Journal of Hazardous Materials 163 (2009) 1353–1359

Contents lists available at ScienceDirect

Journal of Hazardous Materials

journal homepage: www.elsevier.com/locate/jhazmat

A multivariate assessment of sediment contamination in dredged materials from Spanish ports

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ARTICLE INFO

Article history:
Received 19 January 2007
Received in revised form
20 November 2007
Accepted 25 July 2008
Available online 5 August 2008

Keywords:
Discrimination
Cluster analysis
PCA
Dredged material

ABSTRACT

This paper summarises the performance of simple multivariate exploratory analyses to investigate on their potential application for dredged material characterization and management. The data from 25 sampling stations located at 7 different Spanish ports, produced a matrix consisting of 300 observations on 10 variables: the metals Cd, Cu, Cr, Hg, Ni, Pb and Zn, the metalloid As, the sum of PAHs and PCB congeners, and the proportion of fines and organic matter content. The cluster analysis was suitable to separate the sediments according to the extent of contamination while the PCA indicated that the data was defined by two principal factors: a first one that accounts for 56.4% of the total variance, describing the metallic contaminants except Ni and Cr, and a second one that accounts for 14.4% of the total variance describing the organic contaminants PAHs and PCBs. The use of these two simple and untutored multivariate techniques seems a cost-effective approach for future pre-dredging investigations in the studied areas providing useful information for dredged material characterization and management with little additional effort.

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1. Introduction

In the last 40 years national governments have become active in their attempts to monitor and control environmental pollution at the time different international conventions on marine environmental protection were implemented [1]. In this context dredged material management has become a key issue for the protection of aquatic environments due to the potential harmfulness during dredging and subsequent relocation of sediments in estuaries and marine ecosystems. Two main factors contribute to pollutant accumulation in harbour sediments: (1) ports and waterways are usually an important focus of industrial (shipping, loading and unloading, accidental spills) and urban (waste-water emissions) activities and (2) they have been designed to minimise hydrodynamic energy on the inside [2]. The disposal of dredged material is usually controlled by a license system, which actually requires the issue of a permit previous analysis and scientific evaluation of the dredged material and the intended disposal site. Economic, engineering, environmental, regulatory and social aspects all need close consideration for dredged material management though information on the physical, chemical and biological characteristics of the sediment are usually necessary to determine potential dredging methods through a tiered approach, focusing step by step more on details when necessary [3].

As per recommendation of the 1996 Protocol to the London Convention, sufficient information for chemical characterization may be available from existing sources thus a first important step when following this type of assessment framework is to examine all ready available data which can be of some help for dredging operations. In some cases additional measurements of the potential impact of similar material at similar sites may not be required if gathered information is sufficient to make sound conclusions. Nonetheless large-scale studies, designed to monitor sediment contamination in coastal areas that make it possible to compare contaminant levels over different scales of distance and time periods, are available only for few countries. This type of information is scarce only in other countries such as Spain, where environmental quality studies have been performed in some coastal areas and efficient networks to coordinate at national level in sediment contamination are recent [4].

This paper focuses on the use of untutored multivariate statistical techniques to explore the physico-chemical properties of dredged materials from several Spanish ports. These type of techniques have been used for exploratory data analysis, it is: (1) to determine similarities and dissimilarities between sampling stations and sampling areas; (2) to identify data structure features and trends in the behaviour of pollutants [5–8], and (3) for recognising contaminant sources as latent factors which could be interpreted as

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doi:10.1016/j.jhazmat.2008.07.106

Table 1
Range of physico-chemical characteristics in sediments from Spanish ports (all concentrations in mg/kg dry weight except PCBs in µg/kg dry weight)

Port	% Coarse	% Sand	% Fines	TOC (g kg^{-1})	As	Cd	Cr
Cádiz	0.03–0.30 (0.14)	0.38–99.8 (39.6)	0.04–99.6 (60.3)	1.1–24.3 (14.9)	3.4–30.7 (14.7)	0.92–1.3 (1.2)	0.1–14.9 (9.4)
Huelva	0.03–80.4 (20.2)	9.6–56.0 (23.8)	0.01–90.2 (56.1)	1.0–20.3 (9.6)	4.7–840 (412)	n.d.–4.4 (2.8)	8.1–32.9 (18.7)
Barcelona	1.4–5.5 (3.1)	39.9–64.7 (51.2)	33.9–58.7 (45.8)	3.1–17.6 (7.5)	17.4–29.0 (21.5)	0.62–2.9 (1.5)	59.5–105.2 (90.6)
Cartagena	0.9–5.2 (2.8)	38.2–67.2 (52.3)	31.9–57.8 (45.0)	7.2–10.5 (9.2)	62.6–101.5 (79.2)	6.8–98.5 (38.7)	29.5–66.64 (49.8)
Bilbao	0.19–38.1 (13.6)	6.2–20.3 (13.7)	47.4–93.6 (72.8)	14.8–16.7 (15.6)	21.7–104 (64.3)	0.04–2.0 (1.4)	3.5–23.1 (15.0)
Pasajes	0.84–3.7 (2.1)	5.1–38.5 (24.2)	59.6–91.2 (73.8)	14.4–19.8 (17.6)	23.8–39.7 (30.6)	0.04–0.70 (0.47)	18.6–26.7 (22.9)
A Coruña	n.a.	n.a.	49.7–84.3 (69.6)	5.1–7.5 (6.2)	13.6–27.4 (21.2)	0.25–0.96 (0.58)	28.7–33.4 (31.2)

Port	Cu	Hg	Ni	Pb	Zn	ΣPCBs[a]	ΣPAHs[b]
Cádiz	7.0–202.8 (72.2)	0.05–2.0 (0.59)	0.06–21.3 (14.6)	2.3–86.9 (28.0)	21.27–378.3 (150.2)	n.d.–144.9	n.d.
Huelva	1.9–1938 (1052)	0.04–2.4 (1.4)	0.8–129 (42.9)	5.3–384.7 (247.7)	20.9–2458 (1378)	n.d.–2 (2.2)	n.d.
Barcelona	74.9–601.1 (234.5)	0.94–4.1 (1.8)	18.9–32.3 (25.63)	86.7–455.3 (184.3)	219.7–1165 (515.6)	49.2–272.9 (136.4)	0.28–1.8
Cartagena	171.1–665.9 (400.9)	21.6–136.4 (76.5)	15.3–29.0 (20.8)	486.7–1397 (1007)	900.8–8661 (3689)	107.6–468.2 (204.4)	0.66–1.2
Bilbao	23.0–204.1 (109.9)	0.18–1.4 (0.78)	15.7–32.0 (24.7)	40.7–285.9 (158.0)	122.4–777.5 (458.7)	22.1–256.2 (130.0)	0.6–66.7 (27.1)
Pasajes	162.5–167.1 (162.6)	1.1–1.4 (1.2)	19.6–167.1 (27.2)	154.9–293.7 (231.5)	576–1085 (808.0)	240–740 (530.0)	n.d.–1.1 (0.66)
A Coruña	35.3–209.1 (99.2)	0.47–6.4 (2.5)	19.2–20.0 (19.7)	54.1–259.6 (132.0)	134.9–513.2 (279.8)	40.4–254.4 (117.9)	1.9–7.4 (5.5)

N.a. means not available data; n.d. means not detected (see Section 2.1).

[a] Σ_7-PCBs.

[b] Σ_{12}-PAHs.

responsible for the chemical content of the environmental samples [9–13]. More recently it has been used specifically to implement dredged sediment characterization and management [14,15] as these types of techniques have been recognised by the related regulatory and scientific communities. We used the cluster analysis plot to describe the association of samples and variables, and the principal component analysis to evaluate the variability associated with each variable and/or groups of variables. The results are further discussed for a better understanding of sediment contamination in a database comprising several Spanish ports and specifically in the context of navigational dredging and dredged material management in Spain.

2. Materials and methods

2.1. Sampling and sample analysis

The data used in this study included sediments from 7 commercial ports located all around the Spanish coast: three ports are located in the Cantabric coast (Pasajes, Bilbao and La Coruña), two are located in the Atlantic (Huelva and Cádiz) and two in the Mediterranean (Cartagena and Barcelona). These ports stand different maritime traffic and other industrial processes allocated in the surrounding areas due to logistic reasons. Three to four sampling sites were selected in each port trying to cover a wide range of chemical and non-chemical sediment properties and to provide a broad spatial coverage. In each sampling site sediments were collected with a 0.025 m^2 Van Veen grab from approximately the top 20 cm of the sediment and were brought to the laboratory and stored at 4 °C and darkness prior to analysis. The following parameters were determined: As, Cd, Cr, Cu, Hg, Ni, Pb, Zn, sum of polynuclear aromatic hydrocarbons (PAHs) and polychlorinated byphenils (PCBs congeners #28, 52, 101, 118, 138, 153 and 180). Total organic matter content and sediment grain size were included in the analyses as tracers of the solid sediment phase.

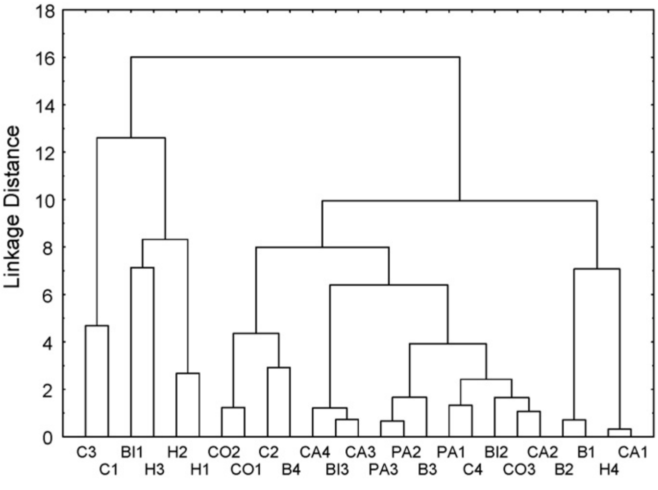

Fig. 1. Hierarchical dendogram for sediment data clustering of sampling sites.

Further details concerning sampling strategies, quality assurance and analytical methods have been reported previously [16]. Briefly, the metals were determined in microwave acid-digested samples in Teflon vessels. For Hg the cold vapour technique was used and for As hydride generation, and both quantified using atomic absorption spectrometry. The concentrations of Cd, Pb, Cu, Zn and Cr were determined using flame or furnace atomic absorption spectrometry, depending on the metal content. PCB congeners #28, 52, 101, 118, 138, 153 and 180 and polycyclic aromatic hydrocarbons (PAHs) were quantified after extraction with cyclohexane and dichloromethane by means of ultrasound treatment and concentration and clean-up with column chromatography. Determination of PCBs was made with gas chromatography with electron capture detection (GC-ECD) (EPA 8080) and PAHs (acenaphtylene, acenaphtene, anthracene, benz(a)anthracene, benz(a)pyrene, chrysene, dibenz(a,h)anthracene, phenanthrene, fluoranthene, fluorene, naphthalene and pyrene) were determined with HPLC with fluorescence detection (EPA 8310). Detection limits were 0.8 and 10–30 µg kg-1 dry weight of sediment of PCBs and PAHs, respectively. Recoveries of analyses were in the range of acceptability.

2.2. Environmetric approaches for exploratory data analysis

The data from the 25 sampling stations produced a matrix consisting of 300 observations on 10 variables. This is summarized by presentation of the mean and range of concentrations at each port (Table 1). Values below the detection limit and non-detected values were set to one half of the detection limit value. Different approaches were used to pre-treat the data. Specifically the log transformation of experimental data followed by the addition to all variable entries of a constant to remove large negative values

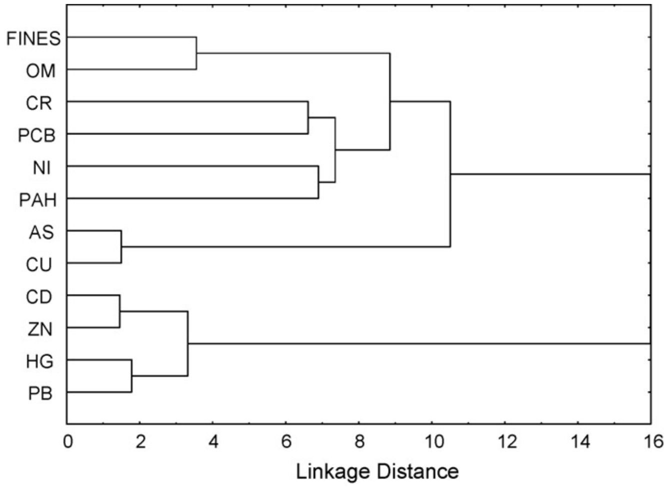

Fig. 2. Hierarchical dendogram for sediment data clustering of variables.

was tested. This approach, which is recommended for skewed data sets such as those in environmental studies where the majority of the values are low values with a minor contribution of high values [6], did not offer any improvement on the results (data not shown). Thus, data were just log-transformed before performing the multivariate statistical analyses.

The statistical analyses included firstly a cluster analysis to investigate for similarities between the objects in the variable space, which were represented visually as dendograms (tree diagram) of sampling stations or variables using the Euclidean distance as distance measure and the Ward's method as linkage method. This methodology was selected since it seems a reasonable compromise for quantitative data [13,17]. In addition, the principal component analysis (PCA) was used as a chemometrical approach to reduce the size of the variable space and substitute a large number of parameters by a small number of independent factors (principal components), which allows data interpretation and data structure explanation [12,18]. This was done by means of the principal variable loading and the bi-plot of factor scores for the sampling sites in an attempt to correlate both types of information. All statistical analyses were performed with the STATISTICA® 6.0.

3. Results and discussion

3.1. Sediment characterization

The general characteristics of the sediments varied significantly among and between ports (Table 1). Sediments were principally mud except one station from Cádiz and one station at Huelva. The muddy sediments had percentages of fines between 31% and 99.59% while total organic matter followed a similar trend, varying between 1% for coarse and sandy sediments to 24%, value reported in Cádiz. The chemical characterization showed that most of the sediments contained mixtures of contaminants, including trace elements, PAHs, and PCBs. The highest concentrations for most of the compounds were found in Cartagena and Huelva. The highest concentration of PCBs was found in the port of Cartagena although this type of compounds was also present in Barcelona, Bilbao and Pasajes. In Cádiz and Huelva PCBs were always below detection limits. The PAHs concentrations were most often less than the detection limit with the highest concentrations present in the port of Bilbao.

3.2. Multivariate statistical methods

3.2.1. Cluster analysis

The result of the cluster analysis performed on the sampling stations (Fig. 1) presents the formation of two major clusters, each one with different subgroups. The first cluster includes the most severe polluted sediments from Cartagena (sites C1 and C3, located in the east bay), Huelva (sites H1, H2 and H3) and Bilbao (site BI1), all characterised by high metallic concentrations. A subgroup was formed for sites at Cartagena due to its determining concentrations of Cd, Hg, Pb and Zn, which in turn was separated from the three inner stations at Huelva and the inner sediments from Bilbao. The ports of Huelva and Bilbao are located in the Tinto-Odiel and the Nervión estuaries, respectively, and both are affected by surface metal enrichment. In Bilbao it is caused by effluent discharge from metal-working industries and subsequent deposition due to reworked upstream mine-waste [19] while in Huelva it is due to acid mine drainage [20]. This hypothesis would also explain that station 1 from Huelva (H1), located in the fishing harbour, was not clustered with the other inner harbour sediments due to its metallic contamination.

The second cluster comprised the moderately and low contaminated sites, which are clustered in different subgroups according to different sediment properties and characteristics. One cluster was formed by sediments with the lowest proportion of fines and organic matter content. Sites 1 from Cádiz (CA1) and 4 from Huelva (H4) were linked at the lowest linkage distance due to their particular sediment properties and low contamination, which in turn were linked to a new cluster with sites 1 and 2 from Barcelona (B1 and B2), that reported the lowest proportion of fines and organic matter content among the harbour mud. The higher chemical concentrations in Barcelona cause the large linkage distance with the not polluted sediments CA1 and H4.

The other sediments were clustered together, with secondary clusters according to different grades of contamination. A first

Table 2
Binary correlations (coefficients) of the variables

Variable	FS	OM	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn	PCBs	PAHs
% Fines (FS)	1.00											
Organic matter (OM)	0.74^{**}	1.00										
As	0.30	0.19	1.00									
Cd	−0.09	−0.06	0.03	1.00								
Cr	−0.20	−0.26	−0.07	0.28	1.00							
Cu	0.26	0.20	0.95^{**}	0.19	0.08	1.00						
Hg	−0.18	−0.12	0.00	0.92^{**}	0.29	0.16	1.00					
Ni	0.06	0.04	0.23	0.01	−0.02	0.26	−0.03	1.00				
Pb	−0.12	−0.03	0.17	0.82^{**}	0.33	0.35	0.93^{**}	0.05	1.00			
Zn	0.00	0.03	0.28	0.96^{**}	0.28	0.45^{*}	0.88^{**}	0.11	0.87^{**}	1.00		
PCBs	0.10	0.24	−0.24	0.00	0.09	−0.15	0.01	0.01	0.18	0.05	1.00	
PAHs	0.14	0.10	−0.07	−0.07	−0.13	−0.13	−0.09	0.01	−0.09	−0.09	−0.01	1.00

* means significant at $p = 0.05$ and ** at $p = 0.01$.

Table 3
Factor loading and percentage of the total variance explained for 4 factors

	Factor 1	Factor 2	Factor 3	Factor 4
% Fines	0.24	0.18	0.86*	0.29
Organic matter	0.16	0.10	0.93*	−0.02
As	0.73*	−0.36	0.38	0.14
Cd	0.87*	0.13	0.00	−0.06
Cr	0.30	0.21	0.23	0.84*
Cu	0.79*	−0.06	0.48	0.25
Hg	0.86*	0.30	0.06	0.30
Ni	0.23	0.11	0.73*	0.55
Pb	0.79*	0.29	0.29	0.41
Zn	0.86*	0.11	0.35	0.29
PCBs	0.21	0.75*	0.14	0.44
PAHs	0.05	0.89*	0.13	0.03
% Explained variance	56.4	14.4	12.4	5.3

Factor loadings > 0.60 are marked with an asterisk.

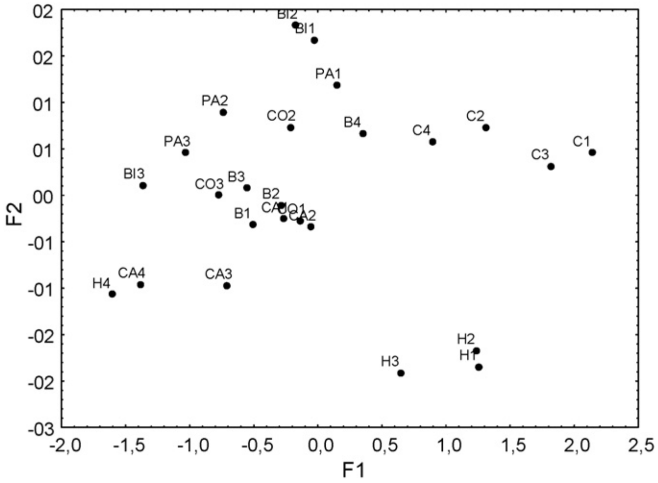

Fig. 3. Principal component score plot of sampling sites from Spanish Ports.

group represented the high contamination of sites 1 and 2 from A Coruña (CO1 and CO2), station 2 from Cartagena (C2) and 4 from Barcelona (B4), which are more affected by harbour activities due to its location in the inner part of these ports. Site 2 from Cádiz (CA2), site 3 from A Coruña (CO3), 2 from Bilbao (BI2) and, at a higher distance, site 1 from Pasajes (PA1) and 4 from Cartagena (C4) were also affected by harbour activities but less than the sediments mentioned above. In this way, sites 2 and 3 from Pasajes (PA2 and PA3) and site 3 from Barcelona (B3) formed a third group of medium-low contaminated sediments, and finally sites 3 and 4 from Cádiz (CA3 and CA4) and site 3 from Bilbao (BI3) formed a group of mud with low contamination.

The cluster analysis of the variables according to Ward (Fig. 2) identified two major clusters that separate chemical compounds

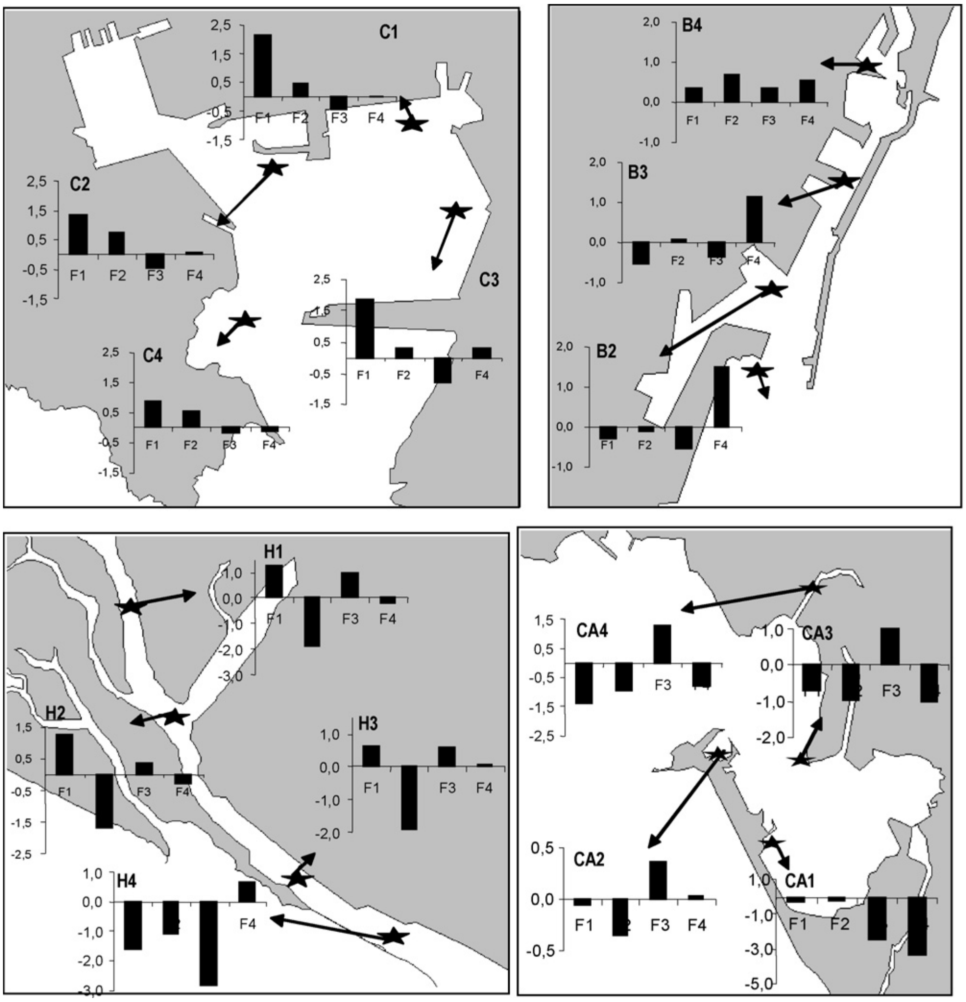

Fig. 4. Sampling stations and loadings for the extracted factors.

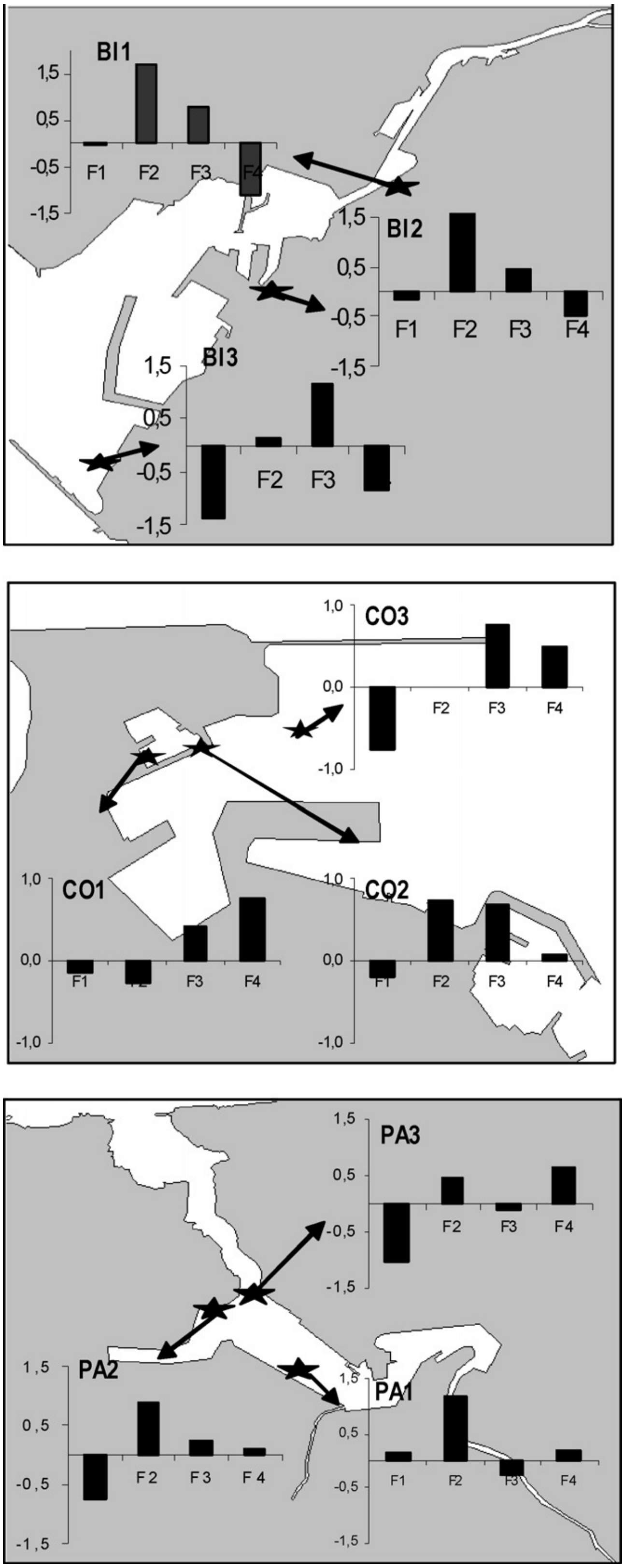

Fig. 4. (*Continued*).

that are linked to the sediment properties (percentage of fines and organic matter content) from those that are not. A first group was formed by the pairs of metals Hg and Pb and Zn and Cd, both at similar linkage distances. The most conspicuous concentrations of these metals were present in Cartagena, that stands important contamination sources: a Zn smelter, that explains the close association between Cd and Zn as these two elements are closely associated in its geochemistry, with Cd being obtained as a by-product from smelting of sulphide ore minerals in which it has substituted for some of the Zn [21], and Hg and Pb that seem to be more related to other industrial sources of metallic contamination such as a Pb smelter or other metal processing industries. In addition previous studies have shown a close relationship between these four metals in areas heavily affected by steel plant contamination [22].

The second cluster comprised other chemical compounds, the proportion of fines and organic matter content. These two sediment properties appeared linked at a low distance indicating that organic matter adsorbs onto fine particulates in harbour sediments, possibly due to the low hydrodynamic energy in the inside. Furthermore, these variables are used as tracers of the solid phase sediment in data interpretation. Organic matter content reveals important complexation processes in the sediment phase with participation of anthropogenic effects and the grain size is related to the processes of metal adsorption [7,13]. Previous results have reported the strong complexation of metals by organic ligands, particularly Cu, Ni, Cd and Zn [14,23], but the cluster analysis evidences a close relationship between the sediment properties and the pairs of compounds formed by Ni and PAHs, Cr and PCBs, and also As and Cu at a higher distance.

The relationship between As and Cu is explained by the high concentrations of these metals in the sediments from Huelva, that show similar patterns in nature due to their common sources and their association with same type of carrier particles with similar sedimentological properties. Similarly, the sediment organic fraction is supposed to play a major role in binding hydrophobic compounds [24], which explains the cluster of PAHs and PCBs with the organic matter content of the sediment. Nonetheless PCBs were linked to Cr while PAHs were grouped with Ni, which indicates a more complex distribution of these compounds in the sediments studied.

The correlation matrix was developed to study the distribution of variables in pairs (Table 2). As expected the highest correlations were found for the pairs As-Cu, Cd-Zn and Hg-Pb (0.95, 0.96 and 0.93, respectively), even though similar coefficients appeared between the pairs Cd–Hg, Cd–Pb, Zn–Hg and Zn–Pb. On the contrary no significant correlation was found between the other four variables considered, namely PAHs, PCBs, Ni and Cr. Even if the correlation analysis did not identify a straightforward relationship between these compounds, it is worthy mentioning that this high variability may be determined by the more homogeneous distribution of these compounds among the ports studied.

3.2.2. PCA

Due to the large variability among ports the possibility of extracting seven principal components, each one explaining the variability associated to each area, was considered and seven factors were firstly explored. Nonetheless the results indicated that two to four factors were enough to explain the variance associated to the data studied. The first extracted factor, that explains more than 50% of the total variance, includes all metallic compounds except Ni and Cr, which were explained in later extracted factors (Table 3). While this first factor seems to explain most of the variability associated to the metallic compounds, the second extracted factor, that explains 14% of the total variance, accounted principally for the organic contamination including both PAHs and PCBs. The third extracted factor, that explained 12% of the total variance, included the sediment properties and Ni while the variability associated with Cr was explained by a fourth extracted that also included Ni, Pb and PCBs.

Since the two first extracted factors accounted for a great proportion of the variability associated to metallic and organic contaminants, we explored the distribution of the sediment samples in the space defined by these two factors (Fig. 3). A first area of low contamination is represented when F1 and F2 had negative factors. The sediments from Cádiz and sample H4 were located in this area according to the low chemical concentrations, nonetheless some influence of the harbour activities is suspected due to the presence of samples CA2, B1 and B2 from Barcelona, with negative scores for F1 and F2 but a clear increasing trend according to the increasing organic and metallic contamination in the inner harbour sediments (Fig. 4). Sediments from A Coruña (CO#) also presented low factor scores for F1 and F2, although increasing scores for F1 and F3 indicate some surface metal enrichment related to the proportion of fines. Samples CO1 and CO2 reported similar PAHs concentrations while CO1 had a PCBs concentration five-fold than that for CO2, which may explain the need of two factors, F2 and F4, instead of a single one (F2) to explain the organic chemical contamination in this port. In addition high scores for CA3 and CA4 in F3 point out some organic matter enrichment in the inner sediments from Cadiz due to recent urban wastes [12].

The variability for the port of Barcelona appeared explained principally by F4. This factor, that reported decreasing values moving inside the harbour (Fig. 4), was the most difficult to interpret. Nonetheless, F1 and F2 indicate a clear trend in the organic chemical load of the sediments to increase along the harbours of Barcelona. This was also observed in the port of Bilbao and the port of Pasajes and both showed a clear contamination gradient seaward along the area, with the highest scores for PA1 and a clear decreasing trend for PA2 and PA3. The type of contamination is principally organic, with high positive scores in factors that account for these compounds and some metal enrichment in the inner sediments (Fig. 3). As for other ports, some variability of Pasajes was also explained by F4, which principally accounts for Cr with some other contributions of other compounds such as PCBs, Ni and Pb (Fig. 4).

A clear group was formed by sediment samples from Huelva H1, H2 and H3, with high loadings for F1 and negative loadings for F2 (Fig. 3). In the case of Huelva some conclusions are reached after considering the chemicals of concern in the area and the factor loadings for the sediments along the estuary (Fig. 4). Considering that As and Cu reported the highest contamination levels it is of significance the clear decreasing trend in F1 and F3 loadings seaward along the estuary, which is in accordance with previous results reporting a clear sediment contamination gradient along the Tinto and Odiel estuary for some metals [20,25] and a clear gradient in the organic content and the proportion of fines. Similarly a group was formed by sediments from Cartagena located up on the right side (Fig. 3), which corresponds to sediments with high metallic and organic contamination. The results gave a very complete distribution of contaminants inside the bay of Cartagena (Fig. 4), with a clear source of metallic compounds on the eastern bay further discussed above that correspond to high scores in F1 for sediments C1 and C3 and lower in the sediments from the western bay. On the contrary organic contamination, accounted for in F2, trends to increase in the west of the bay due to the proximity of the principal harbour facilities though a secondary source of PCBs is possible in the vicinity of station C3 according to the high score for F4.

4. Conclusions

The database available in this study presented a high variability, with very different chemical concentrations and sediment properties among ports and between sampling stations. Nonetheless, these and other examples on the use of multivariate statistical techniques indicate that simple untutored tests may provide a useful tool to study dredged material contamination. Considering that the same statistical software performs several tests with little extra effort, the application of this type of techniques on existing data, or data from pilot or screening studies, can help to estimate distributions in relevant physico-chemical measurements. In this sense, its application to design simple models for a better selection of sampling strategies seems further recommended [26]. Later on, this type of studies is a known valuable source of information that reduces the costs of the investigations without reducing the confidence on the final assessment.

Acknowledgements

Thanks are due to the Port Authorities of Cádiz, Huelva, Barcelona, Cartagena and La Coruña for their help during the sampling. Part of the results was obtained from a joint research between the Centro de Estudios y Experimentación (CEDEX) and the University of Cadiz. This research was supported by the projects CTM2005-07282-C03-C01/TENCO and PHB2005-0100-PC. M.C. Casado-Martínez was funded by the Spanish Ministerio de Ciencia y Tecnología REN 2002_01699/TECNO.

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