Distribution of chemical constituents according to particle size in torrential rivers of the Basque country

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Abstract

Surface sediment samples of the rivers Bidasoa, Lea and Oria (NE of Spain), with torrential character, were taken at 26 selected points, dried and sieved to eight fractions for size analysis (from >2 mm to <63 microns; first fraction >2 mm was rejected). Determined parameters include major (carbonates, organic carbon, Ca, Mg, Na, K, Si, Al and Fe) and microconstituents (Cu, Mn, Zn, Cr, Pb, Ni and Cd), measured mainly by AAS (flow spoiler and graphite furnace) and FIA. Results are discussed by calculation of factors of enrichment in the fine fraction and by graphs of correlation coefficients of parameters versus size fractions. Most microconstituents, organic carbon and A1 (related to clays) were accumulated in the fine fractions of the sediments, suggesting pollution phenomena. The rest of the constituents seemed to be related to coarse fractions (Mg, carbonates) or not related to any particular size fraction. To discuss pollution phenomena, the comparison with major conservative elements (A1, Fe or Mg) was tried without success. A method was then proposed with the establishment of an index of quality for each pollutant constituent of sediments, based on the average earth's crust composition (or the average sedimentary rock content for organic carbon). With this method, fine samples show lower quality indices for most constituents, specially in the case of organic carbon, and total samples have much better indices suggesting organic carbon contamination only in seven cases.

Introduction

Sediment samples are often used as heavy metal pollution indicators in surface waters (Förstner and Salomons, 1980). The presence of heavy metals is affected by particle distribution of sediments (Thorne and Nickless, 1981). In this sense, Yamagata and Shigematsu (1970) pointed out that heavy metals should be analysed not only in total sediment, but also according to particle size. Oguna et al. (1979) showed that the concentration of a pollutant is distributed into two fractions of the sediments, i.e. the fine and the coarse. Salomons (1980) considered that both:

- the adsorption capacity by unit of weight usually increases as the particle size of the solids decreases; and
- the capacity of flow transport increases as the particle size decreases.

Förstner and Salomons (1980) also suggested that it is necessary to consider the distribution of size particle to estimate the natural metal levels of river sediments, because there is a marked decrease in the metal content as the particle size increases. Ackermann (1980) used an assay procedure to correct for different particle sizes on heavy metal content in estuarine and coastal sediments. He found that the best results are obtained using cesium for correction.

The comparison of the content of a heavy metal with a conservative element has been suggested to estimate sediment pollution at different depth levels. Magnesium (Hilton et al., 1985), iron (Allen and Brunskill, 1977) or aluminium (Bruland et al., 1974) have been mainly proposed as conservative elements. But the distribution of conservative elements according to sediment particle size may be variable because of changes in mineralogical constituents.

In this paper results are presented which form part of a broader study entailing the identification, origin and physico-chemical factors affecting pollution of torrential river sediments by heavy metals. This work is firstly focused on establishing relations between chemical parameters and size data for studied sediment samples, with special emphasis on estimating the distribution of pollutants in river sediments according to size particle. A second objective is to propose pollution criteria when considering river sediments which vary in both chemical composition and size particle.

Materials and methods

Sampling area

Sediments were collected from the Lea, Oria and Bidasoa Rivers with torrential regime, belonging to the hydrographic basin of the Cantabric Sea, Spain (Fig. 1). Geographic locations and characteristics are the following:

The Bidasoa River has its basin divided between Navarra and Guipuzcoa. It rises in the Lesete peak near the Izpegaray pass. It flows for 50 km in Navarra and for 11 km in Guipuzcoa, forming the border with France until it discharges in the Cantabric Sea (Fuenterrabia). The drainage area is 706 km² (681 km² in Navarra). The most important tributaries are the Ezcurra, Echalar, Marin and Ondalasco (where most of the industrial factories are located).

The Oria is the most important river in Guipuzcoa. It rises near Otzaurte (Aitzgorri mountain) and flows into the Cantabric Sea at the town of Orio. Its length is 63 km, with a catchment basin area of 850 km². Numerous tributaries, such as Araxes, Agaunza, Leizaran, Mutiloa and Estanda, feed into this river and may influence the characteristics of the main river.

The Lea River is quite short (22 km) and located in Vizcaya. It rises in the slopes of Oiz peak (1 026 m) and flows into the Cantabric Sea at the town of Lekeitio. Its elongated basin has an area of 80 km². Along its course many small streams are incorporated, but its main tributaries are the Zulueta, Telleria, Muxo and Murela.

The three rivers have much reduced flows during the summer

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and quite strong flows in autumn and spring. Mean annual rainfall, for the period 1940/41 to 1982/83, is 1 564 mm for the Lea River area, 2 148 mm for the Bidasoa and 1 680 mm for the Oria (CHNE, 1986). The main human activity, along the Lea and Bidasoa Rivers (Ondalasco area was not considered) is agriculture with a few industrial factories in the Lesaca and Vera de Bidasoa areas. Oria River is subject to a high degree of industrial and urban pollution. The most important pollutants include suspended solids, cyanides, Zn, Ni, Cr, solvents, oils and fats. Data published (Basque Government, 1986) attribute 80,6 per cent of the COD content to industrial pollution.

Collection, sieving and chemical analysis

Surface sediment samples were taken at 26 points along the three rivers (ten for Bidasoa/Oria, and six for Lea) using 500 me polyethylene bottles, previously washed with 20% HCl and distilled water. Sediments were dried at low temperature, for 24 to 48 h and later homogenised. Three representative parts were obtained to carry out size distribution and chemical analyses of both total (<2 mm) and fine (<0,063 mm) samples.

For particle size analysis eight fractions were obtained, namely F-1 (>2 mm, rejected), F-2 (2 to 1 mm), F-3 (1 to 0,75 mm), F-4 (0,75 to 0,5 mm), F-5 (0,5 to 0,25 mm), F-6 (0,25 to 0,1 mm), F-7 (0,1 to 0,063 mm) and F-8 (<0,063 mm), and depicted as differential and accumulatives curves. The mean particle diameters (Perry, 1966) were also calculated as functions of volume ($D_{\rm V}$) and surface area ($D_{\rm SA}$) of particles.

Standard analytical methods for soils (ASA-SSSA, 1982), with some modifications, were applied after calibration with a certified sediment sample (NBS, 1988). These methods are summarised in Table 1 and have been described in detail (Ruiz et al., 1991). Conventional statistical techniques were used for data treatment as described by Calvo (1978). The critical value for the

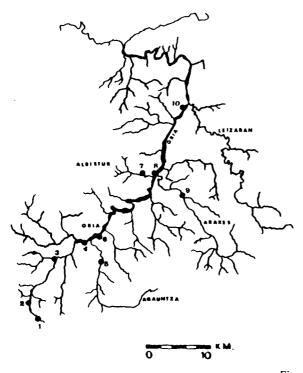


Figure 1
Location of sampling points

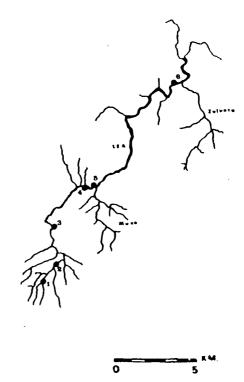


TABLE 1 EXPERIMENTAL METHODS						
Parameter	Procedure	Reference				
Size distribution	Wet sieving	ASA-SSSA (1982)				
Carbonates, as CaCO ₃	Gas evolution	MAPA, 1981				
Organic carbon	Direct titration	MAPA, 1981				
Cation exchange capacity	NH ₄ replacement	MAPA, 1981				
Sample solutions	Wet digestion	FAO, 1979; Zazoski and Burau, 1977;				
	Ç	Christensen et al., 1982				
Major constituents (%):						
Ca, Mg	AAS	•				
Na, K	Flame photometry					
Si	Spectrophotometric. FIA.	Borggaard and Jorgensen, 1985.				
Al	Spectrophotometric.	Rios et al., 1985; Zaki and El-				
		Didamony, 1988.				
Fe	Spectrophotometric. FIA.	Mortatti et al., 1982				
Microconstituents (mg/kg):						
Cu, Mn, Zn	AAS	Zazoski and Burau, 1977; Pagenkopf and Cameron, 1979				
Cr, Pb, Ni, Cd	AAS, graphite furnace	Nielsen and Hrudey, 1984; Ramondetta and Harris, 1978				

Pearson correlation coefficient is $r_{critic} = 0.388$ at 95% confidence level (two-sided).

See also Table 1 for experimental methods.

Results and discussion

Analytical results

Table 2 includes mean values for studied rivers, and average values, standard deviations and extreme values of measured parameters for all collected samples. Individual results (more than one thousand data) are omitted for brevity, but can be obtained from the author on request.

Differences in mean data for each river, including particle size and composition of total or fine fractions, are evident. When total contents are considered in estimating metal pollution: Lea River is almost unpolluted; most of the samples from Bidasoa River are polluted by Pb and two samples from the tributary Ondalasco also by Zn, Mn and Cd, due to industrial activities; and Oria River, with smaller particle size, is the most contaminated one, especially by Pb, Cd, Zn and Cu.

Particle size differences

Mean results of sieve analysis for the studied rivers are shown as accumulative curves in Fig. 2, and it is evident that Oria sediments are finer than the other ones. An additional comparison of particle size, based on average particle diameters derived from volume and surface area, is made in Table 3. Again the smaller size of Oria River sediments can be seen clearly. There are no substantial differences between the sediments of the other two rivers, which have consequently greater sizes than average data.

Composition of fine sample and distribution of chemical constituents according to size particle

From Table 2, it is evident that there is an average enrichment of most of the chemical parameters in the fine sample, relative to the total sample, in the following order of percentages:

$$\begin{split} &C_{\rm org}\left(360\right) > {\rm CEC}\left(285\right) > {\rm Cd}\left(270\right) > {\rm Na}\left(195\right) > \\ &Cr\left(175\right) > {\rm Cu}\left(155\right) > {\rm Mg}\left(145\right) > {\rm K}\left(135\right) > \\ &Zn,\,{\rm Al},\,{\rm CO}_3\left(125\right) > {\rm Ni},\,{\rm Mn}\left(115\right) \end{split}$$

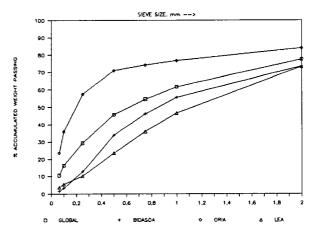


Figure 2
Mean values for studied rivers, average data, standard deviations and intervals of parameters for collected samples

TABLE 2 SIZE DISTRIBUTION AND CHEMICAL COMPOSITION OF STUDIED SEDIMENTS

Size fractions	Bidasoa River	Oria River	Lea River	Average n=26	σ	Maximum	Minimur	
			26,62					
% 2-1 mm	17,89			15,80	12,5	42,5	0,23	
% 1-0,75 mm	9,44	2,50	10,72	7,07	5,17	21,7	0,48	
% 0,75-0,5 mm	12,4	3,24	12,33	8,86	7,14	22,7	0,98	
% 0,5-0,25 mm	21,01	13,42	13,42	16,30	13,6	42,8	0,57	
% 0,25-0,1 mm	9,39	21,79	4,58	13,10	13,6	43,7	0,59	
% 0,1-0,063 mm	1,78	12,17	1,74	5,77	7,6	29,1	0,12	
% <0,063 mm	1,57	23,54	3,77	10,50	17,9	78,2	0,16	
Sample <63 μ								
% C _{org}	3,28	1,61	1,25	2,17	1,46	5,70	0,51	
% Carbonates*	6,97	12,84	8,99	9,69	4,99	24,7	2,05	
% Na	0,50	0,46	1,81	0,79	0,70	2,98	0,15	
% K	2,29	1,66	1,69	1,91	0,86	3,38	1,07	
% Ca	1,04	4,37	1,83	2,50	2,23	7,30	0,34	
% Mg	1,29	0,49	0,52	0,80	0,57	2,75	0,34	
% A1	7,59	6,51	10,11	7,76	2,31	13,3	4,06	
% Fe	3,51	5,69	4,96	4,69	1,98	11,2	1,61	
% Si	22,88	27,66	25,19	25,30	3,59	32,3	18,4	
CEC (meq/100g)**	25,54	13,51	16,85	18,90	8,92	39,0	7,89	
mg/kg Cr	59,18	105	58,95	76,70	37,4	195	30,4	
mg/kg Pb	38,43	64,06	38,58	48,30	27,6	140		
mg/kg Ni	44,32	70,0	33,93	51,80	23,8	130	9,9	
mg/kg Cu	55,96	123,2	27,48	75,20			27,6	
mg/kg Zn	187	336	27,48 171,4	75,20 240	85,7	465	14,1	
mg/kg Mn	972	723,2	171,4	240 920	140	580	9,97	
mg/kg Cd	0,54				380	1660	270	
lig/kg Cu	0,34	0,69	0,27	0,54	0,53	2,7	0,2	
Total sample (<2 mi		1.00	0.04	0.71				
% C _{org}	0,43	1,00	0,24	0,61	0,69	3,38	0,10	
% Carbonates*	1,28	15,2	6,66	7,88	9,05	41	0,38	
% Na	0,34	0,37	0,59	0,41	0,19	0,96	0,17	
% K	1,71	1,30	1,02	1,39	0,43	2,60	0,64	
% Ca	0,48	5,76	3,47	3,20	3,93	13,2	0,04	
% Mg	0,80	0,35	0,53	0,56	0,37	1,56	0,17	
% A1	6,18	7,33	4,21	6,17	3,05	14,1	1,62	
% Fe	4,61	5,71	6,91	5,56	2,73	12,8	1,56	
% Si	32,76	26,89	30,28	29,90	4,40	36,0	15,6	
CEC (meq/100g)**	4,82	8,51	6,68	6,67	3,19	19,3	3,62	
ng/kg Cr	42,73	59,84	20,00	44,10	46,2	262	9,90	
ng/kg Pb	44,78	90,08	11,73	54,60	52,5	250	6,0	
ng/kg Ni	35,94	59,53	33,88	44,50	22,2	110	13,6	
ng/kg Cu	30,78	76,96	32,70	49	40,0	180	14,6	
ng/kg Zn	121	296,3	132,8	190	140	495	38,3	
ng/kg Mn	812	631,5	1320	860	720	289	150	
ng/kg Cd	0,05	0,43	0,07	0,20	0,23	0,80	0,0	

^{*} As CaCO₃
** CEC cation exchange capacity

TABLE 3 MEAN CALCULATED PARTICLE DIAMETERS (mm) FOR STUDIED RIVERS						
	$D_{\mathbf{v}}$	$\mathbf{D_{SA}}$				
Bidasoa	0,12	0,43				
Oria	0,05	0,09				
Lea	0,09	0,38				
Global	0,07	0,18				

Pb
$$(90) > Si$$
, Fe $(85) > Ca (80)$

Such ordering suggests the following comments:

- Very large increments of the organic carbon and the cation exchange capacity for the fine samples.
- Considerable enrichment in the fine sample of metallic microconstituents (all those studied except Pb), clay minerals (as a consequence of increments of Al) and carbonates (but not Ca).
- Relative impoverishment of the contents of Si, Fe and Ca (also Pb) in the fine samples.

In searching for the relation between contituents and particle size, the correlation coefficients between each chemical constituent and the weight percentage of each size fraction were calculated for total (<2 mm) samples from individual data (26 sediments). The relationship becomes apparent when the correlation coefficients of a selected fraction (and related constituents) with all size fractions are depicted versus size fractions, from F-2 (2 to 1 mm) to F-8 (<63 microns). From these graphs, it can be concluded that:

- There are eight parameters (Al, C_{org}, CEC, Pb, Cu, Zn, Cd and probably Ni) whose correlation coefficients with particle size fractions follow an evolution paralled to the correlation coefficients of the finest fractions (F-7 or F-8) (Figs. 3 and 4). This would indicate that the organic matter, clay minerals (related with Al content) and most of the metallic microconstituents should be found in the two finest fractions of the total sample. The cation exchange capacity (depending on the organic carbon and the clay fraction) follows a parallel trend. The behaviour found for most of the microconstituents (Ni, Cu, Cd, Zn and Pb) suggests the possibility of pollution phenomena.
- The distribution of Mn and most of the major constituents of the sediments does not show any statistical relation at 95% confidence level with particle size fractions, except for Mg which is significantly related to the F-4 fraction (0,75 to 0,5 mm) and carbonates, negatively related to the same fraction. This fact (not presented in graphs for the sake of simplicity) suggests an origin mainly natural for the mentioned constituents in the total samples.

Pollution of sediment samples

To estimate pollution in the studied sediments, the increase in the content of a metal relative to suggested conservative elements was rejected, because of very important variations in Al, Fe or Mg contents or their molar accumulations for the first total samples (unpolluted or scarcely contaminated) of the studied rivers, as can be seen in the following data:

This method would require establishing different pollution criteria for each river, or even each tributary.

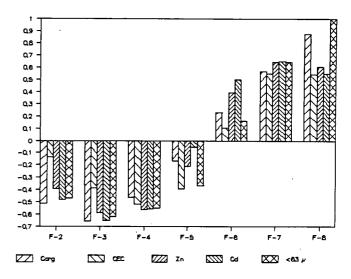
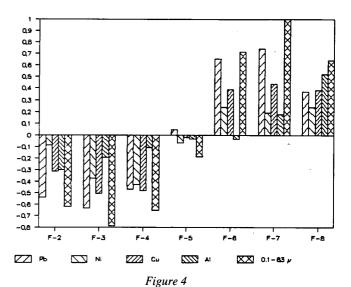


Figure 3
Chemical constituents related to particles <63 µm in total samples



Chemical constituents related to particles with size in the interval 0,1 to 0,063 mm in total samples

The enrichment of a metal in the fine fraction, relative to a conservative element was also tried. According to data included in the former paragraph and considering any major constituent from Mg to Fe as conservative element, average contamination would be expected for the constituents $C_{\rm org}$, Cd, Cr and Cu, following an increase in CEC due to the organic matter content.

This second method was also rejected because almost 50 per cent of the samples with total cadmium content below or close to detection limit (0,01 ppm) had to be classified as contaminated as a consequence of accumulation, possibly natural, of Cd in the finest fractions.

The comparison of each element with a mean natural value seems to be therefore the best method. As average values the amounts in the earth's crust for metals and the content in sedimentary rocks for organic carbon (CRC, 1973-1974) were selected. The following data in parts per million:

TABLE 4
QUALITY INDICES FOR EACH POLLUTANT CONSTITUENT OF SEDIMENTS

				IQS				
Muestra	Cr	Pb	Ni	Cu	Zn	Mn	Cd	C _{org}
Bid-1	89,7	34,6	76,2	72,1	64,4	69,2	100	79,5
Bid-2	77,30	20,00	78,30	73,10	76,50	81,40	100,0	86,80
Bid-3	79,50	21,40	74,30	71,40	63,70	83,80	100,0	68,00
Bid-4	83,60	19,20	73,30	64,80	62,90	41,50	100,0	40,00
Bid-5	86,6	34,40	65,20	69,60	56,70	69,10	100,0	60,6
Bid-6	81,90	43,50	66,90	77,60	69,80	72,40	88,20	63,5
Bid-7	85,00	46,50	64,40	79,70	55,50	75,40	100,00	86,80
Bid-8	83,50	34,00	62,60	73,00	52,60	67,10	83,30	33,00
Bid-9	78,00	16,20	62,20	57,00	25,50	38,30	42,90	71,00
Bid-10	80,50	26,80	70,50	62,90	42,90	27,90	42,90	72,50
Oria-1	88,20	40,70	62,70	73,80	53,00	71,10	33,30	75,00
Oria-2	88,10	35,70	66,50	73,80	48,50	70,90	42,90	75,00
Oria-3	89,00	21,30	69,00	52,80	24,20	54,90	20,00	30,60
Oria-4	43,30	17,60	42,20	35,20	34,40	52,10	27,30	62,90
Oria-5	76,10	10,20	43,20	28,10	21,10	47,60	20,00	62,30
Oria-6	78,60	6,00	55,90	39,10	23,00	54,30	23,10	34,70
Oria-7	90,70	34,30	72,50	82,70	64,60	86,90	60,00	52,40
Oria-8	74,80	12,00	49,10	37,80	21,50	59,20	23,10	16,30
Oria-9	92,00	19,30	66,40	70,90	41,00	68,40	15,80	42,60
Oria-10	84,80	12,70	66,30	46,10	26,70	65,90	33,30	36,30
Lea-1	95,00	65,00	85,40	82,20	77,50	77,60	78,90	65,30
Lea-2	93,90	61,50	71,60	67,80	55,90	66,70	75,00	70,20
Lea-3	92,20	72,70	67,20	63,10	46,80	53,30	100,0	78,60
Lea-4	87,90	71,50	69,90	63,30	33,90	30,80	93,80	81,50
Lea-5	89,60	41,60	65,40	69,20	47,80	47,20	42,90	75,00
Lea-6	87,20	46,60	65,40	66,70	55,90	25,70	60,00	73,30

were used to propose for each constituent an index of quality of sediment (IQS) by the expression:

$$IQS = 100 [1-\{x/(x+av)\}]$$

where:

x = content of a constituent in a particular sample; and av = its average natural value.

The proposed method leads to IQS in the range 0 to 100 with the following values according to the ratio of the content to natural average value:

For calculating a global IQS for a sample sediment, two procedures can be followed, namely:

- To obtain the arithmetic mean value (used in this work)
- To establish differences among constituents by using ponderation factors for each one (not tried in this work)

The application of the proposed method to studied total samples of sediments led to results included in Table 4. By considering

that a sample is contaminated when its content exceeds 1,5 times the natural average (an IQS lower than 40), the following deduction can be made on total samples:

- No contamination was found in the case of two metals, namely Cr and Ni.
- Total sediments of the Lea River are almost unpolluted, with the exception of two samples contaminated by Mn and just one by Zn.
- Bidasoa River is polluted by Pb, Zn and Mn in several samples, particularly in the cases of sediments from the tributary Ondalasco (Samples 9 and 10), where most of the industrial activities are located. A sample (Number 8) is also contaminated by organic matter. This pollution can be attributed to an urban origin, as it is situated after quite a big town, Tolosa.
- Oria River, the most polluted one, has quite a low IQS for Pb, Cu, Zn, Cd and organic carbon in many samples. These results are probably consequences of urban, farming and industrial activities alongside the basin.

For the fine samples (<63 microns) an analogous treatment was carried out because of the probable influence of sediment composition on aquatic life. Condensed results from this calculation, expressed by the interval of variation of IQS and percentage of polluted samples (exceeding 1,5 times natural averages), are the following:

	Bidasoa		Oria	Lea		
Cr	71,8 - 81,3	0	80,8 - 50,5 0	68,9 - 86,8	0	
Pb	16,4 - 61,8	70	10,3 - 35,6 100	24,5 - 37,9	100	
Ni	61,1 - 73,8	0	38,1 - 69,2 10	67,5 - 74,3	0	
Cu	43,6 - 71,8	0	13,0 - 62,6 40	52,9 - 83,2	0	
Zn	21,5 - 57,0	20	18,5 - 47,0 60	33,2 - 52,0	17	
Mn	37,6 - 58,9	20	41,8 - 78,7 0	38,2 - 65,1	33	
Cd	5,3 - 42, 9	60	8,6 - 33,3 100	33,3 - 42,9	67	
C_{org}	10,4 - 40,2	90	14,3 - 41,8 90	26,4 - 56,4	67	
Mean	33,5 - 61,0	33	28,2 - 52,3 50	43,1 - 62,3	35	

Again Oria River has the lower IQS and the greater mean percentage of contaminated fine samples. The percentages of contaminated fine samples are almost coincident for the Lea and Oria Rivers but mean intervals of IQS suggest greater pollution in the case of the Bidasoa River.

Conclusions

Results are included in this paper on three rivers of torrential regime from the NE of Spain. The variations in size particle and composition among samples, even from the same basin, are so great that the treatment of data by conventional methods for estuarine, lake or coastal sediments was not adequate.

For estimating the distribution of chemical constituents among size fractions a method based on correlation coefficients between total content and percentages of size particle fractions was tried. This method may probably be used with success for most sediments.

The comparison with a natural background content was necessary to establish pollution phenomena in this type of river. For that, an index of quality of sediment, based on average earth's crust metal amounts and sedimentary rocks content was designed and applied. Calculated indices agree with considerations derived from presumably urban, agricultural and industrial inputs for the studied zones, and may probably also be applied to most types of sediments.

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