg kg^{-1}).

Element	JA01	JA02	JA03	JA04	JA05	JA06	JA07	JA08	Range	Mean
As	<2.8	5.7	3.2	3.4	6.5	4.8	6.8	<3.4	2.8–6.8	4.6
La	39.7	57.8	43.3	48.3	30.0	29.8	38.0	43.9	29.8–57.8	41.3
Lu	0.57	0.75	0.62	0.68	0.43	0.43	0.64	0.57	0.43–0.75	0.59
Nd	33.7	57.6	38.4	40.6	24.7	24.7	37.6	42.8	24.7–57.6	37.5
Sm	7.48	11.33	8.19	9.06	5.09	4.97	7.82	8.07	4.97–11.33	7.75
U	6.13	5.01	3.30	4.62	2.83	3.77	4.94	3.49	2.83–6.13	4.26
Yb	3.85	5.35	4.00	4.96	2.93	2.90	4.63	3.97	2.90–5.35	4.07

determined were As, La, Lu, Nd, Sm, U, Yb, Ce, Co, Cr, Cs, Eu, Fe, Hf, Rb, Sb, Sc, Sr, Ta, Tb, Th, Zn, Zr, Al, Ba, Ca, Dy, K, Mn, Na, Ti, and V. For the determination of several rare earth elements, INAA is also favorable, in particular, the elements Ce, Eu, La, Lu, Nd, Sm, and Yb. The mean concentrations of the major elements such as Al, Ca, Fe, K, Na, and Ti were 7.30%, 0.87%, 2.31%, 2.80%, 1.36% and 0.33%, respectively. Concentrations of the hazardous elements such as As, Ba, Cr, Mn, Sb, V and Zn were 4.6 mg kg^{-1}, 894 mg kg^{-1}, 18.6 mg kg^{-1}, 387 mg kg^{-1}, 0.72 mg kg^{-1}, 39.9 mg kg^{-1}, and 77.3 mg kg^{-1}, respectively. The relative errors (%) of most of the determined elements were within 5%.

INAA is also favorable for the determination of several rare earth elements (REE), such as, Ce, Eu, La, Lu, Nd, Sm, and Yb. Table 6 shows the mean concentration of the REEs in the eight sediment samples in ppm.

For the assessment of the contamination level of the Julian Adame-Alatorre dam, the elemental concentrations for As, Ba, Cr, Fe, Mn, and Zn were compared with the guidelines of USEPA [4], which is presented in Table 7. By this comparison, it can be concluded that As in seven of the samples, and Cr and Zn in two of the samples were moderately polluted. Ba in eight samples, Fe in four samples, and Mn in one sample were highly polluted in some of the sampling locations for sediments from Julian Adame-Alatorre. On the other hand, elements generally known to be associated with air pollutants, such as As, showed moderate levels of pollution [12,13]. On the other hand, for the assessment of the contamination levels at the Julian Adame-Alatorre dam, the elemental concentrations for As, Ba, Cr, Ta, and V were compared with the guidelines of México, NOM-147-SEMARNAT/SSA1-2004 which is presented in Table 8. By this comparison, it can be concluded that all concentration values are below guidelines of México.

Comparison of the elemental concentration in sediment samples with continental crust values, enrichment factor (EF) has been calculated, and the following expression was used:

$$EF_X = (X/Fe)_{\ Sediment} / (X/Fe)_{\ Continental\ Crust}$$

where EF_X stands for the enrichment factor of an element X in sediment samples, after having its concentrations (X) in sediment samples normalized to (Fe) concentrations in sediment samples or crust. A value of EF > 1 for a particular element indicates the enrichment of that element in sediment samples [6,7]. The data used to calculate

Table 5
Concentrations of major and trace elements in sediments of Julian Adame-Alatorre dam for large-lived elements (in mg kg^{-1}).

Element	JA01	JA02	JA03	JA04	JA05	JA06	JA07	JA08	Range	Mean
Ce	72.6	104.0	82.9	90.5	60.7	60.2	71.8	84.5	60.2–104.0	78.4
Co	3.22	7.60	6.00	5.39	2.51	2.60	4.09	5.87	2.51–7.60	4.66
Cr	14.8	25.7	31.4	24.0	6.6	7.8	15.5	22.8	6.6–31.4	18.6
Cs	12.5	30.8	26.1	23.7	4.9	4.7	12.2	18.9	4.7–30.8	16.7
Eu	1.14	1.70	1.28	1.41	1.15	1.15	1.13	1.16	1.13–1.70	1.27
Fe (%)	1.92	3.31	2.87	2.75	1.54	1.51	2.05	2.51	1.51–3.31	2.31
Hf	12.2	10.3	12.0	12.0	11.2	10.7	11.7	9.5	9.5–12.2	11.2
Rb	140	174	145	166	158	155	142	126	126–174	151
Sb	0.71	0.92	0.85	0.83	0.50	0.54	0.70	0.69	0.50–0.92	0.72
Sc	6.09	9.88	8.56	7.73	3.53	3.38	6.08	7.79	3.38–9.88	6.63
Sr	99.8	195	140	130	116	104	120	94.9	94.9–195	125
Ta	1.57	1.74	1.68	1.73	1.49	1.49	1.60	1.24	1.24–174	1.57
Tb	1.14	1.64	1.16	1.39	0.75	0.69	1.18	1.06	0.69–1.64	1.13
Th	13.1	15.7	13.5	13.6	11.4	11.0	12.5	12.4	11.0–15.7	12.9
Zn	70.8	93.4	132	78.0	43.3	48.6	73.4	79.1	43.3–132	77.3
Zr	308	265	306	318	325	272	284	239	239–325	290

Table 6
Concentrations of rare-earth elements in sediments of Julian Adame-Alatorre dam (in mg kg^{-1}).

Element	JA01	JA02	JA03	JA04	JA05	JA06	JA07	JA08	Mean
Ce	72.6	104.0	82.9	90.5	60.7	60.2	71.8	84.5	78.4
Eu	1.14	1.70	1.28	1.41	1.15	1.15	1.13	1.16	1.27
La	39.7	57.8	43.3	48.3	30.0	29.8	38.0	43.9	41.3
Lu	0.57	0.75	0.62	0.68	0.43	0.43	0.64	0.57	0.59
Nd	33.7	57.6	38.4	40.6	24.7	24.7	37.6	42.8	37.5
Sm	7.48	11.33	8.19	9.06	5.09	4.97	7.82	8.07	7.75
Yb	3.85	5.35	4.00	4.96	2.93	2.90	4.63	3.97	4.07

enrichment factors of various elements in sediment samples and the enrichment are shown in Table 9 and in Fig. 2, respectively. It is clear from this figure that the concentrations of most elements are slightly higher in the sediment samples as compared to the background continental crust, which indicate that there are some migration and accumulation of these elements in the sediments. While $EF \leq 1$ is usually regarded as nondescript for the elements Cr, Co, Ca, Sc, Sr, Mn, and Fe. Elements with EF values that are considerably higher than 1, $EF > 1$, can be considered not originated from the local soil background and may be attributed to long distance transport phenomena from other natural and/or anthropogenic sources; those elements are Na, Ti, Ta, Al, Eu, Zn, Lu, Tb, Sm, Ce, Nd, K, Th, Yb, La, U, Rb, Zr, Ba, and Dy in sediment samples. However, the elements As, Sb, Hf, and Cs showed highest enrichment factors; the widely accepted threshold for substantial enrichment over the natural continental crustal background is $EF \geq 10$ [8,14].

For a better overview of the data, the metal concentration variations in the sediments were plotted. See Fig. 3. The As and Cr concentrations were multiplied by 10, and the Sb concentration was multiplied by 100 to improve the visualization of the data. For a better overview of the data, the rare-earth element concentration variations in the sediments were also plotted. See Fig. 4. The Eu and Lu concentrations were multiplied by 10, to improve the visualization of the data [15].

Finally, the elements may be divided into the following three groups according to their median values [9]:

(1) Major elements (1–10%): Fe, Al, K, and Na.
(2) Minor elements (10 ppm–1%): Ca, Ti, La, Nd, Ce, Cr, Cs, Hf, Rb, Sr, Th, Zn, Zr, Ba, Mn, and V.
(3) Trace elements (<10 ppm): As, Lu, Sm, U, Yb, Co, Eu, Sb, Sc, Ta, Tb, and Dy.

4. Conclusions

In this study, sediment samples from the Julian Adame-Alatorre dam were analyzed by INAA and 32 elements were determined. When the elemental contents for As, Ba, Cr, Fe, Mn, and Zn were compared with the guidelines of USEPA, the dam sediments turned out to be moderately contaminated.

The differences in the elemental concentrations between guidelines of USEPA and guidelines of México are large. The results of this study indicated that sediments from Julian Adame-Alatorre dam present concentration levels of As, Ba, Cr, Fe, Mn, and Zn higher than the not polluted USEPA guidelines values. Concentration levels for As, Ba, Cr, Ta, and V are lower than residential use México Guideline values.

The main lessons learned from the present study can be expressed as follows: (1) sediments in the Julian Adame-Alatorre dam are contaminated with Ba, Fe, and Mn, in eight, three and one sediment samples, respectively. The contamination is probably derived from air pollution; but the levels are low and indicate that atmospheric deposition is not a major contributing factor to the sediment contamination. (2) In all cases sediment concentrations for As, Ba, Cr, Ta, and V did not exceed the "minimum threshold of safety" according to Mexican legislation. The enrichment factors found through this study show the sediments to be efficient elemental

Table 7
Comparison of elemental contents with USEPA guidelines (mg kg^{-1}).

Element	USEPA guidelines for classification of great lakes harbor sediments			Present work	
	Not polluted	Moderately polluted	Heavily polluted	Range	Mean
As	<3	3–8	>8	2.8–6.8	4.6
Ba	<20	20–60	>60	607.4–1139.1	894.3
Cr	<25	25–75	>75	6.6–31.4	18.6
Fe	<17,000	17,000–25,000	>25,000	15,100–33,100	23,100
Mn	<300	300–500	>500	247.3–751.5	387.3
Zn	<90	90–200	>200	43.3–131.9	77.3

Table 8
Comparison of elemental contents with NOM-147-SEMARNAT/SSA1-2004 guidelines of México (mg kg^{-1}).

Element	NOM-147-SEMARNAT/SSA1-2004 guidelines of México		Present work	
	Commercial, agricultural, and residential use	Industrial use	Range	Mean
As	22	260	2.8–6.8	4.6
Ba	5400	67,000	607.4–1139.1	894.3
Cr[a]	280	510	6.6–31.4	18.6
Ta	5.2	67	1.24–1.74	1.57
V	78	1000	25.7–51.2	39.9

[a] Hexavalent chromium.

Table 9
Enrichment factors of elements in sediments relative to continental crust, taking Fe as a crustal reference.

Element	This work, mean (mg kg^{-1})	Continental crust average[a] (mg kg^{-1})	EF
Al	73,000	82,300	2.2
As	4.6	1.80	6.2
Ba	894	425	5.1
Ca	8700	41,500	0.5
Ce	78.4	60	3.2
Co	4.66	25	0.5
Cr	18.6	100	0.5
Cs	16.7	3	13.6
Dy	6.63	3	5.4
Eu	1.27	1.2	2.6
Fe	23,100	56,300	1.0
Hf	11.2	3	9.1
K	28,000	20,900	3.3
La	41.3	30	3.4
Lu	0.59	0.5	2.9
Mn	387	950	1.0
Na	13,600	23,600	1.4
Nd	37.5	28	3.3
Rb	151	90	4.1
Sb	0.72	0.2	8.8
Sc	6.63	22	0.7
Sm	7.75	6	3.1
Sr	125	375	0.8
Ta	1.57	2	1.9
Tb	1.13	0.9	3.1
Th	12.9	9.6	3.3
Ti	3300	5700	1.4
U	4.26	2.7	3.8
V	39.9	135	0.7
Yb	4.07	3	3.3
Zn	77.3	70	2.7
Zr	290	165	4.3

[a] Taylor, S.R. 1964.

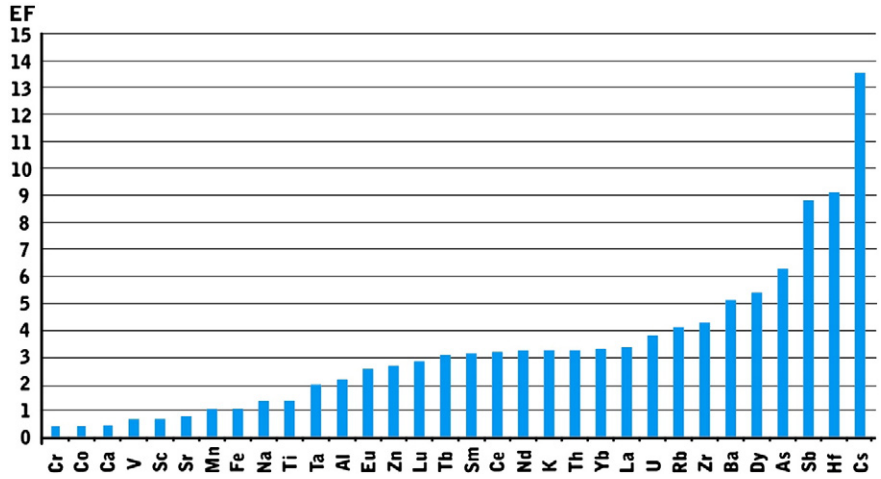

Fig. 2. Enrichment Factors (EF) for major, minor, and trace elements in sediments.

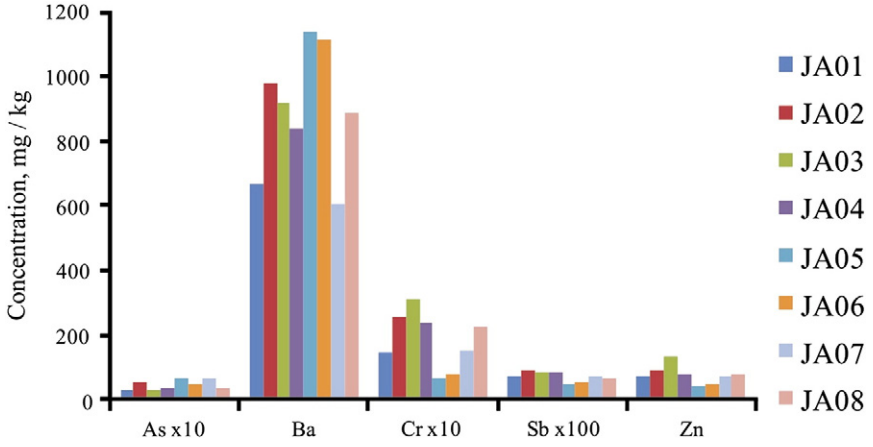

Fig. 3. Concentrations of metal elements in sediments of Julian Adame-Alatorre dam.

accumulators from the environment. The calculated EF for quantified elements identified As, Sb, Hf, and Cs as elements with very high EF values, which suggests that these elements may be derived not only from the local soils but also from other natural and/or anthropogenic sources by long transport phenomena.

Disclaimer

"Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the Department of Energy".

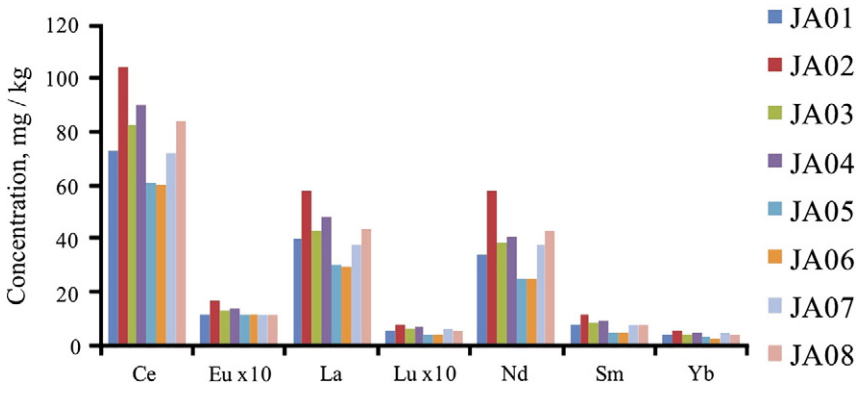

Fig. 4. Concentrations of rare-earth elements in sediments of Julian Adame-Alatorre dam.

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