Environ Geol (2007) 52:1581–1593
DOI 10.1007/s00254-006-0602-9

ORIGINAL ARTICLE

Partitioning, bioavailability and origin of heavy metals from the Nador Lagoon sediments (Morocco) as a basis for their management

I. González · E. Águila · E. Galán

Received: 24 July 2006 / Accepted: 28 November 2006 / Published online: 23 December 2006
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Abstract Nador Lagoon sediments show low trace element concentrations, and, in relation to the lagoon geochemical baseline, only some anomalies for As, Cd, Cu and Pb in the NW of the lagoon deserve to be outstanding. The distribution of major, minor and trace elements in the lagoon allows a breakdown in four zones. Between ''Beni Ensar'' and ''Atelouane'' (zone A), a quite confined zone rich in organic matter and S, the most important trace-element anomalies (As, Cd, Co, Cu, Mn, Pb, Zn) were found, mainly around industry and old mining activities. In the surrounding of the city of Nador (zone B), the anomalies correspond to Mn, Cu and Zn. The coastal barrier and Kebdana channel (zone C) show moderately concentrations of Cd, Cr and Ni at specific sites. The less polluted area is the SE of the lagoon (zone D), with no outstanding anomaly. In lagoon sediments, metal bioavailability is very low. The metal partitioning patterns show that Cu, Pb and Zn present a low availability because they are bounded to the residual, non-mobile phases of the sediments. Only in some sites, the fraction was associated with organic matter, which could be liberated easily. Arsenic is concentrated in both the residual phases and the organic matter, the latter being more available. Cadmium is mainly concentrated in some samples in the interchangeable fraction, which could be considered as a potentially toxic element because it is easily released. Concerning the origin of these trace elements, those found in zone A correspond mostly to a natural source by weathering of mount Gourougou volcanic rocks (As, Co, Cu, Pb and Zn), and to an anthropogenic origin (Cd) owing to the presence of industry and old mines. In zone B, contributions of Cu and Zn enter the lagoon through soil weathering and river-borne, and as anthropogenic pollution from urban wastes. In zone C the most important pollutant is Cd deduced to be of anthropogenic origin from the close industry and intensive agriculture area. In spite of the intense socio-economic activities developed in the Nador Lagoon (agriculture, industry, fishing, tourism) trace element concentrations in the sediments are low and with scarce bioavailability. Only the NW sector is relativity polluted because of geogenic features.

Keywords Heavy metals · Geochemistry · Bioavailability · Geoaccumulation index · Lagoon sediments · Nador · Morocco

I. González (✉) · E. Águila · E. Galán
Departamento Cristalografía, Mineralogía y Química Agrícola, Facultad de Química, Apartado 553, Universidad de Sevilla, 41071 Seville, Spain
e-mail: igonza@us.es

Introduction and aims

Lagoonal sediments may be polluted with heavy metals owing to natural processes such as weathering and erosion of adjacent materials, and/or as the result of human activity such as ore and metal processing, leaching from dumps, decantation ponds, fertilizer leaching, atmospheric deposition, animal excretion and urban sewage outflows (Carignan and Nriagu 1985; Förstner 1983; Förstner and Wittmann 1979; Galán 2003; Shahidul and Tanaka 2004). Evidences for this recent increase comes from chronological reconstructions of trace metal depositions in dated lake sediments (Nriagu et al. 1982; Schmidt et al. 2004).

Concerning heavy metal pollution, several studies have stressed the relevance of geochemical partitioning on lagoon sediments to understand metal mobilization, associations, availability, etc. Pioneer studies like those of Förstner and Wittmann (1979) and Salomons and Förstner (1984) provide toxic metals' concentration values (μg/g) for supposedly unpolluted soils and sediments from the Rhine basin. Carignan and Nriagu (1985), Förstner (1983), Boudreau (1999) and Boyle (2001) pointed out four main factors, i.e. pH, redox conditions, organic complex and salinity, which influence the mobility and bioavailability of sediment-bound metals.

Studies carried out on polluted rivers and lakes in several countries (Dinelli et al. 1997; Rasmussen et al. 1998; Soares et al. 1999; Bellucci et al. 2002; Da Silva et al. 2002) show that heavy metals resulting from anthropogenic pollution are either associated with organic matter present in the fine fraction of the sediments, or adsorbed on Fe/Mn oxy-hydroxides, or precipitated as hydroxides, sulphides and carbonates (Manceau et al. 1992; Wen and Allen 1999; Boyle 2001; Bilali et al. 2002; Nikolaidis et al. 2004). Clay minerals have a high capacity for sorbing heavy metals, in particular, Cd, Cu, Pb, Zn and Ni on illite and smectites (Brigatti et al. 1994; Alietti et al. 1995; Galán 2000).

Because of coastal lagoons are the most productive ecosystems in the biosphere and that human activity has given rise to a variety of impacts on these water bodies with the consequent disruption of the natural equilibrium (Shahidul and Tanaka 2004), a better knowledge of the content of pollutants in sediments, their possible sources, and the effect they have on ecosystems are necessary to formulate possible measures for prevention.

This work looks on the environmental problems affecting Nador Lagoon, which is a place of great interest, given the diversity of the aquatic life found there, the fact that it is an area with important economic and social development owing to the fishing, iron and steel and other metallurgical industries located on its shores, in addition to the region's tourism growth in recent years (Mahjoubi 1991; Hamoumi and Irzi 1998; Hamoumi and Benssaou 2001; Chalouan et al. 2001). It is for these reasons that the possible degradation of the area is of concern to its inhabitants, who depend on it for their livelihoods, and to companies and public institutions, which worry about the decline this would mean in economic investment as well as the resulting social problems this would imply.

The specific objectives of this work were (a) the determination and spatial distribution of trace element in sediments of the lagoon and possible tributary streams, (b) the sediment mineralogical characterization in order to identify the different mineral phases to which they are associated, (c) an approach to the real pollution risk of some toxic elements determining their bioavailability and chemical speciation and (d) the possible pollution origins (geogenic and/or anthropogenic)

General features of the study area

Nador Lagoon is situated on the western Mediterranean coast, between Cape Tres Puntas in Melilla (Spain) and the Moroccan district of Kariat Arekman, on the northern tip of the Bou Areg Plain. Its coordinates are 2°45′W–2°55′W longitude and 35°05′N–35°14′N latitude. Besides being the only lagoon on the Mediterranean coast of Morocco, it is also the largest in extension in the country, with a surface area of approximately 115 km^2 (Fig. 1).

Nador Lagoon is located in the Neogene basin of Melilla–Nador, the southern edge of which is formed by a succession of mountain chains belonging to the Rif Mountain Range, which may be of direct influence through its contribution of sediments to the lagoon. In the *Rif Mountain Range* a series of ultramafic massifs of ''alpine type'', essentially made up of lherzolites, are cropping out (Gervilla et al. 1988; Gervilla and Leblanc 1990; Alouan 2001). The multiphase tectonic activity affecting this coast during Neogene and Quaternary times was accompanied by a calc-alkaline vulcanism of shoshonitic affinities (Azdimoussa and Bourgois 1993).

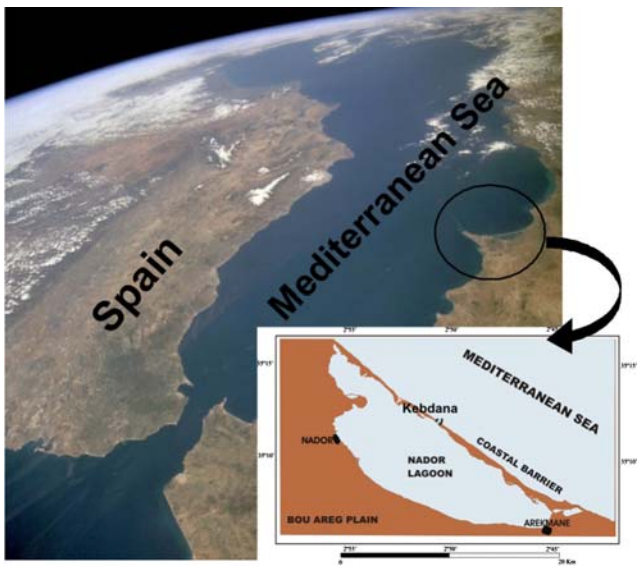

Fig. 1 Location of the study area

The *Melilla–Nador Neogene basin* is a subsident basin which was individualized in the Upper Miocene after the formation of the outer layers of the Rif range. The coastal strip is characterized by a narrow carbonated reef platform which becomes a marly-diatomitic basin laterally towards the south.

The lagoon complex is made up of a number of differentiated zones (Irzi et al. 1997, 1998; Mahjoubi 1991; Irzi 1987, 2002):

The continental fringe (*Bou Areg Plain*) is characterized by salt marshes, several intermittent streams, or wadis (*oued*), some of which presently serve as sewage outflows for urban areas upstream, flow into the marshes.

Nador Lagoon, still called ''*Sebkha de Bou Areg*'' or ''*Mar Chica*'', is a restricted lagoon with a maximum depth of 8 m. It has a semi-elliptic shape and a surface area of 115 km^2, measuring 25 km at its longest point and 7.5 km at its widest.

The coastal barrier is oriented NW–SE and at no point is over 20 m above sea level. It is 25 km in length and 300–400 m in width, reaching 2 km in its southeasternmost part. There is an entrance channel (*the bokhana*), which was recently reinforced with two dikes to ensure connection with the Mediterranean Sea, and remains of earlier openings with well-developed internal deltas (Fig 1).

Nador Lagoon is fed by groundwater from the Bou Areg Plain water table, and by intermittent flows from inland areas through wadis, among which the Selouane Oued is the most notable (large amounts of raw sewage enters the lagoon through it), as well as by irrigation canals coming from Nador, Beni Ensar and Kariat Arekman (Fig. 2).

The presence of a multitude of small, torrential wadis which cut through the massifs of Beni Said, Gourougou and the base of Kebdana is common. Owing to percolation, evaporation and the small amount of water that flows through them, these wadis contribute little water to the lagoon (Inani 1995).

The most important socio-economic activities in the area that are potential sources of pollution are related to the (a) *agricultural sector* (over 60% of the population), dedicated to intensive cereal farming in the southeast, (b) *industrial sector*, located mainly in two areas, Selouane (where the largest iron and steel complex in the country is found) and Nador (with its neighbouring metallurgic industrial area), (c) *fishing sector*, with a fleet of more than 200 ships, several ports and considerable industrial activity related to this sector (fish farming, canning industries) and (d) *tourism sector*, thriving because of the natural attraction of the district—mountains in Gourougou, ''Mar Chica'' beaches and spa resorts in Kariat Arekman—as well as its culture—handicrafts, gastronomy, etc. (Quaranta et al. 2004) (Fig. 3).

Materials and methods

Materials

Two sampling campaigns were performed, one in summer and the other in winter. Samples were taken randomly on the perimeter of the lagoon, more densely in those areas where there was a greater possibility for natural and/or anthropogenic pollutants to be found, such as the mouths of wadis and urban and industrial areas. Lastly, sampling transects perpendicular to the longitudinal axis of the lagoon were made in order to determine the distribution of trace elements in the centre of the lagoon.

In the *summer* of year 2002, a total of 25 lagoonal samples were taken at 0–5 cm (''level 1'') at 25 sampling points. In order to determine the vertical evolution of pollution and possible pollutant sources, a more detailed sampling was carried out in winter of 2003 (Fig. 4). In particular, lagoonal samples were taken at three levels of depth (levels 0, 1 and 2). In order to determine possible trace element sources, stream samples in the proximity of the lagoon (D-I upstream and D-II at the mouth) and some representative soil samples were taken (Table 1).

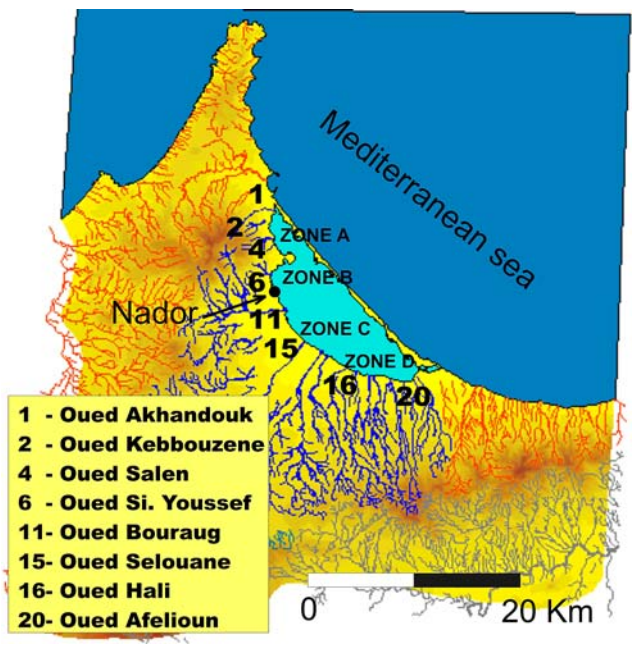

Fig. 2 Main watercourses feeding the Nador Lagoon and zone subdivision

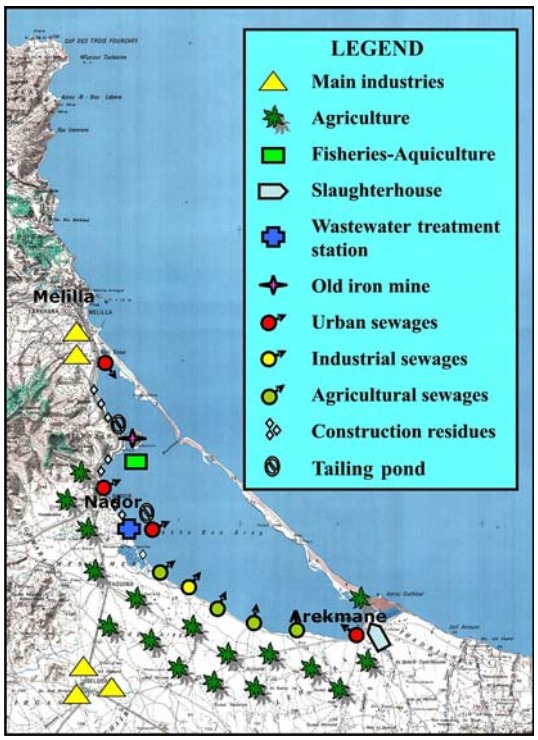

Fig. 3 Most important activities and potential sources of pollution in the Nador Lagoon

Methodology

The pH for samples from the summer and winter campaigns was determined with a solid/water ratio of 1:2.5, i.e., 10 g of sediment sample was mixed with 25 ml of distilled water and shaken for 10 min in a magnetic agitator. The sample was left to stand for 30 min before the pH measurement, which was carried out using a previously calibrated Crison-pH-2002 pH meter.

Chemical analyses of major, minor and trace elements were performed on the fraction < 2 mm (the percentage in the whole sample determined by sieve), by Instrumental Neutron Activation (INAA) and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) at Activation Laboratories Ltd. (1428 Sandhill Drive; Ancaster, Ontario, Canada). Detection limits for the determined elements and the method of analysis used for each element are given in Table 2.

The geochemical baseline of Nador Lagoon was determined by statistical analysis of the data obtained in the study of lagoonal sediments. The median, percentile 90 and percentile 95 were used as parameters. The median was used as the baseline, as it is a much more robust indicator than the mean for data populations without a normal distribution. Moreover, the systematic use of this parameter as representative of a population of geochemical data from natural systems is recommended (Rock 1988), given that it is not influenced by extreme values. The percentiles 90 and 95 have been used as standard risk values in Spain. This analysis was performed for As, Cd, Co, Cr, Cu, Ni, Pb and Zn, elements considered to be potentially toxic by the US Environmental Protection Agency (EPA).

According to the results obtained by chemical analysis of trace elements, the samples with the greatest concentrations, collected in both summer and winter, were selected for mineralogical characterization. This was determined by X-Ray Diffraction (XRD) with a Philips PW-1710 device with automatic slit, using the randon powder method on a flat sample slide. Diagrams from 3° to 60° of 2θ angle were obtained. The fraction < 2 μm was studied by oriented aggregate (OA) using different treatments: solvation with ethylene glycol and with dimethyl sulphoxide, and heating at 550° for 2 h. The interval from 3° to 30° was explored. Percentage estimates for the identified minerals were made for the whole sample and for the fraction < 2 μm, using the reflecting powers proposed

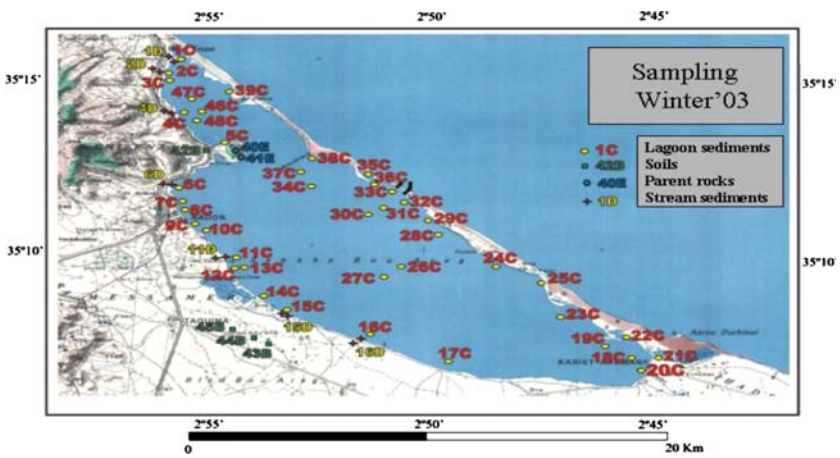

Fig. 4 Location of sampling points of lagoon sediments (*C*), stream sediments (*D*), soils, (*B*) and parent rock (*E*)

Table 1 Sampling and materials studied

Sampling	Sampling type			Sampling point	Number of samples
Summer 2002	Lagoon sediments	Level 1 (0–5 cm)	Core PVC (30 cm)	25	25
Winter 2003	Lagoon sediments	Level 0 (Mud)	Direct sampling	41	34
		Level 1 (0–5 cm)	Core PVC (30 cm)		41
		Level 2 (10–20 cm)	Core PVC (30 cm)		6
	Stream sediments	D-I (0–20 cm)	Direct sampling	15	8
		D-II (0–20 cm)	Direct sampling		7
	Soils	Level 1 (0–25 cm)	Direct sampling	4	4
		Level 2 (25–50 cm)	Direct sampling		4

Table 2 Method of analysis and detection limit in ppm, except where indicated

	ICP-OES													INAA				
Element	Al	Ca	Cd	Cu	K	Mg	Mn	Ni	P	Pb	S	Ti	Zn	As	Co	Cr	Fe	Na
Detection limit	0.01%	0.01%	0.3	1	0.01%	0.01%	1	1	0.001%	3	0.01%	0.01%	1	0.5	1	2	0.01%	0.01%

by different authors and adjusted for automatic grid (Schultz 1964; Barahona 1974; Galán and Martín Vivaldi 1973; Dinelli and Tateo 2001).

On selected anomalous samples (values of some trace elements higher than percentile 90 or 95) metal bioavailability was assessed by extraction of the sample with EDTA 0.05 M (Quevauville et al. 1998) and metal mobility using the Tessier procedure (Tessier et al. 1979).

A correlation analysis between chemical and mineralogical data was carried out in order to assess possible associations between them. The index of geoaccumulation (Igeo) introduced by Muller (1981) was used to measure heavy metal pollution in sediments: Igeo = log 2 (C_n/1.5 B_n), where C_n is the measured concentration of the element "n" in unpolluted sediments. B_n is the average concentration of element n in the background used by Muller (1981). The factor 1.5 is used because of possible variations of the background data due to lithogenic effects. Samples were divided into five groups: unpolluted (Igeo < 1), very low polluted (1 < Igeo < 2), low polluted (2 < Igeo < 3), moderately polluted (3 < Igeo < 4), highly polluted (4 < Igeo < 5) and very highly polluted (Igeo > 5). This index has been used to assess metal enrichment of riverine sediments (Herr and Gray 1997).

Results and discussion

Chemical characterization

For *lagoonal sediments*, the percentage of fraction < 2 mm is high (>60%) in most of the samples, and pH values (~8) are quite homogeneous in the whole lagoon, in samples collected in both summer and winter, and for the different levels at which samples were taken. This pH homogeneity implies that trace element mobility must be conditioned by other factors such as the presence of clay minerals and Fe–Mn oxyhydroxides, or by the solid/liquid ratio effect (Dinelli et al. 1997; Bordas and Bourg 2000; Howari and Banat 2000).

For the *summer sampling*, results of chemical analysis for *major elements* (data not shown) present low content in aluminium (<10%) and calcium (<6%) in almost all the samples from the northern half of the lagoon. In the south, Ca percentages are slightly higher (8–12%), and on occasions over 25%. The highest Fe contents correspond to samples 5SC (44.8%) and 11SC (10.3%). The high sulphur percentage (1.3 and 2.6%) in samples from the northern zone should be pointed out. *Trace element* contents are not significant. As values oscillate between 5 and 16 ppm, the highest being from the central part of the lagoon, and Cd content is lower than 1 ppm. Sample 5SC stands out in that it shows very high concentrations of As (84.5 ppm), Cd (6.2 ppm), Co (120 ppm), Cu (466 ppm), Mn (5,141 ppm), Pb (416 ppm) and Zn (1,190 ppm).

The data obtained in the *winter sampling* have allowed a zone breakdown to be made (Fig. 2) according to major, minor and trace element contents. The highest aluminium contents are found between Beni Ensar and the city of Nador (zones A and B), but in no case is this greater than 15%. In addition, the sulphur is concentrated in the former zone, reaching 2.14% in sample 48°C. This sulphur concentration may be due to organic matter, given that this zone is quite confined.

In zone B, iron is the indicator of geochemical difference with a higher percentage than in the rest of the lagoon. Ca content is in the order of ~20% in samples taken from the zone C, and ~15% in the SE area of the lagoon (zone D).

The distribution of trace elements at *level 0*, confirm that the aforementioned zones are maintained or are even more differentiated (Table 3). Between *Beni Ensar and Atelouane* (zone A), the anomalies encountered are Cd, Co, Cr and Pb. Sample 5SC is once again an exception giving very high concentrations of As (76 ppm), Cd (2 ppm), Co (80 ppm), Cu (384 ppm), Mn (2,520 ppm), Pb (297 ppm) and Zn (821 ppm). The trace element content in this zone is higher than in the rest of the lagoon.

In the surroundings of the *city of Nador* (zone B), the As content is slightly lower than that found in the confined area of NW, although on occasions is over 20 ppm in samples taken from the mouth of the Bouroug wadi (11SC, 12SC and 13SC). The anomalies present in this zone correspond to Mn, Cu and Zn. *The coastal barrier and the Kebdana channel* (zone C) shows moderately concentrations at specific points of Cd (2.7 ppm), Cr (120 ppm) and Ni (62 ppm) in sample 16SC, and of Cd (2.4 ppm) in sample 30SC. *SE of the lagoon* (zone D) there are no outstanding values except sample 20SC (2.1 ppm Cd).

Trace element values for *level 1* show a similar distribution, but with lower concentrations than in level 0, except samples 46(0–5)C, 47(0–5)C and 48(0–5)C where As is over 30 ppm (Table 4).

The *level 2* samples were taken from the area designated as zone C. In general, the fine fraction and pH show a slight increase with regard to what has been described for levels 0 and 1. It is to be noted the arsenic values which are above 15 ppm in all the samples, reaching 37.6 ppm in 31(10–14)C.

The statistical study has also allowed *the geochemical baseline* of the sediments in the lagoon to be defined Table 5). In general, Nador Lagoon does not present important heavy metal or arsenic anomalies; in fact the values determined as geochemical baseline are lower than those found by other authors (Salomons and Förstner 1984;Wright and Mason 1999; Salminen and Gregorauskiene 2000).

According to geochemical baseline, in the *summer 2002*, only sample 5SC can be considered polluted (>percentile 90 value). Between Beni Ensar and the city of Nador (zones A and B) only isolated samples were found to be above the percentile 90 value. Arsenic in sample 31SC from zone C, and Cr and Ni in sample 25SC from zone D are also above this percentile. In general, anomalous values for studied samples from the lagoon are very low.

In *winter 2003*, As, Cd, Co, Cr, Pb and Zn values are above the median in zone A. The same occurs with Zn, Cu and Mn in the area around the city of Nador (zone B). In both zones values close to percentile 90 predominate and several are even above it. Between Selouane and Arekmane (zones C and D) only three samples are found to be above the percentile 95 for Cd. Values obtained from the majority of samples from these zones are below the median (Tables 3, 4).

In summary, values for trace elements in winter are slightly higher than in summer and with a very similar spatial distribution (Tables 3, 4, 5). This seasonal variation has been described by other authors for estuarine sediments in England (Wright and Mason 1999). Out of the samples collected in July 2002, only sample 5SC can be considered polluted, and in the samples corresponding to February 2003, those from the northern area present As, Cd, Co, Cr, Pb and Zn

Table 3 Trace element contents and statistical values of lagoon sediments samples calculated at level 0. February 2003

	Sample	As	Cd	Co	Cr	Cu	Mn	Pb	Ni	Zn
ZONE A	1SC	19.4	1.7	15	72	39	406	ND	34	61
	2SC	3.9	0.9	22	59	47	624	67	25	120
	3SC	5.4	ND	24	64	44	797	71	35	163
	4SC	9.3	1.2	34	139	59	773	39	57	121
	5SC	75.6	2.0	80	50	384	2520	297	28	821
	46SC	15.8	ND	24	39	96	801	77	15	211
	47SC	13.1	ND	16	52	59	335	57	31	116
	48SC	7.7	ND	19	71	52	320	54	32	119
ZONE B	6SC	9	ND	19	96	55	679	52	28	110
	7SC	4.8	0.9	15	80	45	673	62	18	139
	8SC	11.7	0.8	19	29	61	729	61	8	161
	9SC	13.8	1.2	23	61	64	476	66	34	215
	10SC	15.6	ND	22	36	68	657	56	20	187
	11SC	19.9	ND	3	44	4	206	15	26	16
	12SC	20	ND	26	36	99	873	62	18	202
	13SC	24.5	ND	25	64	79	910	65	29	181
	14SC	14.2	ND	17	42	37	800	32	21	104
	15SC	7.5	0.4	11	52	21	497	22	18	68
ZONE C	16SC	6.6	2.7	9	120	17	359	ND	62	51
	17SC	11.9	ND	14	77	34	414	37	36	92
	26SC	12.2	ND	15	81	39	721	56	29	112
	27SC	10.3	ND	16	76	40	635	41	36	106
	28SC	11.2	0.5	4	36	10	322	11	31	24
	29SC	11.4	ND	4	22	4	310	16	12	20
	30SC	16.9	2.4	13	84	25	619	ND	49	56
	31SC	14.6	0.5	7	37	14	391	26	19	44
	32SC	13.9	ND	2	30	5	263	ND	17	16
	34SC	13.4	ND	13	68	35	627	53	29	94
	37SC	11.3	ND	4	41	10	197	22	25	23
ZONE D	18SC	8.2	ND	15	87	34	473	28	36	81
	19SC	15.2	0.6	7	33	13	231	14	16	32
	20SC	4.1	2.1	12	85	27	322	ND	50	56
	21SC	9.5	1.1	4	38	4	183	14	23	15
	24SC	11.7	ND	5	37	16	261	26	21	38

ND: Not detected.

≤ 50%	50%<P90≤ 90%	90<P95≤ 95%	> 95%

Table 4 Trace element contents and statistical values of lagoon sediments samples calculated at level 1. February 2003

	Sample	As	Cd	Co	Cr	Cu	Mn	Ni	Pb	Zn
ZONE A	1(0-5)C	15.9	0.3	10	110	31	372	66	ND	49
	2(0-5)C	10.4	ND	28	73	43	416	30	57	114
	3(0-5)C	6.4	ND	28	66	45	619	33	57	113
	4(0-5)C	7.5	ND	27	75	58	960	35	34	117
	39(0-5)C	13.9	ND	7	42	9	196	28	7	23
	46(0-5)C	35.7	ND	17	50	48	473	30	12	74
	47(0-5)C	31.1	ND	18	108	39	401	60	16	72
	48(0-5)C	33.7	ND	28	75	77	533	32	29	145
ZONE B	6(0-5)C	20.2	ND	28	100	60	569	42	18	93
	7(0-5)C	10	ND	16	80	53	535	27	54	149
	8(0-5)C	10.5	ND	16	91	46	470	51	76	174
	9(0-5)C	12.2	0.5	18	38	56	504	27	57	190
	10(0-5)C	16.4	ND	23	93	57	522	57	23	143
	11(0-5)C	18.1	ND	27	49	94	700	29	52	194
	12(0-5)C	16	0.7	24	43	84	894	24	65	192
	13(0-5)C	15.6	0.3	23	53	62	699	21	52	147
	14(0-5)C	10.5	0.7	18	46	43	658	15	35	113
	15(0-5)C	8.5	0.4	11	55	19	384	4	43	38
ZONE C	16(0-5)C	7.3	ND	10	66	17	351	13	42	43
	17(0-5)C	18.4	ND	23	88	64	496	37	54	128
	26(0-5)C	18.6	1.7	16	82	39	598	44	55	84
	27(0-5)C	19.1	0.8	14	71	51	652	33	60	96
	28(0-5)C	18.7	ND	7	31	10	284	6	35	ND
	29(0-5)C	12.8	0.4	7	39	7	275	13	42	ND
	30(0-5)C	26.9	ND	16	59	37	700	36	29	82
	31(0-5)C	18.6	ND	7	38	10	349	43	15	26
	32(0-5)C	21.3	ND	4	25	5	274	38	6	1
	33(0-5)C	17.1	ND	3	31	2	175	35	ND	ND
	34(0-5)C	15.2	ND	12	64	28	560	38	19	69
	35(0-5)C	18.8	ND	10	36	10	238	44	ND	9
	36(0-5)C	15.3	ND	4	47	3	191	47	5	2
	37(0-5)C	12.8	0.4	4	23	5	170	26	ND	4
	38(0-5)C	14.3	ND	4	34	3	181	33	ND	3
ZONE D	18(0-5)C	13.2	ND	17	65	31	384	21	31	53
	19(0-5)C	18.1	ND	8	33	15	247	10	22	7
	20(0-5)C	9.3	ND	14	76	26	493	12	22	57
	21(0-5)C	15	ND	8	44	20	357	14	29	28
	22(0-5)C	9.6	ND	5	20	4	186	ND	29	ND
	23(0-5)C	15.2	0.4	10	38	28	299	1	33	18
	24(0-5)C	16.8	ND	8	38	19	286	5	38	12
	25(0-5)C	8.2	0.4	6	33	10	380	ND	30	2

ND: Not detected.

≤ 50%	50%<P90≤ 90%	90<P95≤ 95%	> 95%

values above the median. The same occurs with Zn, Cu and Mn around the city of Nador. Sample 16SC, found in an area of intensive agriculture close to Selouane, gives anomalous values for Cr, Ni and Cd.

For *stream sediments* the pH oscillates between 7.6 and 8.9, and in general the fraction < 2 mm is higher (>70%) than in the lagoon sediments. No significant differences between sampling points I (upstream) and II (close to the mouth) were found in these sediments. Aluminium percentages are over 10% in most of the samples for wadis that enter the lagoon in zones A and B. This is due to the lithology of the Gourougou volcanic complex through which these streams drained. Ca percentages are very low in these zones and rise to 25% in zone C samples owing, in this latter case, to the fact that streams pass through sediments with abundant carbonate materials. Fe is not above 6% in any case and K is over 3% in almost all the samples from zones A and B, the lowest readings being in the zones where the wadis cross Tertiary sediments.

These results may indicate that major elements are brought into the lagoon by streams, and, moreover, are

Table 5 Statistical values for the samplings of July 2002 and February 2003

	Sampling of July 2002			Sampling of February 2003		
	Median	Percentile 90	Percentile 95	Median	Percentile 90	Percentile 95
As	11.4	16.5	16.6	13.9	20.2	31.1
Cd	0.3	1.0	1.2	0.3	1.2	2.0
Co	14.0	26.0	27.0	15.0	27.0	28.0
Cr	57.0	77.0	85.0	52.0	91.0	108.0
Cu	39.8	81.5	94.3	34.5	68.4	94.2
Mn	577.5	826.4	920.4	469.6	797.0	894.4
Ni	25.8	30.4	35.2	28.5	48.8	57.3
Pb	37.1	67.4	73.4	31.7	65.2	70.9
Zn	95.8	189.0	212.3	74.2	187.0	202.0

Table 6 Trace element content (ppm) in stream sediments. February 2003

Zones	Samples	As	Cd	Co	Cr	Cu	Ni	Pb	Zn
Zone A	1SD-I	7.3	ND	21	45	65	23	92	221
	1SD-II	4.5	ND	21	37	55	32	40	150
	2SD-I	8.6	ND	35	67	47	19	40	123
	2SD-II	6.3	ND	30	65	43	26	57	84
	4SD-I	7.1	ND	31	83	62	42	25	81
	4SD-II	5.2	ND	30	72	55	28	44	109
Zone B	6SD-I	6	ND	25	76	52	41	31	96
	6SD-II	6.8	ND	25	75	62	38	57	149
	11SD-I	12.2	2.0	20	101	95	42	88	254
	11SD-II	4.9	ND	30	69	60	37	25	113
	15SD-I	6.3	ND	10	83	18	33	27	60
	15SD-II	6.6	ND	10	33	7	17	18	25
Zone C	16SD-I	7.8	3.6	8	132	15	86	ND	45
	16SD-II	9.6	ND	8	53	7	25	7	38
Zone D	20SD-I	7	ND	16	98	30	40	22	75

ND not detected

totally linked to the type of lithology through which these streams pass, volcanic rocks in the northern area and marshy sediments to the south.

No significant values of the studied trace elements have been found (Table 6). In *zone A* there is no high value for any trace element analysed, and given that the heavy metals' distribution in lagoonal sediments are related to the detritus input owing the nature of the source areas, it can be deduced that the high As, Cd and Co values found in lagoonal sediments in this zone were not carried by these wadis. In *zone B* isolated anomalies were found for Cd, Cu and Zn (sample 11D-I), and the same occurred in *zone C* for Cd, Cr and Ni (sample 16D-I). A comparison of these data with the values obtained for lagoonal sediments, allows to deduce that some of these high trace element contents in zones B and C may be due to wadi-borne sediments. A similar moderate Cd pollution also was encountered by Howari and Banat (2000) in Jordan and Yarmouk river sediments. In *zone D* no outstanding anomalies appear in either lagoonal or stream sediments (Table 6).

In *soil samples*, the pH is higher than 8 and the percentage of fraction < 2 mm is very high. The soil taken from the vicinity of the Gourougou volcanic complex (zone A) differs from the rest in that it presents a very high Fe content (43.4%) and low calcium content, which is logical owing to the lithological characteristics of the area. Agricultural soils 43B, 44B and 45B share a similar composition with Al and Ca percentages between 11 and 14% and low iron content. With regard to trace elements (Table 7), soils from zone A give high values in As (68.2 ppm), Co (96 ppm), Mn (4,254 ppm), Cu (586 ppm), Zn (646 ppm) and Pb (300 ppm). It is therefore possible that erosion releases these elements and may be the source of those found in the lagoon, as occurs in the study by Johansson et al. (1995) where soils and sediments in Sweden show similar contents and distribution for trace element concentration. These results for this zone agree with the existing mineralizations of chromite-Ni arsenide and Ni–Fe–Cu sulphide ores exist in the lherzolites (Gervilla and Leblanc 1990). Soils

from zone C present high Cd contents, and they may therefore be deduced as the direct cause for the values found in this area of the lagoon. The same cannot be said for Cr and Ni, as they do not appear in the studied samples from this zone (Table 7).

Mineralogical characterization

Samples from the northern zone of the lagoon show an overall mineralogy made up of high percentages of feldspars and iron oxides. The phyllosilicate content varies from 5 to 35%. For sample 5SC, taken from the vicinity of former mine waste decant ponds, a semi-quantitative estimate could not be made owing to the high percentage of amorphous iron oxides. However, quartz, feldspars, phyllosilicates, hematite, magnetite and jarosite were identified.

The mineralogical association for *zone A* is feldspars > phyllosilicates > quartz, with dolomite, ilmenite, hematite, calcite, halite and magnetite as minor components. For the fraction < 2 μm, smectites are the major minerals followed by illite and chlorite + kaolinite in small proportions.

Zone B samples show greater feldspar and quartz contents and lower phyllosilicate content than the preceding zone. With regard to mineralogy of fraction <2 μm, the association is illite > smectites > kaolinite + chlorite.

Zone C samples show a significant increase in calcite. The mineralogical association is calcite ≥ quartz > phyllosilicates, and feldspars, dolomite, hematite, halite and magnetite as minor components. Illite > chlorite > kaolinite > smectites were found in fraction <2 μm.

Zone D shows a high calcite content. Major minerals are calcite ≫ phyllosilicates > quartz, with minor proportions of feldspars, dolomite, halite, hematite and magnetite. The mineralogy of clays is similar to that of zone C (illite > chlorite > kaolinite > smectites).

Statistical study

In order to make a preliminary assessment of the mineral phases the trace elements are associated with, a *correlation analysis* between aluminium (essentially contributed by phyllosilicates and feldspars) and iron (contributed by iron oxides, fundamentally) was made. The results obtained indicate that high correlation coefficients exist between Pb, Zn, Co and Cu with Fe in lagoon sediments instead there is no correlation with Al. These correlations are very similar to those obtained by Gobeil et al. (1999) and Bilali et al. (2002) from sediments from the Arctic Ocean basin and Lake Ontario (Canada).

Trace element anomalies in *zone A* must be related with phyllosilicates and/or iron oxides. Pb, Zn, Cu and Co would be associated with iron oxides, and probably Cr and Co to phyllosilicates (Bilali et al. 2002), but in the latter case the correlation would not be clear owing to the low percentage of these minerals in the zone.

It should be pointed that in none of the studied zones are As and Cd related to Al and Fe, and therefore their origin may be deduced as anthropogenic, or as isolated cases eroded from nearby materials but which have not been adsorbed/absorbed by iron oxy-hydroxides or phyllosilicates.

Metal mobility and bioavailability

Lagoon sediment samples with anomalies of Cu, Pb and Zn show a low availability of these elements because they are bounded to the residual, non-mobile, phase of the sediment (Fig. 5). Only such elements can be considered as available in samples from wadi, where they are associated to organic matter. Pb may be liberated easily from soil sample 42B, because it is bounded to organic matter or Fe-hydroxides. The bioavailability of trace elements is very low for all the cases studied. Only it is to be noted the extraction of

Table 7 Trace element content (ppm) in soils. February 2003

Zones	Samples	As	Cd	Co	Cr	Cu	Ni	Pb	Zn
Zone A	42(0–25)B	5.6	0.4	22	56	62	30	69	151
	42(25–50)B	68.2	0.5	96	58	586	18	300	646
Zone C	43(0–25)B	10.4	1.1	15	102	33	43	ND	142
	43(25–50)B	10.2	1.9	14	86	31	41	10	178
	44(0–25)B	11.6	2.4	14	99	44	43	5	94
	44(25–50)B	11.5	1.2	13	100	32	36	16	157
	45(0–25)B	14.7	2.9	21	140	37	54	20	ND
	45(25–50)B	10.1	2.9	15	115	30	40	11	117

ND not detected

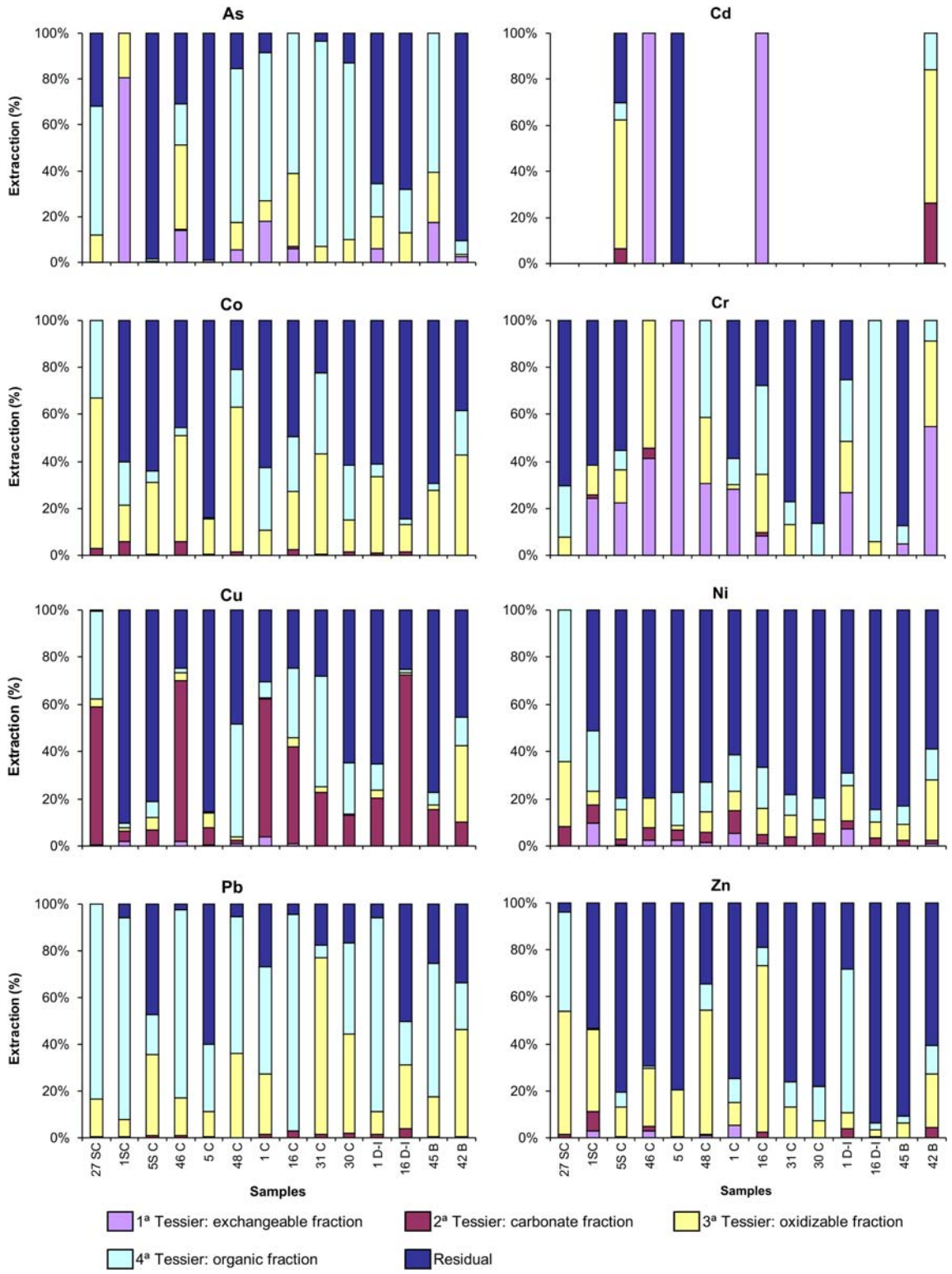

Fig. 5 Geochemical partitioning of trace elements

Cu from soil sample 42B (≈30%) and Pb from sediment sample 5C (≈39%).

It is important to note the binding of As to sediment minerals. Only in samples 5C and 48(0–5) C (zone A) show a notable concentration of arsenic (75 and 33 ppm), but it is bound to the residual fraction. A high As concentration in level 1 (26–37 ppm) can be a problem because it is bound to organic matter and

Table 8 Index of geoaccumulation for sampling February 2003 at level 0

ZONES	Sample	As	Cd	Co	Cr	Cu	Mn	Ni	Pb	Zn
	L-0	I-geo	I-geo	I-geo	I-geo	I-geo	I-geo	I-geo	I-geo	I-geo
ZONE A	1SC	-0.87	1.92	-0.58	-0.12	-0.42	-0.79	-0.34	-	-0.89
	2SC	-2.75	0.85	-0.03	-0.61	-0.15	-0.17	-0.79	0.48	0.11
	3SC	-1.95	-	0.09	-0.29	-0.22	0.02	-0.30	0.58	0.55
	4SC	-4.00	1.31	0.60	0.75	-0.01	0.13	0.42	-0.32	0.12
	5SC	1.86	1.38	1.83	-0.64	2.89	1.84	-0.60	2.19	2.88
	46SC	-0.40	-	0.09	-1.33	-1.73	0.19	-1.47	0.69	0.92
	47SC	-0.67	-	-0.49	-0.58	0.19	-1.07	-0.47	0.26	0.06
	48SC	-1.44	-	-1.03	-0.14	-0.02	-1.14	-0.44	0.18	0.00
ZONE B	6SC	-4.38	-	-1.03	-2.29	0.08	-0.05	-0.59	0.13	-0.02
	7SC	-2.12	0.98	-0.58	0.04	-0.20	-0.07	-1.24	0.36	0.24
	8SC	-0.83	0.66	-1.03	-1.89	0.23	0.05	-2.37	0.35	0.53
	9SC	-0.60	1.42	0.03	-0.35	0.30	-0.57	-0.34	0.47	-2.36
	10SC	-0.42	-	-0.03	-1.12	0.40	-0.10	-1.89	0.23	0.75
	11SC	-0.40	-	-2.91	-0.83	-3.82	-1.78	-0.71	-4.66	-2.88
	12SC	-0.06	-	0.21	-1.12	-2.18	0.31	-1.26	0.38	0.86
	13SC	0.23	-	0.15	-0.29	0.47	-2.79	-0.58	0.46	0.70
	14SC	-4.12	-	-0.40	-0.89	-0.50	0.18	-1.05	-0.58	-0.10
	15SC	-1.48	-0.51	-1.03	-0.58	-1.28	-0.76	-1.27	-1.11	-0.71
ZONE C	16SC	-1.66	2.54	-4.49	0.62	-1.62	-0.98	0.54	-	-1.13
	17SC	-0.91	-	-4.49	-0.02	-0.60	-4.10	-0.26	-0.40	-3.21
	26SC	-0.77	-	-0.58	0.05	-0.74	0.03	-0.58	0.25	0.01
	27SC	-1.02	-	-0.49	-0.04	-0.36	-0.15	-0.23	-0.22	-0.07
	28SC	-0.90	0.08	-2.49	-1.12	-4.80	-1.13	-0.46	-2.15	-2.21
	29SC	-0.87	-	-2.49	-1.83	-3.60	-1.18	-1.83	-1.54	-2.45
	30SC	-0.37	2.39	-0.79	0.11	-1.03	-0.20	0.19	-	-0.98
	31SC	-3.70	0.04	-1.68	-1.08	-5.40	-1.18	-1.18	-0.89	-1.34
	32SC	-0.67	-	-3.49	-1.38	-3.29	-1.42	-1.30	-	-2.80
	34SC	-0.64	-	-0.79	-0.20	-0.58	-0.17	-1.01	0.15	-6.80
	37SC	-0.88	-	-2.49	-0.93	-2.31	-2.59	-0.77	-1.14	-2.27
ZONE D	18SC	-1.35	-	-0.58	0.16	-0.63	-0.57	-0.24	-0.77	-0.46
	19SC	-0.46	0.37	-1.68	-1.24	-1.95	-1.61	-1.38	-5.53	-1.80
	20SC	-2.35	2.23	-0.91	0.12	-0.93	-1.13	-0.04	-	-0.98
	21SC	-3.80	1.23	-2.49	-1.04	-4.06	-1.94	-0.88	-1.80	-2.89
	24SC	-0.83	-	-2.17	-1.08	-1.77	-1.44	-1.04	-0.92	-1.55

Igeo < 1 --> Unpolluted
1 < Igeo < 2 --> Very low polluted
2 < Igeo < 3 --> Low polluted
3 < Igeo < 4 --> Moderately polluted

could be leached under degradation. Also Cd could be a problem in sample 16C because it is in the interchangeable fraction (Fig. 5).

In general, in these sediments most trace elements are associated to Fe amorphous oxy-hydroxides, or take part of the residual fraction. A similar distribution was found by Kersten and Förstner (1989) and Howari and Banat (2000) for the Jordan River sediments, and Da Silva et al. (2002) for the Brazil's river sediments.

Geoaccumulation index

Values obtained for the geoaccumulation index are generally <1 for the whole lagoon, which may indicate that it is not polluted. Sample 5SC is an exception as it reached level 4 (moderately polluted) in both sample collections. Cd is the only element which appears with a geoaccumulation index < 2 (very low polluted) for the summer sampling in zone B, and with an Igeo < 3 (low polluted) for in some samples winter, especially from level 0 (Table 8). In the levels 1 and 2, the Igeo values are lower than 1.

Conclusions

The studied sediment samples from Nador Lagoon show low concentrations of trace elements. The geoaccumulation indices indicate that the sediments are unpolluted. Only a few samples have been found with anomalous As, Cd, Cu and Pb values.

No significant differences were found between results obtained from sample campaign carried out in summer and winter, nor are there any variations with depth as far as trace element content is concerned.

The distribution of trace elements and chemical and mineralogical composition data, have allowed four areas in Nador or lagoon to be differentiated: *Beni Ensar-Atelouane Zone* (zone A), *Nador Zone* (zone B), *Centre-Kebdana Channel* (zone C) and *SE* (zone D).

The chemical composition of *stream sediments* is completely conditioned by the lithology of the drainage area. The streams which enter the lagoon in *zone A* cannot be considered sources of As, Cd and Co pollution, owing to the insignificant amounts present in samples, but it is possible that they *contribute with Cr and Pb*, *because they are bounded to organic matter*. On the contrary, those that enter the lagoon in *zones B and C* can be considered sources of pollution owing to the *Cu–Zn* and *Cr–Ni–Cd* values obtained, respectively, in sediments from wadis.

There is a clear difference between *soils* from the northern area (Gourougou volcanic complex) and the southern, agricultural area (Tertiary marls). Zone A soils could also be a source of As, Co, Mn and Pb, owing to the high values of these elements in soils, but as they are concentrated in the residual fraction, their availability will be very low.

Concerning possible sources of pollution both a natural and anthropogenic origins occurred. In *zone A*, As, Co, Cu and Pb derived from materials of Mount Gourougou by soil weathering and erosion, and Cd has an anthropogenic origin owing to the presence of industry and former mines. In *zone B*, contributions of Cu and Zn, both through soil weathering and riverborne, are *anthropogenic* pollution, given that urban waste outflows in the area are coming from the diverse human activities carried out on its shores. In *zone C*, the most important pollutant Cd is deduced to be of *anthropogenic* origin as the watercourses by which it enters the lagoon pass through areas of industry and intensive agriculture.

Acknowledgments The authors thank an anonymous reviewer and the associate editor Dr J. Schwarzbauer for their critical reading of the paper. The financial support of the European Union (project ICA3 CT-2002–10012 COLASU, ''Sustainability of Mediterranean Coastal Lagoon Ecosystems under semiarid Climate'') is gratefully acknowledged. Also, the authors thank Prof. Naima Hamoumi (University of Rabat) and her team for supplying samples for this study in the frame of COLASU project (http://www.colasu.com).

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