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Comparative sediment quality assessment in different littoral ecosystems from Spain (Gulf of Cadiz) and Brazil (Santos and São Vicente estuarine system)

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Abstract

The goal of this work was to establish comparisons among environmental degradation in different areas from Southern Spain (Gulf of Cádiz) and Brazil (Santos and São Vicente estuary), by using principal component analyses (PCA) to integrate sediment toxicity (amphipods mortality) and chemical–physical data (Zn, Cd, Pb, Cu, Ni, Co, V, PCBs, PAHs concentrations, OC and fines contents). The results of PCA extraction of Spanish data showed that Bay of Cádiz, CA-1 did not present contamination or degradation; CA-2 exhibited contamination by PCBs, however it was not related to the amphipods mortality. *Ría* of Huelva was the most impacted site, showing contamination caused principally by hydrocarbons, in HV-1 and HV-2, but heavy metals were also important contaminants at HV-1, HV-2 and HV-3. Algeciras Bay was considered as not degraded in GR-3 and -4, but in GR-3′ high contamination by PAHs was found. In the Brazilian area, the most degraded sediments were found in the stations situated at the inner parts of the estuary (SSV-2, SSV-3, and SSV-4), followed by SSV-6, which is close to the Submarine Sewage Outfall of Santos — SSOS. Sediments from SSV-1 and SSV-5 did not present chemical contamination, organic contamination or significant amphipod mortality. The results of this investigation showed that both countries present environmental degradation related to PAHs: in Spain, at *Ría* of Huelva and Gudarranque river's estuary areas; and in Brasil, in the internal portion of the Santos and São Vicente estuary. The same situation is found for heavy metals, since all of the identified metals are related to toxicity in the studied areas, with few exceptions (V for both Brazil and Spain, and Cd and Co for Brazilian areas). The contamination by PCBs is more serious for Santos and São Vicente estuary than for the investigated areas in Gulf of Cádiz, where such compound did not relate to the toxicity.

Keywords: Sediment toxicity; Sediment contamination; Estuary; Multivariate analysis

1. Introduction

Most of the anthropogenic chemicals and waste materials, including toxic organic and inorganic chemicals, contribute to the degradation of aquatic environments. This is particularly important for the coastal and estuarine ecosystems around the world, which are constantly affected by multiple contamination sources.

Sediments may accumulate contaminants in concentrations higher than those observed in the water column, producing negative effects to the benthic biota and to the organisms that feed on the benthos or on the sediment. Due to the ecological importance and the persistence of pollutants in this environmental compartment, sediments are more appropriate to be monitored in environmental evaluations (Swartz et al., 1982).

Many different approaches can be used in the sediment quality assessment. Among them, chemical analyses and toxicity tests are the most used around the world (Acosta and

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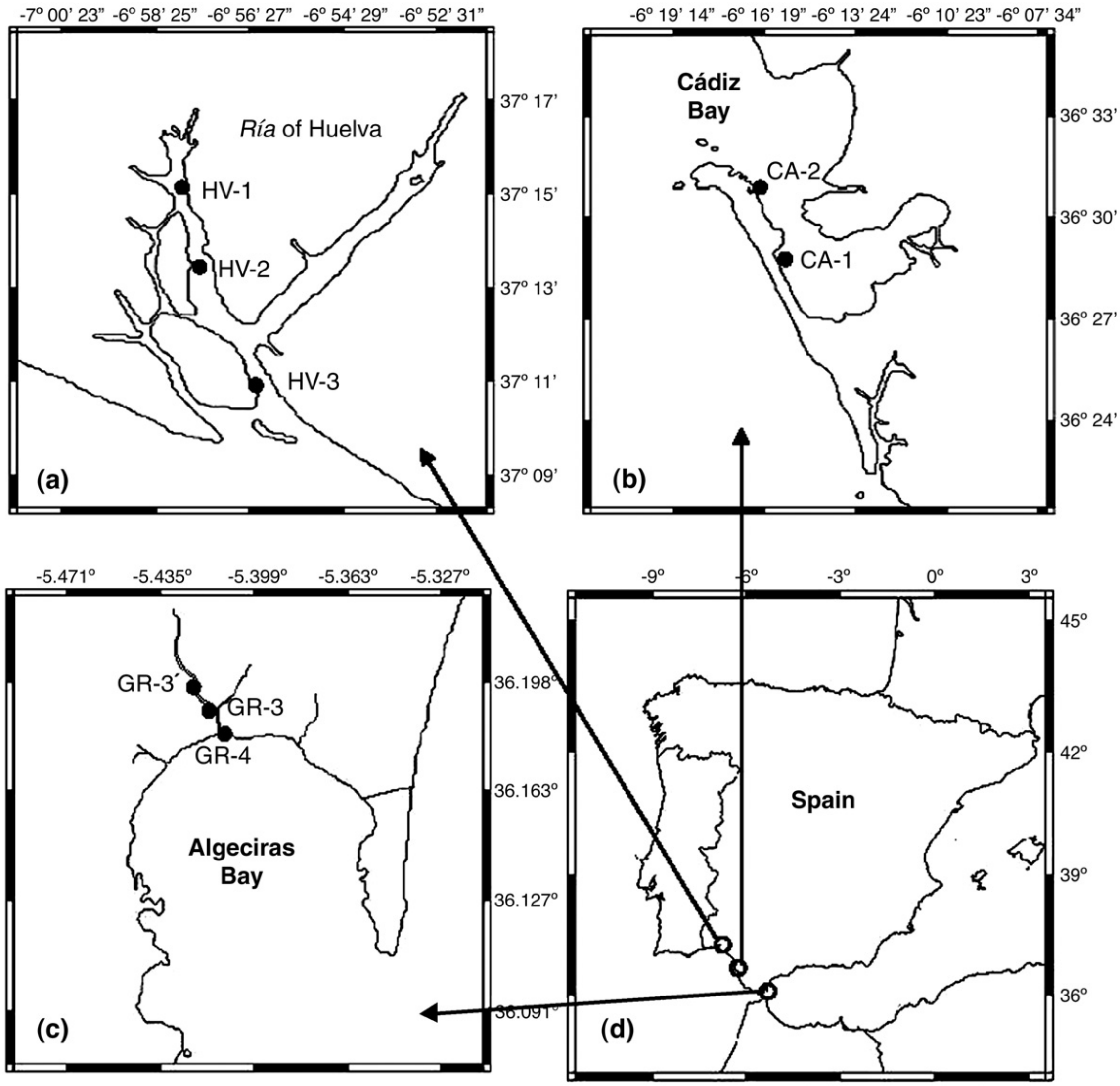

Fig. 1. Localization of the sampling stations in *Ría* of Huelva (a), Cádiz Bay (b), Algeciras Bay (c) and their disposition in Southern Spain (d).

Lodeiros, 2004; León et al., 2004; Evangelista et al., 2005). Such approaches, when applied alone, may result in lack of realism and/or great uncertainties; but when they are used in an integrative way, more reliable information about the environmental condition is provided.

There are several studies utilizing principal component analysis (PCA) to integrate environmental data (Riba et al., 2004a,b; DelValls et al., 2002; DelValls and Chapman, 1998). Thus, by using such multivariate tool, the goal of this work was to determine the environmental degradation in different coastal areas from Spain and Brazil, which are affected by different contamination sources.

Three areas from the Gulf of Cádiz, Southern Spain, were studied: *Ría* of Huelva, Bay of Cádiz, and Guadarranque River's estuary, in the Bay of Algeciras. *Ría* of Huelva and Bay of Algeciras are affected by industrial and harbour activities, whereas Bay of Cádiz is considered a low contaminated area, according to some recent studies (Riba et al., 2004a,b). In Brazil, the studied area was the Santos and São Vicente estuarine system, located in the São Paulo State, South-Eastern Brazil. This area comprises a dense urbanization area, the biggest Brazilian industrial complex – with predominant presence of petrochemical, siderurgy, and fertilizers industries – and also the major Latin American port, so called Port of Santos. There is a vast literature showing high sediment contamination and toxicity in this area (Cesar et al., 2006; Abessa, 2002; Abessa et al., 2001; Lamparelli et al., 2001; CETESB, 1985, 1978).

2. Materials and methods

2.1. Approach

This study was carried out in 8 sediment sampling sites distributed in 3 different regions (*Ría* of Huelva, Bay of Cádiz, and Guadarranque River's mouth) in Southern Spain (Fig. 1); and 6 sediment sampling sites distributed along the Santos and São Vicente estuarine system, in the Brazilian coast (Fig. 2). In Spain, 3 sampling sites were located in *Ría* of Huelva (HV-1, HV-2 and HV-3), an ancient mining area where nowadays an industrial zone and a harbour are installed; 2 sampling sites were positioned at the Bay of Cádiz (CA-1 and CA-2), an area with low level of sediments contamination (Riba et al., 2004a,b); 3 sampling sites were located at the mouth of Guadarranque River, in Bay of Algeciras (GR-4, GR-3 and GR-3′). Such river receives industrial effluents from

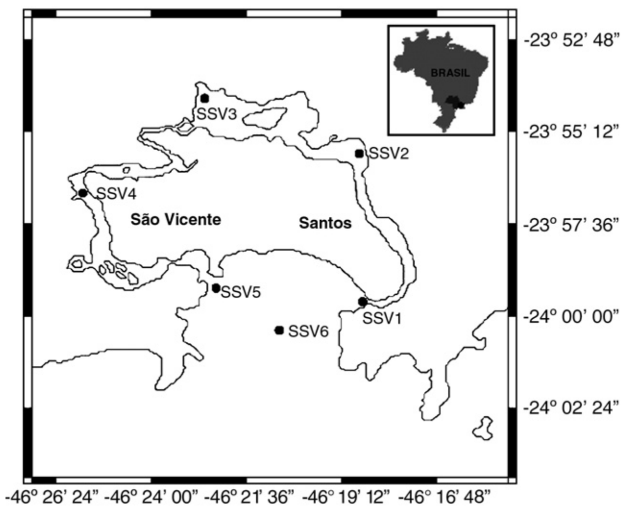

Fig. 2. Localization of the sampling stations in Santos and São Vicente estuarine system — Brazil.

the Algeciras industrial zone. In the Santos and São Vicente estuarine system, 3 sampling sites were located in the Santos Channel, in the influence zone of the harbour and industrial complex (SSV-1, SSV-2 and SSV-3); 2 sampling sites were situated in the São Vicente Channel (SSV-4 and SSV-5), which is mainly influenced by nontreated sewage; and 1 site was located on the central portion of the Santos Bay (SSV-6), near the Submarine Sewage Outfall of Santos (SSOS). Distributed in this way, the sampling sites in Spain and Brazil represent different sources and origins of contamination.

Sediment samples were collected synoptically in each study area, in depths ranging from 1 to 5 m at each site, and stored at 4 °C in the dark. The following examinations were made with the collected sediments: (a) whole sediment toxicity test using the amphipods *Tiburonella viscana* for the Brazilian estuary (Melo and Abessa, 2002) and *Corophium voluntator* for the Spanish areas (Riba et al., 2003), and (b) chemical analysis of homogenized surface sediment for determination of heavy metals (Zn, Cd, Pb, Cu, Ni, Co and V) by DPASV with HMDE (Riba et al., 2002); polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) by using gas chromatography equipped with an electron capture detector (ECD) (U.S. Environmental Protection Agency method 8080); grain size distribution, by standard mechanical dry sieve-shaker techniques, to determine the sand, silt and clay fractions (Buchanam, 1984); and organic carbon (OC) content, using the titration method modified by El Rayis (1985), which is based on the acidification of the sediment sample.

The physical–chemical results of the sediment analyses from both Brazilian and Spanish sites were compared to the Canadian Sediment Quality Guidelines — SQGs (TEL — "Threshold Effect Level" and PEL — "Probable Effect Level") (Environment Canada, 1999), with exception of the nickel concentrations, which were compared to the guidelines proposed by the Florida Department of Environmental Protection (FDEP, 1994). According to the concept of SQG, adverse biological effects are not expected when the concentrations of contaminants are below the TEL values, whereas concentrations of contaminants higher than the PEL values will probably result in adverse biological effects.

2.2. Multivariate analysis

Toxicity and contamination data were integrated by factor analysis, using a principal components analysis as the extraction procedure. This is a multivariate technique to explore variable distributions. The original data set used in this analysis, as for the Spanish sites as for the Santos and São Vicente estuary, included 1 toxicity parameter (amphipods mortality) and 11 physical–chemical parameters (Zn, Cd, Pb, Cu, Ni, Co, V, PCBs, PAHs, %OC, and % of fines). Factor analysis was performed on the correlation matrix, that is, the variables were autoscaled (standardized) so as to be treated with equal importance. All analyses were performed using the PCA option of the *multivariate exploratory technics* procedure, followed by the basic set-up for *factor analysis* procedure from the STATISTICA software tool (Stat Soft, Inc., 2001; version 6).

3. Results

3.1. Sediment toxicity and physical–chemical data

Results of amphipods mortality responses, geochemical matrix characteristics and sediment chemistry are summarized in Table 1 for the Spanish sites and in Table 2 for the Brazilian sites.

The results of the toxicity test with sediments from Southern Spain showed higher toxicity at the *Ría* of Huelva sites and GR-3′ in Bay of Algeciras. Low amphipods mortality was found in Bay of Cádiz sediments, especially in CA-1. In Santos and São Vicente Estuarine System, sediments from the inner estuary (SSV-4, SSV-3, SSV-2) were toxic, as well as the sample collected close to the SSOS (SSV-6). The sediments from SSV-1 and SSV-5 exhibited the lower amphipods mortalities.

The comparison of the chemical data of sediments from Spanish areas to the SQG values (Environment Canada, 1999; FDEP, 1994) showed TEL exceedences for Cd in sediments from Bay of Cádiz (CA-2) and *Ría* of Huelva (HV-1, HV-2, and HV-3); Cu and Zn in Bay of Algeciras (GR-3 and GR-3′, respectively); Ni, at Bay of Cádiz (CA-2) and *Ría* of Huelva (HV-1 and HV-2); PAHs, at Bay of Algeciras (GR-3); and PCBs in Bay of Cádiz (CA-2). Concentrations of contaminants higher than the PEL values were found for Cu and Zn, in Bay of Cádiz (CA-2) and *Ría* of Huelva (HV-1, HV-2, and HV-3); Ni, at *Ría*of Huelva (HV-3) and Bay of Algeciras (GR-3′); Pb, at *Ría* of Huelva (HV-2 and HV-3); and PAHs at the mouth of Guadarranque river, in Bay of Algeciras (GR-3′).

In the Santos and São Vicente Estuarine System, two samples presented contaminants in concentrations higher than TEL: the one collected at the São Vicente Channel (SSV-4) (Cu and PAHs) and that from the Santos Channel (SSV-2), close to the Port of Santos (Pb and Zn). Levels of Cu, at SSV-2 and SSV-3 sediments, exceeded the PEL.

Table 1
Summary of amphipod toxicity test results and physical–chemical characteristics of the sediments for the Spanish stations

Sampling sites	Amphipod* mortality (%)	Trace Metals (ppm)							Organics		Sediment properties	
	Mean (±SD)	Cd	Co	Cu	Ni	Pb	V	Zn	PAH's (ppm)	PCB's (ppb)	O.C. (%)	Fines (%)
CA1	3.3±5.8	0.65	6.80	15.60	8.9	12.20	11,50	18,3	0.074	<0,01	1,10	6,8
CA2	26.7±5.8	1.20	18.30	169.00	29.3	99.20	132,10	360,0	0.096	161,00	2,60	66,4
HV1	100.0±0.0	3.90	26.00	1989.00	42.3	406.00	90,00	1945,0	0.298	3,50	2,10	88,3
HV2	96.7±5.8	2.50	10.00	1543.00	21.2	335.00	111,00	2010,0	0.191	4,60	2,90	89,5
HV3	76.7±5.8	1.60	14.00	789.00	97.2	198.00	76,00	987,0	0.100	1,10	3,90	74,5
GR3	66.7±5.8	0.29	<0.01	20.80	15.5	19.10	24,60	66,0	2.103	<0,01	3,44	75,4
GR4	43.3±5.8	0.10	5.59	3.67	13.1	6.21	0,01	35,3	0.712	<0,01	3,19	54,2
GR3′	100.0±0.0	0.17	12.80	5.01	74.7	21.60	26,10	138,0	12.003	<0,01	2,15	90,5

Table 2
Summary of amphipod toxicity test results and physical–chemical characteristics of the sediments for Santos and São Vicente estuarine system stations

Sampling sites	Amphipod* mortality (%)	Trace metals (ppm)							Organics		Sediment properties		
	Mean (±se)	Cd	Co	Cu	Ni	Pb	V	Zn	PAH's (ppm)	PCB's (ppb)	O.M. (%)	O.C. (%)	Fines (%)
SSV-1	25.0±2.9	<0.1	<0.1	<0.1	4.85	17.4	36.0	73.3	0.106	0.66	6.45	3.75	3.96
SSV-2	72.5±2.5	<0.1	<0.1	167.2	2.96	66.2	24.0	154.2	0.518	4.00	2.13	1.24	4.46
SSV-3	77.5±6.3	<0.1	<0.1	157.7	4.49	22.1	87.8	110.4	0.425	2.61	4.79	2.78	9.68
SSV-4	80.0±5.8	<0.1	<0.1	69.0	3.83	14.9	104.8	66.8	0.950	0.94	4.84	2.82	2.67
SSV-5	40.0±4.1	<0.1	<0.1	<0.1	3.89	8.69	18.6	32.6	0.163	0.58	1.47	0.85	1.42
SSV-6	67.5±4.8	<0.1	<0.2	<0.1	6.02	14.6	<0.1	53.2	0.600	<0.1	1.72	1.00	11.56

3.2. Multivariate analysis

3.2.1. Southern Spain sites

By means of the application of a PCA, the physical–chemical and toxicity data were represented by four new variables, or principal factors (Table 3), which explains 93.12% of the variance in the original data set. Table 3 gives the loadings following varimax rotation for the four factors. Each factor is described according to the dominant group of variables. The first principal factor (F1) is predominant and accounts for 48.88% of the variance; this factor relates almost all heavy metals (excepting Ni) to toxicity responses in the whole sediment toxicity test with amphipods. The second factor (F2) accounts for 20.46% of the variance and shows correlation among PAHs, Ni, percentage of fines, and amphipods mortality. The correlation between chemicals and amphipods mortality is higher in F2 than in F1. The third factor (F3), accounting for 13.02% of the variance, aggregates only PCBs (with a high value), Co and V, without relation to amphipods mortality. Finally, the fourth factor (F4), which accounts for 10.76% of the variance, shows the expected relation between organic carbon and concentration of fines.

3.2.2. Santos and São Vicente estuary sites

Chemical concentrations in sediments were associated by PCA with amphipods mortality, resulting in three principal factors (Table 4). The loadings following varimax rotation for the three factors are found in Table 4. Such factors explained 84.7% of the variance in the original data set. The first principal factor, F1, was predominant and accounted for 41.9% of the variance. This factor combines concentrations of PCBs, Cu, Pb, Zn and lethality to amphipods. The second factor (F2) accounts for 24.28% of the variance and combines organic matter, organic carbon and vanadium in sediments (with higher loadings), but the concentration of this metal and organic characteristics were not associated with biological response. Finally, the third factor (F3) accounts for the lowest variance (18.41%) and associates amphipods mortality (higher than in F1), PAHs and concentration of fines with higher loadings. This factor suggests that the biological effect could be related to the concentrations of PAHs associated to the fine particles of the sediment.

4. Discussion and conclusions

4.1. Sediment toxicity and physical–chemical data

The results of amphipods mortality are coherent to the concentrations of contaminants in the sediments from both Spanish and Brazilian areas. Low amphipods mortality was found in sediments from Bay of Cádiz, especially in CA-1. In CA-2, despite the low sediment toxicity, high concentrations of some metals (particularly Cu and Zn) and PCBs were detected. In Southern Spain, high toxicity was found at *Ría* of Huelva sediments, where the concentrations of all metals exceed either TEL or PEL values. Sediments were also very toxic in GR-3′ (Bay of Algeciras), where the concentration of PAHs exceeded the PEL.

Table 3
Sorted rotated factor loadings (pattern) of the original 14 variables on the four principal factors of Spanish stations

Variable	Factor 1	Factor 2	Factor 3	Factor 4
% Variance	48.88	20.46	13.02	10.76
Cd	0.98	–	–	–
Co	0.64	–	0.51	–
Cu	0.99	–	–	–
Ni	–	0.78	–	–
Pb	0.99	–	–	–
V	0.63	–	0.71	–
Zn	0.98	–	–	–
PAHs	–	0.81	–	–
PCBs	–	–	0.97	–
O.C.	–	–	–	0.97
Fines	0.43	0.72	–	0.43
Toxicity	0.53	0.73	–	–

The loading matrix has been rearranged so that the columns appear in decreasing order of variance explained by factors. Only loadings greater than 0.35 are shown. Factors are numbered consecutively from left to right in order of decreasing variance explained.

Table 4
Sorted rotated factor loadings (pattern) of the original 14 variables on the four principal factors of Santos and São Vicente estuary stations

Variable	Factor 1	Factor 2	Factor 3
% Variance	41.99	24.28	18.41
Cd	–	–	–
Co	–	–	–
Cu	0.88	–	0.37
Ni	–	–	0.35
Pb	0.91	–	–
V	–	0.84	–
Zn	0.91	–	–
PAH's	–	–	0.76
PCB's	0.97	–	–
O.M.	–	0.94	–
O.C.	–	0.94	–
Fines	–	–	0.76
Toxicity	0.42	–	0.87

The loading matrix has been rearranged so that the columns appear in decreasing order of variance explained by factors. Only loadings greater than 0.35 are shown. Factors are numbered consecutively from left to right in order of decreasing variance explained.

In the Santos and São Vicente Estuarine System, some contaminants showed concentrations above TEL and/or PEL values. Copper was the only contaminant found at concentrations higher than PEL (SSV-2 and SSV-3); sediments from these sites were highly toxic to amphipods. The sediment sample collected at the São Vicente Channel (SSV-4) was also toxic to *T. viscana*; in this area, Cu and PAHs concentrations exceeded the TEL values. According to Abessa (2002), 80% sediments exceeding TEL for at least one contaminant produced toxicity. The sediment from SSV-6 showed toxicity, which may be due to the discharges from the Submarine Sewage Outfall of Santos. Abessa (2002) demonstrated that the toxicity of sediments from this area is caused by detergents, sulphur, ammonia, and eventually, metals. Samples collected at areas more influenced by marine waters (SSV-1 and SSV-5) showed lower toxicity.

4.2. Multivariate analysis

Besides the analysis of the variables aggregated by PCA, a representation of estimated factor scores from each station to the centroid of all cases for the original data was done in the present work, in order to confirm the factor descriptions and to characterize the quality of the sediment at each Spanish and

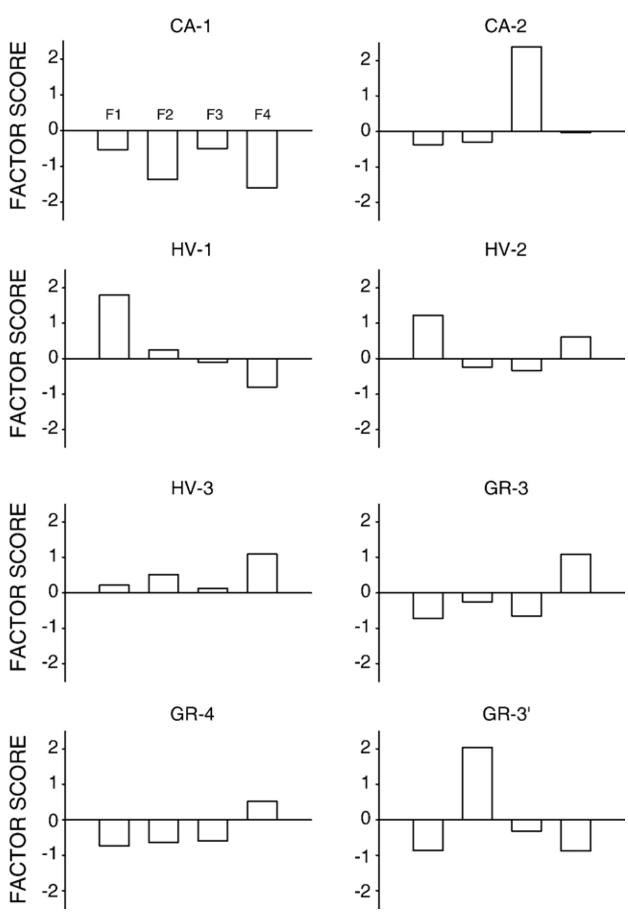

Fig. 3. Estimated factor scores from each of eight cases to the centroid of cases for the original data from Spanish stations. The factor scores quantify to the prevalence of every component for each station and are used to confirm the factor description.

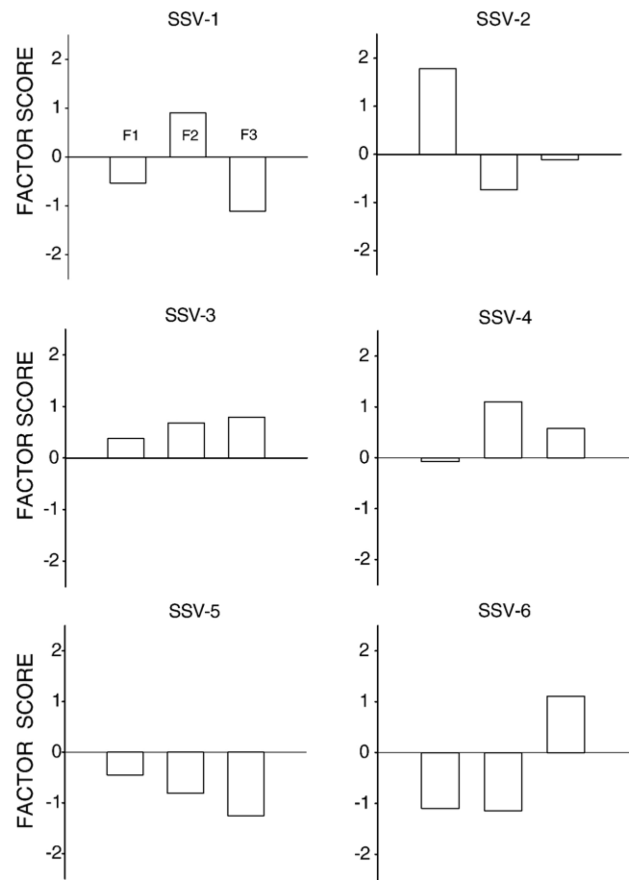

Fig. 4. Estimated factor scores from each of six cases to the centroid of cases for the original data from Santos and São Vicente estuary stations. The factor scores quantify to the prevalence of every component for each station and are used to confirm the factor description.

Brazilian studied site, as can be seen in Fig. 3 for the Spanish sites and Fig. 4 for Santos and São Vicente estuary sites.

4.2.1. Southern Spain areas

The first Factor (F1) shows environmental degradation caused by metals, since the loadings of all heavy metals, in exception of Ni, are related to high mortality in amphipods toxicity test. Indeed, F1 is representative (it has positive factor score) only at *Ría* of Huelva sites (HV-1, HV-2 and HV-3), where the contamination of the sediments by heavy metals is well known (Riba et al., 2004a,b). HV-1 and HV-3 also show a strong environmental alteration due to PAHs and Ni, which were aggregated in the second Factor (F2). Riba et al. (2004b) also found hydrocarbons contamination in sediments from *Ría* de Huelva. One site at Bay of Algeciras (GR-3′) shows positive factor score to F2; it is located at the inner part of Guadarranque River's mouth, receiving directly effluents from the Algeciras industrial complex. The degradation caused by PAHs and Ni can be considered higher than that caused by heavy metals and showed by F1, since the toxicity response loadings are higher in F2. The third factor (F3) demonstrates contamination caused by PCBs, Co, and V; however, this is not related amphipods mortality. Such Factor is positive in CA-2 and HV-3, corroborating with the work of Riba et al. (2004b), which reported PCBs contamination in sediments from Bay of Cádiz and *Ría* of Huelva. The fourth Factor (F4) represents the natural sedimentary matrix with high percentage of fines and organic carbon in *Ría* of Huelva and Bay of Algeciras.

4.2.2. Santos and São Vicente estuary

The first factor (F1) shows environmental degradation related to some heavy metals (Cu, Pb and Zn) and PCBs, since such contaminants were aggregated to the toxicity response, by the PCA analysis. Only for SSV-2 and SSV-3 this factor presents positive scores; indeed, such sites are under influence of Santos harbour and Cubatão industrial complex. PAHs, fines contents and amphipods toxicity are strongly associated to third factor (F3); Cu and Ni also appear to be aggregated, but with lower loadings. It means that the high level of PAHs is leading to environmental degradation at the sites where this factor has positive scores. Such situation is found at SSV-3, SSV-4 and SSV-6. As discussed earlier, SSV-3 in under the influence of the Santos harbour and the Cubatão industrial complex, the probable sources of hydrocarbons for this site; SSV-4 is located on *Mariana* River's mouth, which receives untreated domestic sewage and its river basin drains areas influenced by old irregular industrial landfills, which are contaminated by several chemicals. According to Lamparelli et al. (2001) both sources can contribute to the introduction of PAHs in this area. SSV-6 is located close to the diffusers of the submarine sewage outfall of Santos (SSOS), probably the source of the PAHs found at this site.

In summary, the high degraded environments were found at the inner parts of the Santos and São Vicente Estuary (SSV-2, SSV-3, and SSV-4), confirming existing literature (Medeiros and Bícego, 2004; Abessa, 2002; Prósperi, 2002; Lamparelli et al., 2001), followed by SSV-6, which is near to the SSOS. SSV-1 and SSV-5 did not present chemical contamination, organic contamination or significant toxicity.

The results of this investigation shows that the contamination by PAHs is closely related to amphipods mortality, as in Spanish (*Ría* of Huelva and Gudarranque river's estuary) as in Brazilian areas (inner parts of the estuary); it means that both countries present environmental degradation related to hydrocarbons in those areas. Similar situation was observed for the heavy metals; almost all of the identified metals were related to lethal toxicity in *Ría* of Huelva and Gudarranque river's estuary areas, and the internal zone of Santos and São Vicente estuary. Few exceptions for this pattern were V for both Brazil and Spain, and Cd and Co, only for the Brazilian areas. The presence of PCBs is more serious for Santos and São Vicente estuary than for the studied areas in Gulf of Cádiz, because in Spain such results were not related to amphipods mortality.

Because of the environmental degradation in the studied ecosystems, in special for the Santos and São Vicente Estuarine System, it was difficult to find a satisfactory reference area. By using the Principal Component Analysis technique, such problem was minimized, and thus inferences could be made about the sediment quality of both ecosystems, with or without a reference area.

Finally, the multivariate analysis approach was considered a very useful and reliable tool to identify the contamination and its associated effects in two different ecosystems in the North and in the South Atlantic Ocean. Also, the results demonstrate their feasibility to compare the sediment quality in both ecosystems, with or without the selection of a reference area.

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