*Research Article /Araştırma **Makalesi***

Heavy Metals Distribution and Speciation in Sediments from Ziqlab Dam - Jordan

Ürdüm Ziglab Barajı Sedimanlarinda Ağır Metal Dağılımı ve Türleşmesi

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ABSTRACT

Thirty surface sediment samples from the Ziqlab Dam area were collected and analyzed for nine: elements (Pb, Cd, Zn, Mn, Ni, Cu, Fe, Cr, and Co). Metal separation was determined by sequential fraction, The fractions are, a) exchangeable, b) carbonate» c) Fe / Mn oxides d) organic, and e) residual. The advantage of using these fractions is to provide the mechanism of association of metals with the minerological phases of the sediments. Concentrations of the elements are within, allowable levels except for Pb, Cd. and. Zn and in. some locations Ni. Most of the elements were found to be in the residual fraction which clearly indicates that, these metals are primarily immobile and have or bear the least bioavailability.

Key Words.: Heavy metal, Contamination, Dam. Sediments, Ziglab Dam.

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Ziglab Baraj alanından 31 yüzey sediman örneği toplanarak 9 element için (Pb, Cd, Zn, .Mn, Ni, Cu, Fe, Cr ve Co) analiz edilmiştir.. Metal ayrımı sıralı ayrımlamaya göre yapılmıştır. Ayrımlamalar a) değiştirilebilir, b) kanbonat, c) Fe/Mn oksitlen d) organik ve e) kalıntı sırasıyla gerçekleştirilmiştir.. Bu ayrımlamaları kullanmanın yararı, metallerin sedimanlerdeki mineralojik tarzlarla bir arada bulunma mekanizmasını dikkate almasıdır. Elementlerin değişimi Pb, Cd ve Zn ve bazı alanlarda Ni dışında izinverilebilir sınırlar içindedir.. .Metallerin çoğu kalıntı kısımda bulunmuştur.. Bu da, bu metallerin başlıca hareketsiz ve biyolojik aktivîteye katılımın en düşük düzeyde olduğunu açıkça göstermektedir,

Anahtar Sözcükler; Ziglab Barajı, ağır metal baraj sedimanı, kirlilik..

Introduction.

The study area is located in the co-ordinate of E 2091, N 2144 near the village of El-Aziya in Jordan (Fig. 1). The area under- irrigation by 'the Ziglab dam is about 400 hectares. The mean annual runoff is 13.04 Million Cubic Meter, 9.6% of which is flood run-off, (JVA 1965). The Ziglab River catchment area is about 111 Ion., It consists of steeply graded. hillsides with drainages in deeply incised valleys. The upper catchment area has a maximum elevation of+1050 m. a.s.1 with a sparse natural forest cover. Some parts of the lower catchment area are covered by loamy soil

Limestones and marls of the upper- Ajlun and Balqa series characterize the: whole area, (JVA 1965). The: geological succession in the area is talus, alluvium, cap conglomerates with crystalline and. pisolitic limestones., red pebbly and sandy marls.

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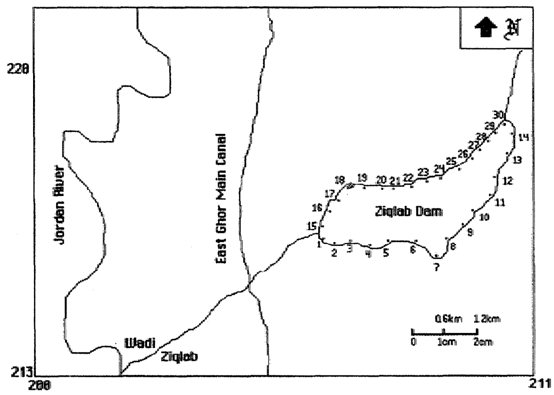

Figure 1: Location and sampling sites of the study area.

cornstones and lenticular calcareous conglomerates, crystalline limestones, glauconite calcareous sandstones and chalk, (JVA 1965) (Fig.2).

Heavy metals tend to be trapped in estuaries and dams and are this of particular concern in this type of environments. Metal concentrations in the particulate form can be 3-5 orders of magnitude higher[1] than in the dissolved form as stated by Balls (1989), and Comber, et al (1995), therefore the bulk of trapped metals tend to accumulate within estuary and dam environments, (Salomons and Forstner 1984). Metals accumulated in this way may be subsequently released to the overlying water column as a result of either physical disturbance, or diagenesis and sediments may be a constant source of pollutants long after the cessation of direct discharges, (Boughriet, et ai 1992; Peterson et al. 1995).

Data on metal concentrations, in the Ziglab River and at Ziqlab Dam area have been scarce until recently. Abu-Rukah and Ghreafat (in press) conducted, the only study concerning ion chemistry of Ziglab Dam and weathering processes, They concluded that: anthropogenic activities, including various development activities, 'waste disposal operations untreated municipal or urban, sewage and agricultural activities within the Ziglab catchment area, contributed to the increase in ionic concentration.

Objectives.

The present study was undertaken to evaluate the effect of industrial, municipal or urban, and agricultural pollutants discharged into the Ziqlab River that settled behind the Ziqlab Dam, in the light of concentration of Pb, Cd, Zn, Cr, Co, Mn, Fe, Cu, and Ni in the sediments of Ziqlab dam area. The extraction method of Tessier, et al (1979) as modified by Ajay and Van Lron (1989) and appeared in Jones and Turki (1997) was followed. The method provides information on five mineralogical fractions, namely 1) exchangeable, 2) carbonate, 3)Fe/Mn oxides, 4) organic, and 5) residual fractions.

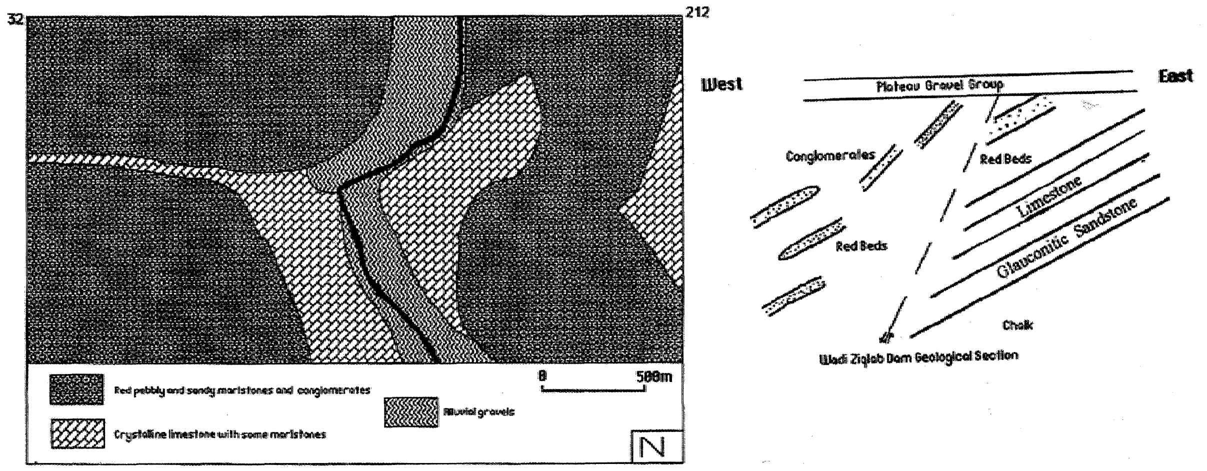

Figure 2: Geological map of the dam site.

Sampling a n i **Analytical Techniques**

A total of 30 sediment samples were collected from the Zlqiab Dam area on 10/12/1998 at depth ranges from 0-5 cm, sampling location are shown in Fig.. 1, The samples were- stored in polythene bags and taken to the Laboratories of the Department of Earth and Environmental Sciences in Yarmouk University. The sediments had a variety of particle sizes. The heavy metal analyses were conducted on. the 0.2 |xm fraction,, which was separated by wet screening with distilled water through a. nylon, sieve. The sieved, samples were: dried at 65 C in an oven for 24 hours. A. half gram of sediment from representative samples was taken for' heavy metal analysis (Pb, Cd,. Zn, Ni, Cu, $Fe_{S!}$ Mn, Cr, and Co) using atomic absorption, spectrophotometer (PYE UNICAM SP9).

The sequential extraction scheme of Tessier et. ai (1979) was followed.. All extractions were carried out in 50-ml glass centrifuge tubes. Continuous magnetic stirring or agitation in a mechanical shaker ensured proper mixing of sediment and extraction solution. Suspensions were centrifugea for 30 min at 3000 rpm subsequent to each extraction step.. The extracted metals, were then separated from, the residual sediment by décantation. A short: description of the 5 fractions most likely to be relevant, in assessing the effect of changing environmental conditions by the polluted sediments is given below..

Fraction 1: . Exchangeable

Metals extracted in the exchangable fraction would include weakly adsorbed metals particularly those retained on the sediment surface by relatively weak electrostatic interaction and those that can be released by ion-exchange processes.. Changes in the ionic composition of the water would, strongly influence these adsorption-desoiption and. ion. exchange processes of metal ions with the major constituents of sediments like clays, and hydrated oxides of iron and manganese.

Procedure: 1 g of sediment was extracted, at room temperature for 1 h. with 8 ml magnesium, chloride solution (1 *M* MgC12, pH=7).

Fraction 2: Bound to carbonates

Significant amount of trace metals like manganese can be co-precipitated with carbonates which are present in many sediments. Lowering, of the pH could cause remobilization of the metals from, the fraction.

Procedure: The residue from 'fraction 1 was leached with 8 ml 1 M sodium acetate/acetic acid buffer at. pH=5 for 5 h at room temperature.

Fraction 3: Bound io Iron and Manganese oxides

Iron and manganese oxides, which can. be: present in sediments as concretions,, cement between particles or coatings on particles, are excellent substrates with, large surface areas for adsorbing trace metals,. Reduction of Fe **(III)** and Mn (IV) order anoxic conditions and their subsequent dissolution could release adsorbed trace metals.

Procedure: The residue from fraction. 2 was extracted under mild reducing conditions with 20 ml of 0.4 M hydroxyl amine hydrochloride (NH2OH.HC1) in 25 % (V/V) acetic acid at 96 ± 3"C in a water- bath for 61i.

Fraction 4: Bound to organic matter

Various forms of organic matter like detritus, living organisms and coatings on. mineral particles may bind trace: metals through complexation or bioaccumulation processes.. Under oxidizing conditions, these substances may be degraded thus leading to a release of soluble metals,.

Procedures: The residue from fraction 3 was treated with 3 ml 0.02 *M* nitric acid and 5 ml 30 % (V/V) hydrogen peroxide. The mixture was heated to 85 ± *2* °C in a water bath for 3 h. After cooling, 5 ml of "3.2 M ammonium acetate in 20 % (V/V) nitric acid. was. added to the sample and diluted to 20 ml.

Fraction 5: Residua! or inert fraction

The residual fraction largely consists of mineral compounds, where metals are firmly bounded within crystal structure of the minerals comprising the sediment. These metals are not likely to be released into solution under normal environmental conditions.

Procedures: The residue from fraction 4 was digested with a 5:1 mixture of hydrofluoric acid and perchloric acid in Teflon beakers.

Result and Discussion

Heavy Metal Distribution:

The- concentrations of metals in the sediments of the Ziglab Dam area are given, in Jable 1 and shown in Fig. 3., Many authors prefer to express the metal ratio with respect to average shale to represent the degree of quantification of pollution.The metal ratios with respect to average shale are given in Table 2, Muller (1979) introduced a. quantitative measure- of the metal pollution in sediments and solid waste materials Le, the index, of geo-accumulation. (I-geo) which is calculated as

I-geo = Iog2 Cn / L5 X B. **(1)**

Where: Cn = is the measured concentration of element n in the politic fraction of sediment (clay) **(< 2 um).**

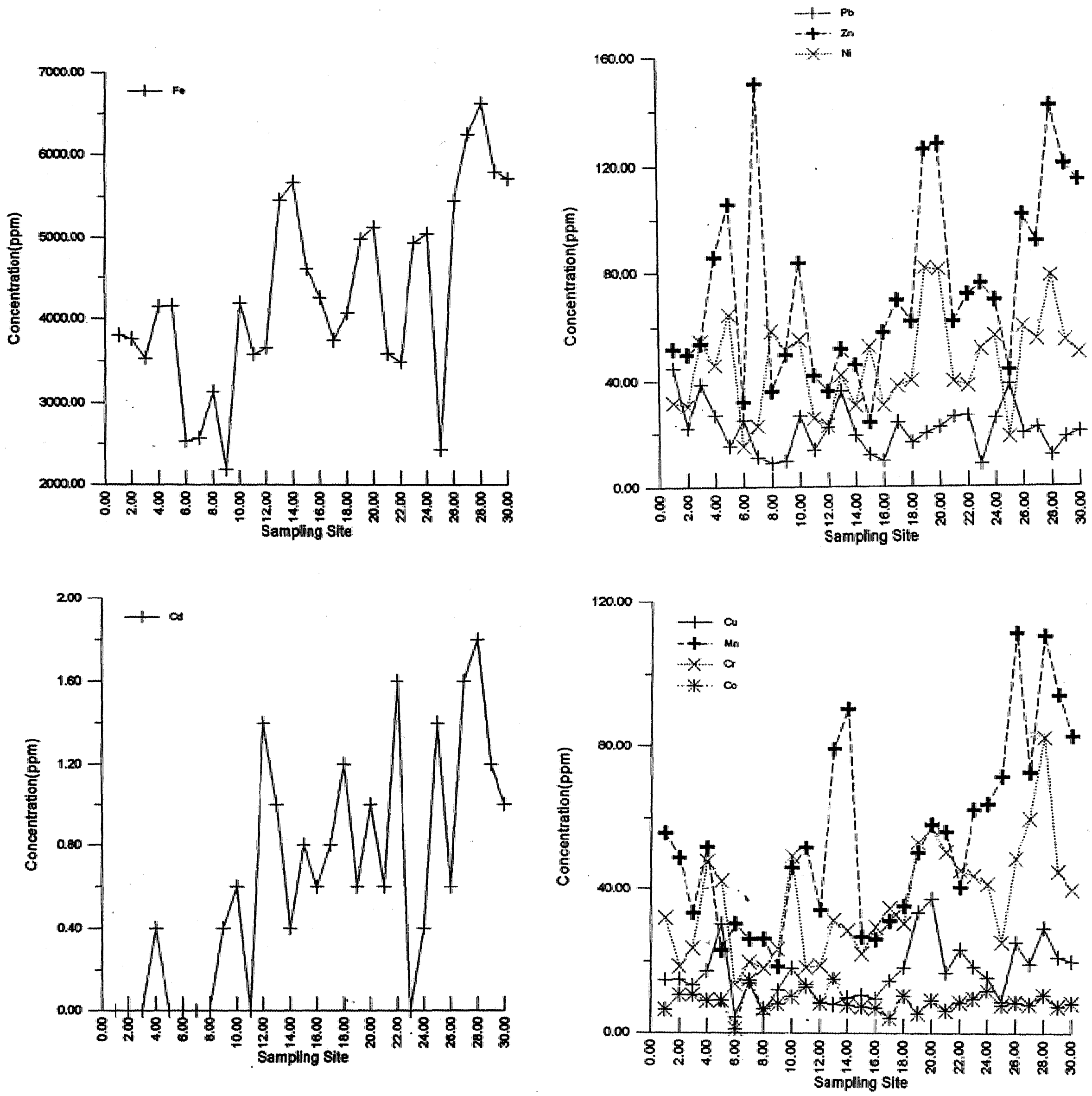

Figure 3: Concentration **of various** elements in tbe collected samples from Ziqlab Dam area.

Table 1: Heavy metal ccmceDtration(ppm) in the clay fraction of Ziqlab dam area sediments

Sample No	Pb	Cd	Zu	Ni	Cu	Fe	Ma	Cr •	Co
Î	448	ÖÎ	52	3L8	ÎÎÎ	3806.0	55ÎS	32LÛ	6,4
.2	21.8	0,0	50	30..4	15.0	3764.0	48.6	18.6	10.4
3	38,8	0.0	54	54.8	13.2	3536.0	33.4	23.6	10.4
4	26.8	0.4	86	46.0	17.4	4154.0	51.6	47.6	8.8
5	15.2	0,0	106	64.6	30.2	4164.0	23.0	42.2	9.0
6	24.8	0.0	32	15.0	4.4	2526.0	30.4	13,4	1.2
7	10.8	0.0	150	22.6	14.0	2564.0	26..0	1.9.8	14.8
8	8.6	0.0	36	5M	52	3136.0	26.2	18.2	6.6
9	9.4	0,4	50'	51.4	11.8	2180.0	18.6	23.4	8.0
10	26.4	0.6	84	55.4	18.2	4196.0	46.0	49.2	10.0
11	13.6	0.0	42	25.6	13,4	3584.0	51.6	18.4"	12.6
12	22.2	1.4	36	22.8	8.4	3662.0	34.2	19.0	8.2
13	36.2	1.0	52	42.2	7.8	5462.0	79.4	31.4	15.2
14	19.0	0.4	46	30.8	9.6	5668.0	90.6	28.6	7.6
15	11.8	0.8	24	52.8	10.4	4602.0	26.8	22.2	7.2
16	9.6	0.6	58	30.8	9.4	4262.0	26.0	29.6	6.8
-17	23.9	0.8	70	38.0	14.6	3752.0	31.2	34,8	4.2
18	16.4	1.2	62	40.0	18.4	4076.0	35.4	30.6	10.2
19	19.8	0.6	126	82.0	33.6	4984.0	50.2	53.2	5.4
20	22.2 "	1.0	128	81.4	37.4	5126.0	58.4	57.2	9.0
21	26.0	0,6	62	39.8	17.0	3594.0	56.4	50,4	6.2
22	26.6	1.6	72	38.2	23.4	3494.0	40.6	45,4	8.4
23	8.2	0.0	76	52.0	18.6	4940.0	62.6	43,8	9.4
24	25.6	0,4	70	56.8	15.6	5048.0	64.2	41.6	11.6
25	38.8	1,4	44	18.4	8.6	2422.0	72.0	25.4	7.6
26	19.8	0.6	102	60.2	25.4	5456.0	112.2	48.6	8.4
27	22.0	1.6	92	55.6	19.4	6242.0	73,2	60.0	7.8
28	11.6	1.8	142	79.0	29.4	6626.0	111.4	83.0	10.4
29	18,4	1.2	121	55.1	21.3	5800.0	95.0	45.0	7.3
30	20.3	1.0	115	50.8	20.0	5717.0	83.4	40.1	8.1

Bn = is the geochemical background for the elementn. Bn is either directly measured or taken from the literature (average shale value) Ntekim» et al (1993). Muller (1979) established seven I-geo classes based on. the numerical index value. Table 3 is a summary of seven classes and their implications with regard to contamination. The index of geo-accumulation has been used to assess the heavy metal levels in the Ziglab Dam area. Results are summarized, in Table 4, which indicates that the Ziglab Dam area is uncontaminated/moderately contaminated with Pb and Cd. The elements of Mn» Zn, Co» Ni, Cr, Cu and Fe are below the contamination level in the sediments of dam area. A comparison of left and right banks of the Ziglab Dam Reservior is given in Table 5., This reveals that concentration of Cd is greater in the left bank and of Pb in the right bank with respect to each, other..

. Heavy metals in sediments are either lithogenic or anthropogenic (Ntekim, et al, 1993).. The present investigation, has revealed high concentrations for Pb, Cd and in some samples for' Zn. (Samples. No, 5, 7,19,20,26,2f,28,29 and 30) and Ni (Samples No.. 19, 20 and 28). These high concentrations may be introduced by anthropogenic sources Le, fertilizers, pesticides», animal manure, sewage discharge from various, sources within, the Ziglab Basin and from several industrial facilities located, along the Ziglab River. The current levels of Cu, Fe, Mn, Cr, Co, Ni

Table 2: Metal ratios with to average shale of Ziqlab Dam area, sediments.

Element	Average concentration, (ppm)	Metal ratio
Pb	2133	**1.07**
Cd	**0.647**	2.16
Zu	74.67	0.79
Ni	46.18	0.68
Cu	**16.53**	0.37
Fe	4285	**0.09**
Mn	**53.81**	**0.06**
Cr	36.54	0.40
Co	**8.57**	0,45

and Zn in the Ziglab Dam ecosystem in general are low.. Lower concentrations of Cr, Ni, and. Co are consistent with the views of Forstner (1980), that these elements are practically unchanged by anthropogenic influences.

Atmospheric pollution, is minimal but Pb and Zn may be derived, from, combustion as. well as from gasoline additives used, in the factories (Ntekina, et al, 1993). These elements may also be derived through corrosion of the numerous abandoned launches along the river as. well as from the municipal pipe systems, (Bellman, 1.972),

Metal Spedation

Median metal concentration in. the Ziglab Dam area, decreases in the order' Pb> Cd> Zn> M> Cu> Fe> Mn>.Cr> Co.. Results of the selective leaching procedure are presented in Fig. 3. In general the sums of extracted fractions lie to within. 10% of independently determined, total metal concentrations. This- supports the overall accuracy of the extraction procedure..

Table 3: Measure of metal contamination in. aquatic sediments and solid waste (Müller 1979).

Index of Geo-accumulation;	I-geo class	Désignation of sedimeol quality
10-5	6	Extremely contaminated
4-5	5	Strongly / extremely contantinated:
3-4	4	Strongly contaminated
2-3	3	Moderately / strongly contaralnatenl
1-2	.2	Moderately cortamiaated
Ö-!	1	Uncontaminated / moderately.contaminated
0	0	uncontajninated

Table 4: Measure of metal contamination in sediments of..the- Ziqlab Dam. area, using geoacumutation index, of Midler, (1979).

Element	Average concen-'(ration (ppm) of Ziqlab Dam.	Average shale (Standard)	Designation of sediment quality
Pb	21.33	20	Uncontaraimated to moderately c0n.tamin.ated
Cd	€.641'	0.3	Unconlamlniatecl to moderately contaminated
Mn.	5.3.81	8.30	Uncootuninated
Zn	74.67	95	UncontaminatBd
Co	8,57	19	Uncontaarinated
Ni.	46,. 18	6K.	Unoontanrinated
Or	3654	90	Unoantamdnated
Co	16.53	45	Uncontaminated
Fe	4285	46.700	UncontamİDated

Pb, Zn, Cd and Ni. are the most abundant metals analyzed and are distributed with the residual Fe / Mn oxides» To a lesser' extent, the organic fraction is of some significance (Fig. 4 and Table 6). The residual fraction is dominated by Pb, Zn, Cr, Co, Fe and Cu. It includes approximately 78%> of the total almost in all the sites. Since the resultant sequential extraction for- Pb, Ca, Zn, Co, Cr, F'e and Cu. is mainly associated with the residual fraction,, it clearly indicates that: those heavy metals are mainly immobile and. are least available biologically.. It should be pointed out that extraction results do not necessarily prove the existence of any of the.defined, phases in. sediments,, but merely reflect the chemical behavior of metals within, the different extracting solutions (Coetzee, 1993). •

The' exchangeable fraction is responsible for 1.4-9.4% of the total concentration,. Where Cd concentrations are the highest (sites 1.2, 13, 18,, 22, 25,, 27,, 28,29 and 30). The residual, fraction is dominant with 84% Pb (sites 1, 3, 13 and 26). This is accompanied by an increase in the: Fe/Mn oxide- fraction of

Table 5: Comparison of mean heavy metal concentrations (ppm) between right and left banks of the Ziglab Dam. reservoir.

Heavy Metal.	Left bank 16 samples	Average Shale (Standard)	Right: bank 14 samples
Pb	20.06	20	22.742
Cd	0..96 •	0.3	0 3
z0	. 81 ..937	95	62.571
Ni	51,931	58	39.428
Oı	•• 20.1.31	45.	13.1
Fe	47.58.8!	46.700	3743
Mn:	62.462	850	43,954
Cr	44.4.31	90!	27,528
Co	8	19	9.228

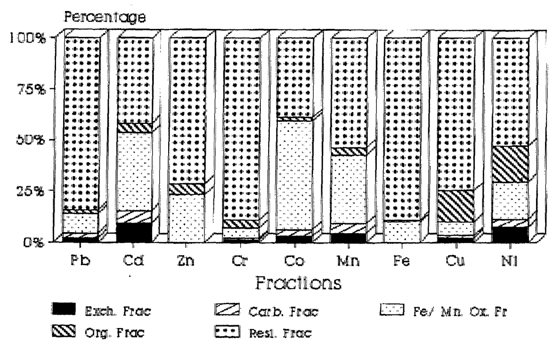

Figure 4: Proportion of the **geochemical** forms of heavy metal in **the** Zigqlab Dam area.

Table 6: Heavy metal percentages in different **geochemical** fractions of the Ziqlab Dam. area sediments.

Element	Exchangeable Fraction	Carbonate Fraction	Fe/Mn Oxides Fraction	Residual Fraction
	Geochemical fraciioos(%)			
Pb	2.1	2.2	9.8	84.0
Cd	9.4	6.2	38.0	41.7
Zn	0.0	0.0	23.4	71.3
Cr	1.4	0.6	5.1	88.8
Co	4.8	5.9	20.9	65.7
Mn	4.5	4.8	33.6	53.6
Fe	0.06	0.04	9.8	88.9
Cu	2.2	1.1	7.0	74.6
Ni	7.6	3.8	18.3	52.7

9.8%, the: carbonate fraction of 22%, and Cr residual fraction of **88.8%.** Fe **/Mn** oxides with 5.1% are important as metals hosts., The distribution of Pb, Cr . and **Fe (Fig.** 4) is similar being dominated, by **residual** and **Fe/Mn** phase with minor[1] exchangeable, carbonate- and organic fractions. Cd and Ni are the only elements for which the: exchangeable fraction, was significant (9.4% and 7.9%, respectively)..

Mn and Ni seem similar as dominants, of the residual fraction **(52.1%** and 53,6% respectively) with, significant amounts of Fe/Mn oxides phase **(18.3%** and **33.6%** respectively) and organic phase **(17.6%** and 3.5%, respectively),.

As shown in Fig,4 and Table: 6 ,, the: affinity of each measured, heavy metal **torward** major- sinks (geochemical phase) can be arranged as follows:

Pb: Residual >Fe/Mn oxides >Carbooate> Exchangeable Organic

Cd: Residual> Fe/Mn oxides> Carbonate. >Exchangeable >Organic

Zn: Residual> Fe/Mn oxides >Organlc> Exchangeable >Carbona«e.

Cr: Residual> Fe/Mn oxides >Organic> Exchangeable >Carbonate.

Co: Residual >Fe/Mn oxides> Carbonate >Exchangeable XDiganic

Mn: Residual> Fe/Mn oxides >Carbonate> Exchangeable >Oiganic

Fe: Residual >Fe/Mn oxides >Orga.nic Exchangeable >Carbonate.

Cu: Residual >Organiic >Fe/Mn Ox> Exchangeable >Carbonate.

Ni: Residual >Fe/Mn oxides >Organic Exchangeable >Carbo:nate..

The potential environmental impact of the metals could be estimated from the degree **of remobilization** which is measurable with the **five** extraction **categories.** These categories, exchangeable, bound to carbonate, bound to Fe/Mn. oxides, bound to **organic** matter, indicate **the** possible release of metals through the lowering of pH (exchangeable and carbonate) and changes, in **redox** potential (organic as Fe/Mn oxides phase). This would be very useful in **assessing** the potential, pollution risk, of **the sediments.** The residual phases do not generally **constitute an** environmental risk., The stable nature of **the** compound and the fact **that** the metals are bonded firmly within a. mineral lattice restrict the bioavailability of these metals **(Coetzee,** 1993),. The relative amount of metal, percentage in the residual, phase may be used as an indication of the degree of contaminant from anthropogenic sources,. The: greater[1] relative amount of metal in the residual phase,,, **the** smaller the: degree of pollution presented by the other phases (Table 4).

Conclusion

Surface sediments at Ziqlab Dam have **low concentrations,** almost, within the allowable levels for most of the heavy metals except for Pb, Cd, Zn and Ni. Metal distribution in dam **sediments is** controlled, to a greater extent by the **lithology** of the surrounded area and. by pollutants from human activities along **the** .Ziglab River **catchment**

The following; chemical fractions are arranged in the order[1] of increasing concentration, of the major heavy metals:

Pk Residual >Fc/Mn oxides >Carbonate> Exchangeable Organic

Cd: Rcs:idual> Fe/Mn oxides> carbonate. >Exchangeable >Org;anic

Zn: Residual> Fe/Mn oxides >Qrgamc> Exchangeable >Carbomate

Cr: Residual> Fe/Mn oxides >örgamic> Exchangeable >Caitonate

Co: Residual >Fe/Mn oxides> Carbonate >Exchangeable >Organk

Mm: Residual> Fe/Mn oxides >Carbo:nate> Exchangeable >Orgarai.c

Fe: Residual >Fe/hfn oxides >O;rgaiiic >Exchangeable >Carbonate

Co: Residual >Organic >Fe/Mn Ox> Exchangeable XDarbonate

Ni: Residual >Fe/M:ni oxid.es> >Organic ^Exchangeable >Carbonaie

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