Chapter 1
Integrated Assessment of the Quality of Harbor Sediments: Case Study Based on a Comparative Analysis of Sediments Quality of Two Industrial Ports: Bourgas (BG) and Bari (IT)

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Abstract In order to evaluate the pollution status of the sediments of two industrial and commercial harbors, an integrated approach was applied. A closer look of chemical and bio-toxicological data selected in 23 different stations located in the different internal basins of Bari port (IT) and Bourgas port (BG) is provided; integration of data by multivariate analysis was conducted, and a comparison procedure is presented as useful tool to elucidate the potential risk of sediments and helpful step towards a harmonized assessment criteria.

1.1 Introduction

Ports play an important role in human society, being areas where economic activities related to transportation, exchange and production of goods are concentrated, but they can heavily influence the marine environment. The port's effectiveness depends on its maritime accessibility, and for this reason, capital or maintenance dredging is frequently needed to ensure an adequate depth for navigation for vessels that visit the port. Depending on their chemical, physical, and ecotoxicological characteristics, dredged sediments may be managed in several ways. For example, unpolluted sediments with an appropriate grain size may be used for beach nourishment, or dumped at sea, while contaminated sediments must be isolated, treated, reused on land, or disposed in confined disposal facilities (CDF). The management of dredged material is usually controlled by a license system, which requires the preparation of a complex and often economically expensive

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managing dredging plan (MDP) based on the pollution level of the sediments. It is therefore clear that an adequate and correct characterization of harbor sediments impact seriously the whole dredging plan. International conventions such as the London Convention (1996, 2007), Barcelona Convention, OSPAR and Helsinki Convention, PIANC (1997) provide useful guidelines on the management of dredged sediments. Nevertheless, all abovementioned guidelines are usually nonmandatory for European countries. Literature studies confirmed that currently the EU legislation does not deal specifically with dredging sediments (Alvarez-Guerra et al. 2007, 2009; Hamburg Port Authority 2011), i.e., harmonized limit values of the contaminants for dredged material are not yet established; thus, the issue of the contaminated dredged material is regulated in each European country with legislation based on different approaches. This heterogeneity of approaches, on one hand, leads to a different assessment of sediment quality and on the other hand, often creates complex, expensive, and sometimes needless execution procedures, difficult to be understood (Choueri et al. 2009).

In this study, an integrated method for the assessment of the quality of sediments sampled in two industrial and commercial ports of two different countries, Bourgas port in Bulgaria and Bari port in Italy, was proposed. The method is based on the combination of the chemistry and biology/ecotoxicology measurements (Chapman 2006). Chemistry gives information on the presence, quantity and chemical form of substances linked to port activities. The integration of biological investigations allows an assessment of toxicity and bioavailability of contaminants, to understand the mechanisms of their toxic action and identification of the area of potential biological impact within port's basins (Hoellert et al. 2002). This method was aimed to develop a general protocol able to standardize the procedure for the quality assessment and managing of contaminated sediments of different countries, thus, allowing an inter-comparison of the obtained results in laboratories of different nationalities.

1.2 Materials and Methods

1.2.1 Study Areas and Sediment Sampling

The port of Bari (IT) and the port of Bourgas (BG) are two important hubs of the port-network respectively alongside Adriatic Sea and Black Sea. Both ports cover a key role in the freight and passengers traffics within the South East European area. The port of Bari is one of the most important harbors of the southern Adriatic coast. It was traditionally considered the gateway of Europe to the Balkan Peninsula and the Middle East. Nowadays, it is a multipurpose stopover equipped with docks for handling all different goods and freight. Several quays for different types of commercial traffic (solid and liquid bulk, containers, packaged goods, steel products, forest products, etc.), as well as wharfs for Ro-Ro ferries and platforms for cruise ships, are daily used. The water design depth varying from 6.9 to 14.9 m, allowing handling of bigger size ships and vessels. The important activity and the increased ship traffic within the port

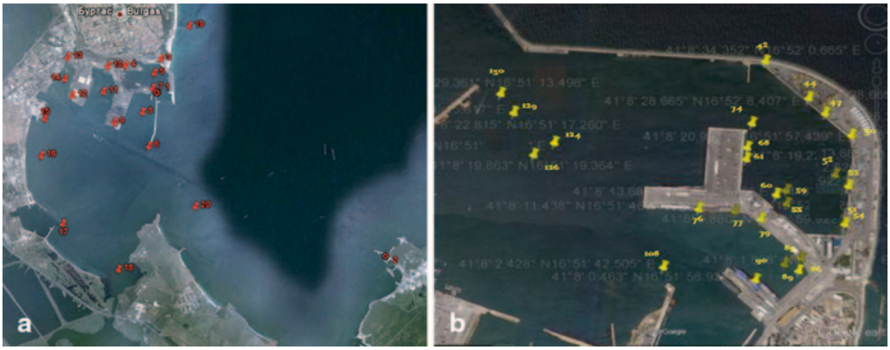

Fig. 1.1 Sampling map. **a** Port of Bourgas. **b** Port of Bari

area impact seriously the quality of port aquatorium and therefore of harbor sediment quality that acts as final sink of all the contaminants ending up in the water column.

The port of Bourgas is one of the biggest and busiest Bulgarian ports. It is situated at the head of the Homonym Bay, on the west coast of the Black Sea. Due to its strategic location, it is actually considered the gateway of the Pan-European Corridor VIII from Italy via Albania and the former Yugoslav Republic of Macedonia to the Middle Asia, Russia and other commonwealth of independent states (CIS) countries. The harbor area has a very easy access from the sea and is well protected from the winds and waves. The port comprises 28 berths with a total length of 4800 m and 24 operative quays with a whole mooring length of 4800 m. The water depth ranges from 6.1 to 15.5 m, allowing the handling of vessels up to 125,000 DWT. There are railway lines in the majority of quays. The port of Bourgas is relatively well equipped with passenger, container, and Ro-Ro terminals, almost linked directly with the cold storage area.

In the present work, the sampling campaign was carried out in 2010–2011, and it was accurately planned, according to Loring and Rantala (1991). Representative samples for each basin's feature were collected, taking into account the problems associated with trace level cross-contaminations. Depending on the sampling location, the data were divided into several groups representing particular features of harbors (Fig. 1.1).

The devise used for sampling is a vibro-corer PF1, equipped with liner for the Bari sediments and grab samplers for Bourgas sediments. In both cases, the support of vessels provided with the differential system global positioning system (GPS) for positioning of sampling cores was utilized. The samples were individually transferred into precleaned polyethylene boxes and stored at 4 °C till they reached the laboratory.

1.2.2 Determination of Total Organic Carbon (TOC)

In Bourgas sediments, TOC was determined by Turin's method based on dipotassium dichromate oxidation (Kaurichev 1980), while loss of ignition and gas chromatography separation techniques through Carbon, Hydrogen and Nitrogen (CHN) analyzer were used for TOC determination in sediments collected in the port of Bari. The analytical procedure was checked with reference-certified materials, 277 Community Bureau of Reference (BCR) and National Research Counsil of Canada (in original language *Conseil national de rechershes Canada*)(NRC CNRC) HS-1 for organic compounds, which allow agreement with certified values higher than 90 %.

1.2.3 Determination of Petroleum Hydrocarbons Content (THC)

Petroleum hydrocarbon content in sediments was determined in two different methods: gravimetrically for Bourgas sediments and by solvent extraction and infrared (IR) spectrometry (USEPA 1994) for Bari sediments.

1.2.4 Determination of the Heavy Metal Concentration

The distribution of 7 heavy metals was analyzed (Cd, Cr, Ni, Pb, Cu, V e Zn). Two analytic methods were used for the determination of the heavy metal concentration. The Bourgas port's sediments were analyzed with the energy dispersive X-ray fluoresce method, while in the Bari port's sediments the heavy metal content was assessed, after sample mineralization, by inductively coupled plasma–mass spectroscopy (ICP-MS, Thermo X Series). Standard reference materials for trace elements offered by advanced research projects agency (ARPA) were used to control the analysis quality. Mineralization of the different matrices prior to metal analyses was carried by microwave irradiation after addition of HCl/HNO3/HF solution to each weight sample. The procedure was followed twice for each sample. As to the Hg concentration, it was determined by cold vapor atomic fluorescence spectroscopy following HNO3–H2SO4 procedure.

1.2.5 Bio-Toxicological Tests

Two different biotests are used: The toxicity of Bourgas port sediments were analyzed using the phosphatase activity of sediment bacterial communities. Phosphorus is the growth limiting factors for phototrophic organism in the sea and its supply depends largely on the regeneration of phosphate from organic matter accumulated on the sea bottom. The regeneration process is carried out by exoenzymes called phosphatases. Sediment bacteria capable of polyphosphate metabolism are directly implicated in sediment phosphorous dynamics and control phosphorus metabolism of marine ecosystems. Therefore, their activity can be used as a sensitive indicator of toxic effects of pollution on marine ecosystem; hence, the test indicates whether

Table 1.1 Toxicity scale according to the criteria reported in ICRAM–APAT (2007)

Bio-species	Class A Absent/negligible toxicity	Class B Moderate toxicity	Class C High toxicity	Class D Severe toxicity
Vibrio fischeri	S.T.I.≤3	3<S.T.I.≤6	6<S.T.I.≤12	S.T.I.>12
Dunaliella tertiolecta (elutriate)	EC20≥90% Δ≤15%	EC20< 90% eEC50 > 100% 15 < Δ%≤30	40%≤EC50 100% 30 < Δ %≤80	EC50 < 40% Δ > 80%
Paracentrotus lividus (elutriate)	EC20≥90% Δ≤15	EC20 < 90% e EC50 > 100% 15 < Δ %≤30	40%≤EC50 <100% 30<Δ %≤80	EC50 < 40% Δ% > 80

sediment bacterial communities in the different points of sampling are intoxicated, and the level of intoxication was determined by the reduction of the bacterial phosphatase activity.

On the other hand, the bioassay battery was used for monitoring of the toxicity of Bari sediments. The bioassay battery includes three tests: the Microtox® Solid Phase Test (SPT), an acute toxicity test for solid matrices based on inhibition of natural bioluminescence of the marine bacterium *Vibrio fischeri*. This test was applied directly to the solid phase (whole sediment or after removal of pore water). In addition, bioassay using the sea urchin *Paracentrotus lividus,* conducted on the extracted liquid phase (elutriate) of the sediments and the algal growth inhibition test (using *Dunaliella tertiolecta)* was also conducted. The analytical methods and toxicity classification criteria were applied according to national or international official procedures (Table 1.1).

1.2.6 Statistical Analysis

Principal component analysis (PCA) was performed with statistical software (STATISTICA (v.7)). The number of factors was detained by the total variance explained.

1.3 Results and Discussion

1.3.1 Chemical Data

The data obtained by chemical analyses showed different levels of contamination within inner basins of both ports due to intensive human activities. Total hydrocarbons (Fig. 1.2) confirmed the literature data for the port of Bourgas. The pollution of Bourgas Bay was well documented by many researchers (Kamburska and Valcheva 2003; Dencheva 2010; Rojdestvenski 1986; Moncheva et al. 2001, 2002) and in

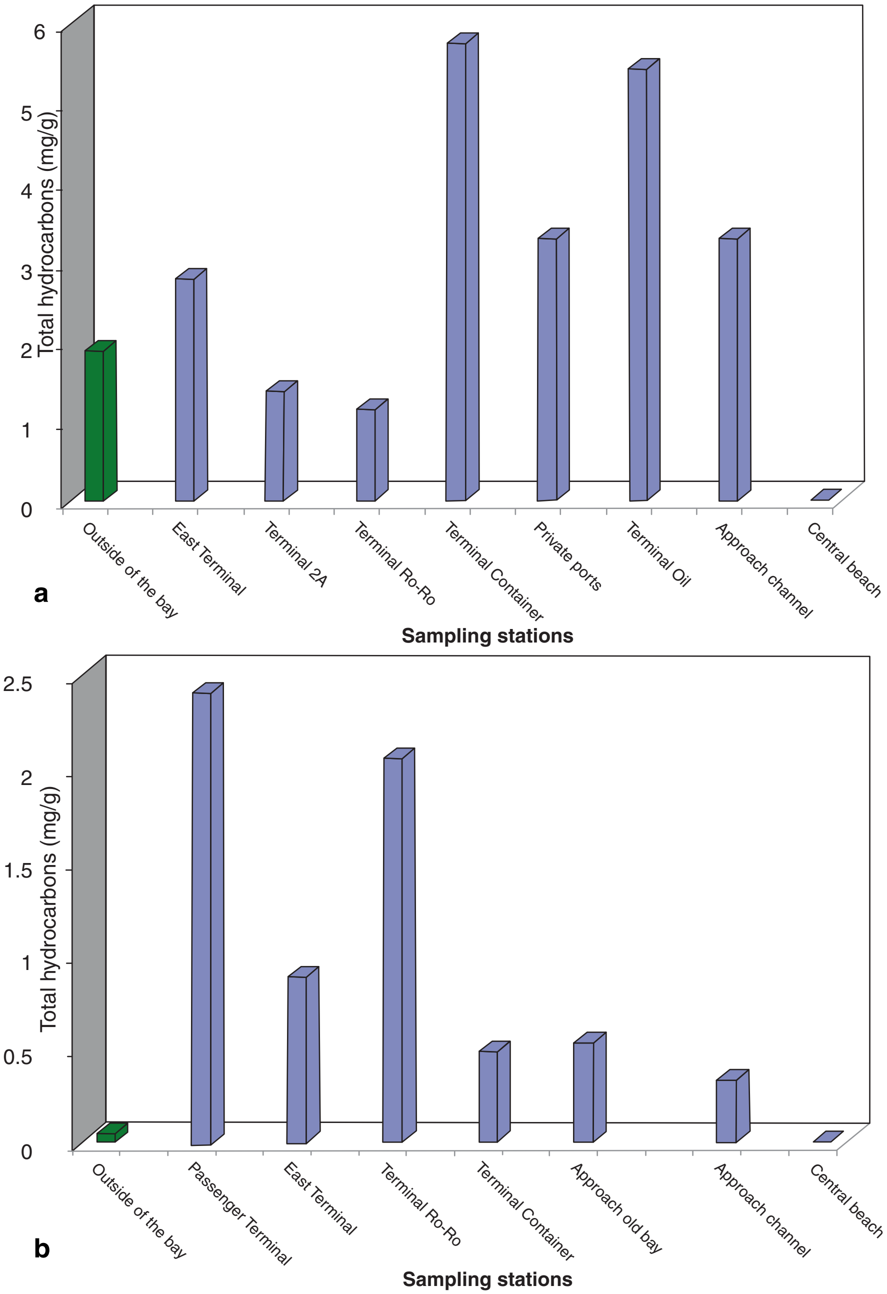

Fig. 1.2 The levels of total hydrocarbons. **a** In the port of Bourgas sediments. **b** In the port of Bari sediments

some monitoring programs (Dencheva 2008) of Black Sea. On the contrary, for the port of Bari, pristine data are not available. In Italy, national guidelines established specific threshold levels of contamination aimed to support the classification of sediments in different risk classes, while in Bulgaria sediment quality guidelines are not yet established at national level and pollution threshold level are not available. For these reasons, the obtained data were herein compared with two guidelines, threshold effect level (TEL) and probable effect level (PEL) according to MacDonalds et al. (2000).

The level of hydrocarbons in Bourgas bay sediments is high—up to 8.97 mg/g dry wet sediment. Based on some published works, the total hydrocarbons content in sediments more than 0.5 mg/g is indicative of pollution, while sediments containing less than 0.01 mg/g of total hydrocarbons may be considered as unpolluted. In accordance with this rule, the following situation was observed: For the Bourgas port, the stations 19, 22, 4, 7, 23, 10 and 12 can be considered as unpolluted since the level of hydrocarbons revealed are under the threshold level allowed; sediments from stations 8 and 9 can be considered as low polluted and the others as heavy polluted. Totally, the basins “Terminal Container” and “Terminal Oil” are the most impacted of oil pollution.

The most polluted stations within the port of Bari are those dedicated to the container and Ro-Ro traffics represented by the stations BA42, BA44, BA51 (an average of 3.0208 mg/g) and near the Passenger Terminal—BA89 and BA90 (accounting 6.25 mg/g). The sampling stations located in the approach channel of the port and those located in the open sea (account 0.02 mg/g) can be considered as unpolluted. The other stations revealed a moderate concentration of pollutant accounting a range of 0.2–1.8 mg/g dry wet sediments.

As to the sediment organic matter, it should be highlighted that in most of the sampling stations in both ports, the bottom bay is quite muddy. Different levels of total organic matter are accordingly revealed. The concentration of organic matter in Bourgas (Fig. 1.3a) varies from 28 mg/g (station 20) to 372 mg/g dry wet sediment (station 13) and the sediments richest in organic matter are those of “Terminal Container,” as well as station 19, where it is recorded decayed biomass of mussels. The accumulation of high concentrations of organic matter on the sea bottom, especially on that of “Terminal Container” is a result of the low self-purification capacity of the Bourgas Bay. Less, but relevant concentrations are revealed within the port of Bari (Fig. 1.3b). High concentration of organic matter is revealed in stations 89, 90, and 76 and 81 (respectively 5.8, 2.5, 4.1, 3.1 mg/g) alongside the Passenger Terminal. Noteworthy, also the high concentration respect the mean of the whole Bari *port aquatorium* in the station located within the Ro-Ro Terminal (BA42, BA44—an average of 4.5 mg/g) and Container Terminal (BA53, BA56—an average of 3.2 and 3.0 mg/g). The lower concentration level of organic matter within the port of Bari is probably related to a different hydrodynamic design of Bari port that permits a major self-purification of sea bottom.

With regard to the heavy metals, the data obtained revealed for both ports higher concentration of heavy metal. The Bourgas Bay sediments revealed higher concentration of copper (Cu), zinc (Zn) and lead (Pb). The concentrations of Cu in the

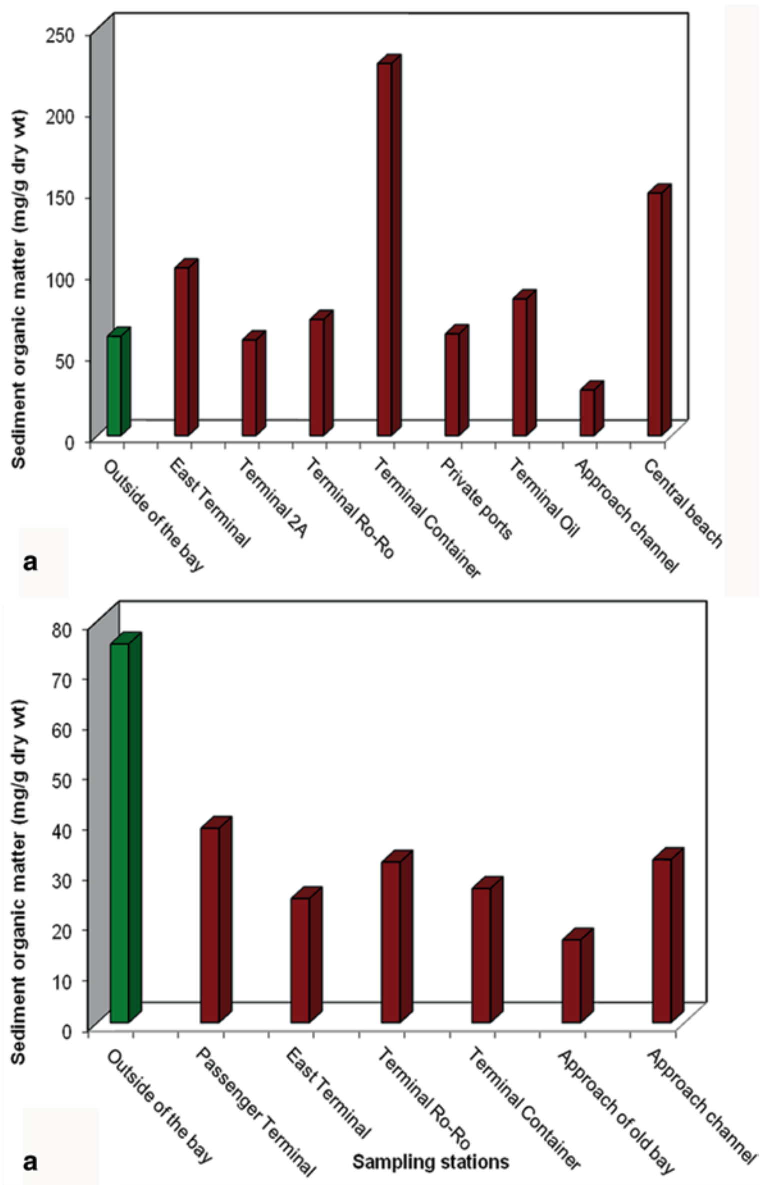

Fig. 1.3 Organic matter concentrations. **a** In the sediments of the Bourgas port. **b** In the sediments of Bari port

sediments of Bourgas Bay's basins exceeds from 7 to 26 times that of control station (32 ppm) as the most polluted is the station 11 located in the Ro-Ro Terminal. The same consideration was provided for Bari port. Higher concentration values are related to Cr, Cu, Pb, and Zn, and the most polluted stations are those related to the Passenger Terminal and Ro-Ro and Container Terminal. In order to establish the toxicity of the each basin, the concentration levels of heavy metals are compared with TEL and PEL sediment quality guidelines according to MacDonald et al. (1996). Overall, more than 95 % of all metal concentrations exceeded the TEL and 45 % exceeded the PEL sediment quality guideline. Highly variable exceedances of the TEL and PEL values, in both ports, were revealed (in Bari: 0 % Cd, 71 % Cr, 100 Cu, 90 % Pb, 100 % Ni, 95 % Zn and V and the PEL (65 % Pb); in Bourgas: 100 % Cd and Cr, 77 % Cu, Ni, Pb, Zn and the PEL (50 % Cu, Ni, Cr, 33 % Pb and Zn); Fig. 1.4a, b).

1.3.2 Bio-Toxicity Data

Bacterial phosphatase activities (µg/mg sediment organic matter) in the basins of Bourgas bay are lower than the average activity of the referent stations (outside of the bay) and the rate of inhibition varies from about 4 % (private ports) to about 57 % (Terminal 2A and Terminal Container) (Fig. 1.5). The sediment bacterial communities from basins “Terminal Container” and “Terminal 2A” could not overcome the anthropogenic stress resulting in inhibited phosphatase activities and therefore reduced share in nutrients’ cycling.

Basin “Terminal Container” is high eutrophic and unfavorable for the growth of sediment aerobic bacteria while the values of environmental factors measured in “Terminal 2A” are much better. The low bacterial phosphatase activity in “Terminal 2A” basin may be a result of the toxicity of Zn whose concentration is the highest among the basin’s sediments measured or with the grater probability—the impact of unstudied factor/factors (environmental and/or anthropogenic) in the survey.

Bioassay battery used for the toxicity of the port of Bari revealed that the worst biological effects were highlighted in the Passenger Terminal. A significant reduction of bioluminescence in the bacterium *V. fischeri* applied to the solid phase of samples was revealed in almost all stations, except the approach channel of the port where low toxicity results are registered. The algal bioassay with *D. tertiolecta* on elutriate underlined a high toxicity in the stations located within some station within the Passenger Terminal, the Ro-Ro Terminal and the Container Terminal (BA 42–BA 51). Similar results are revealed with sea urchin *P. lividus* that evidenced severe toxicity within the same stations revealed by bioassay with algae *D. tertiolecta*. The total toxicity of each sampling stations was expressed as ratio among the R_i = score value assigned for each sampling station and R_{ij}, the maximum total score value achievable ($T = R_i / \sum R_{ij}$) (Fig. 1.6). The worst values registered in the Passenger Terminal are probably related to the bioavailability of contaminants mixtures within this area.

1.4 Multivariate Analysis

The integration of chemical and bio-toxicological data is performed through different multivariate analysis. The analysis of the variables aggregated by PCA, a representation of estimated factors score from each station of the centroid of all

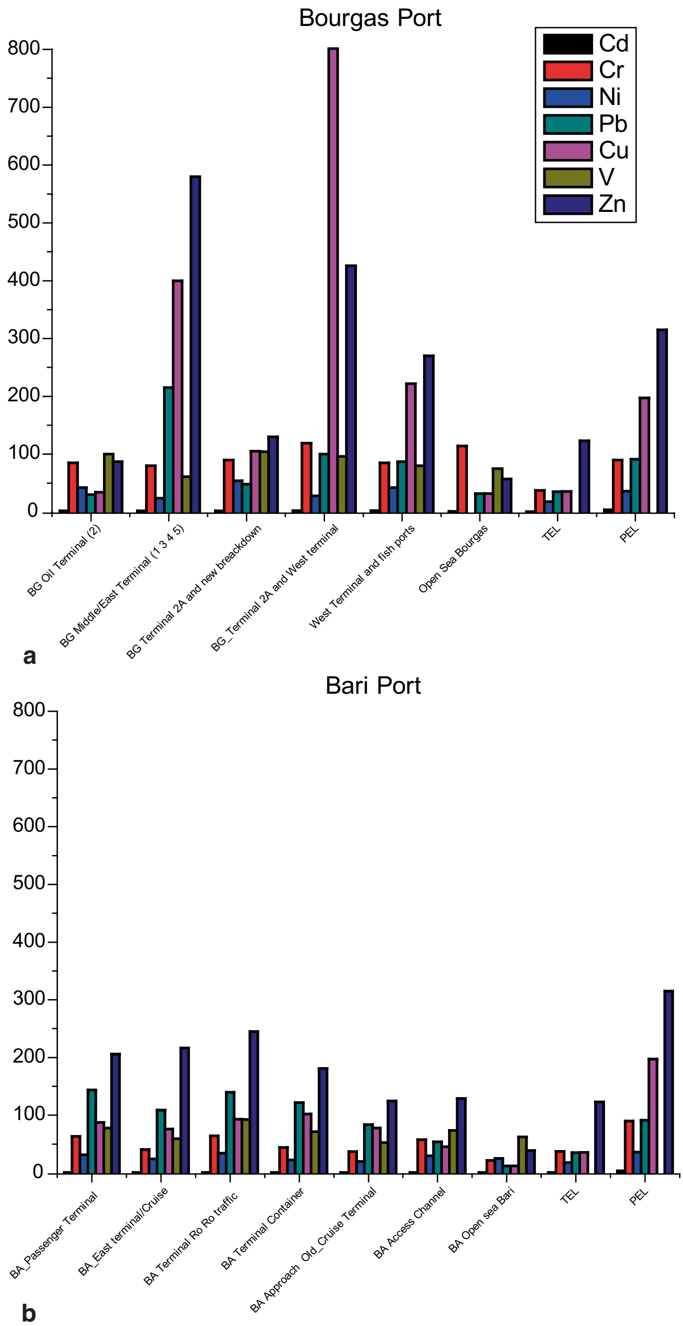

Fig. 1.4 Heavy metal concentrations and comparison with *TEL* (threshold effect level) and *PEL* (probable effect level) threshold values according to MacDonald et al. (1996). **a** Within the sediments of the Bourgas port. **b** Within the sediments of Bari port

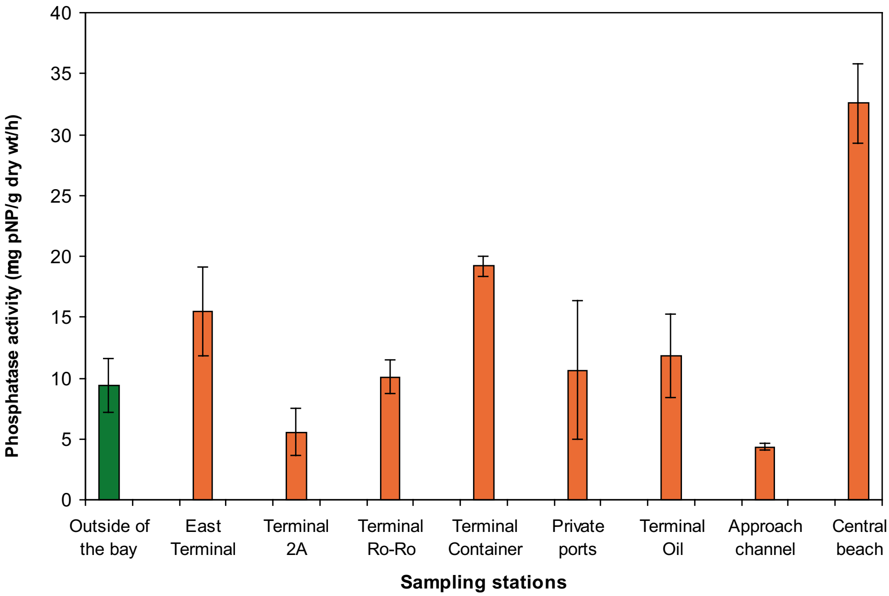

Fig. 1.5 Bacterial phosphatase activity (μg/mg organic matter/h) in the sediments of sampling stations

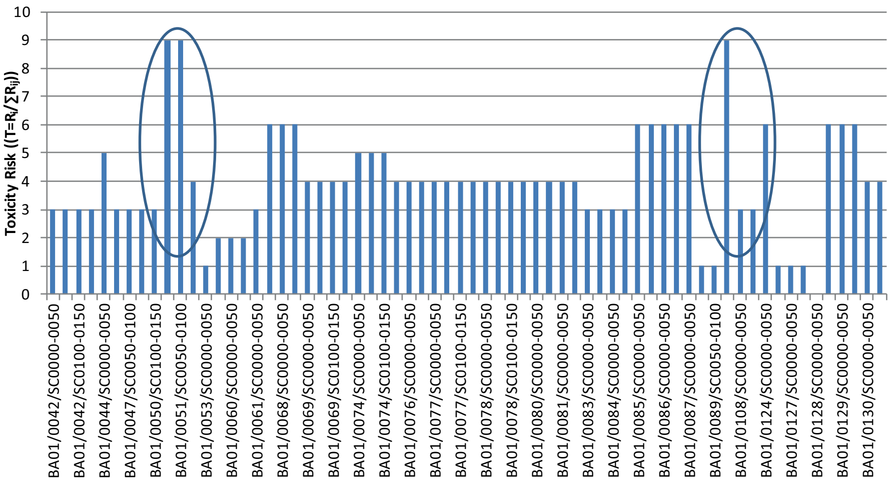

Fig. 1.6 Toxicity in the sediments of Bari sampling stations. The stations which are marked with a *circle* belong to the Ro-Ro Terminal (BA51) and Passenger Terminal (BA89)

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 port.

1.4.1 Port of Bourgas

By means of the application of a PCA, the chemical and bio-toxicological data were represented by three new variables, or principal factors, which explain 89.79 % of the variance in the original data set (Fig. 1.7). The first principal factor (F1) is predominant and accounts for 46.55 % of the variance; this factor related almost all heavy metals (except Ni, V, THC) to toxicity responses in the whole sediment toxicity tests with bacterial phosphatase activity. The correlation between chemical and bacterial phosphatase activity is very much lower in factor 2 and in factor 3 (both accounting 23.60 and 19.64 % of the total variance, respectively). In any case, they show the expected relation between total organic matter and phosphatase activity.

1.4.2 Port of Bari

Chemical concentrations in port of Bari sediments were associated by PCA with toxicity resting in three principal factors (Fig. 1.8). Such factors explained 93.82 % of the total variance in the original data set. The first principal factor (F1) was predominant and accounted for 67.96 % of the total variance. This factor combines the toxicity with the concentration of Cu, Pb, and Zn. The second factor accounts for 16.76 % of the variance and combines the toxicity with total hydrocarbon and concentration of Cd in sediments. The contribution of the Passenger Terminal and Ro-Ro/Cruise Terminal is determinant for the correlation detected.

The results of this investigation show that the contamination is closely related the high concentration of heavy metals in both area, as in Bari as in Bourgas area. It means that the binding forms of such metals or chemical mixtures within sediments are available for the benthic community. The environmental degradations related by hydrocarbon are also revealed in both areas. Almost all the toxic areas identified are related to the anthropogenic activities within port basins. Because of the environmental degradations in the studied ecosystems and the different legal frame assessment, it was difficult to find a satisfactory referee area. By using the PCA, such problem was minims, and thus inferences could be made about the sediment quality of both ecosystem with or without reference area. The multivariate analysis approach was considered also very useful since it combines different types of data—chemical and biological in different ecosystem. A common approach for both areas could lead into definition of international protocols.

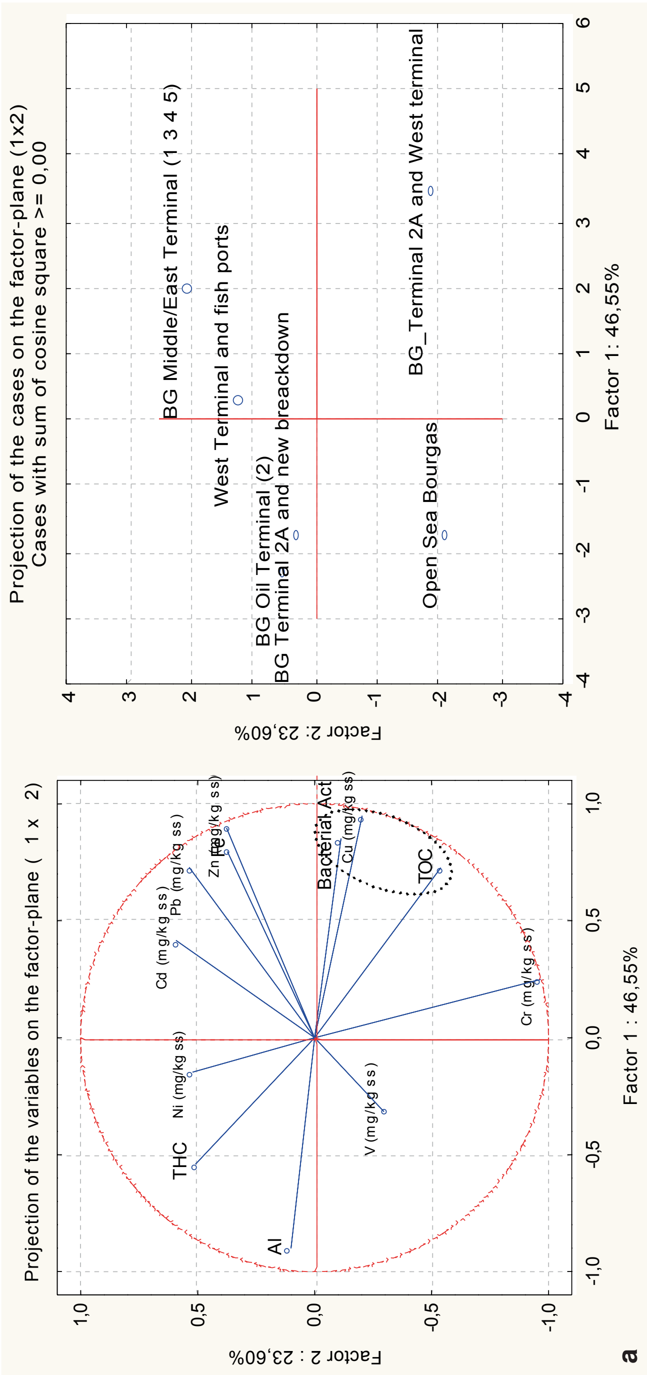

Fig. 1.7 Factor loading projection on factor plans for Bourgas port. **a** Projection of score and loading plot in the 2D on PC1–PC2. **b** Projection of score and loading plot in the 2D on PC1–PC3. *TOC* total organic carbon

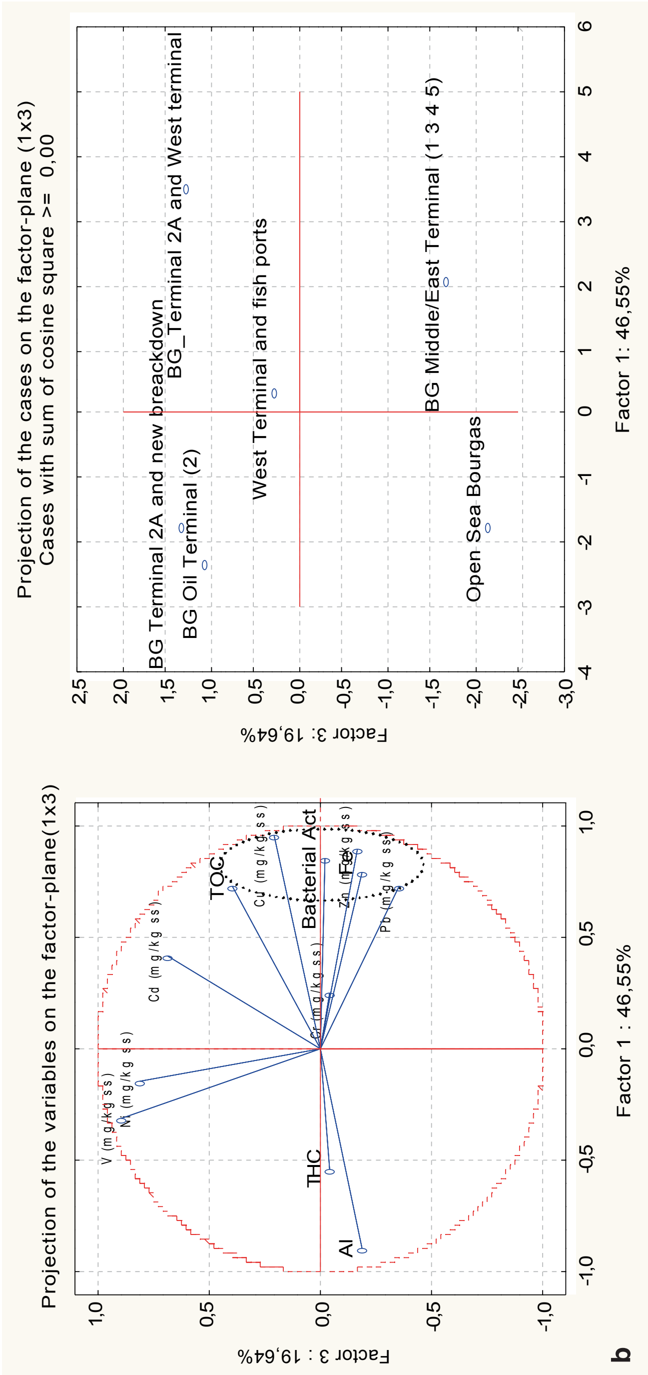

Fig. 1.7 (continued)

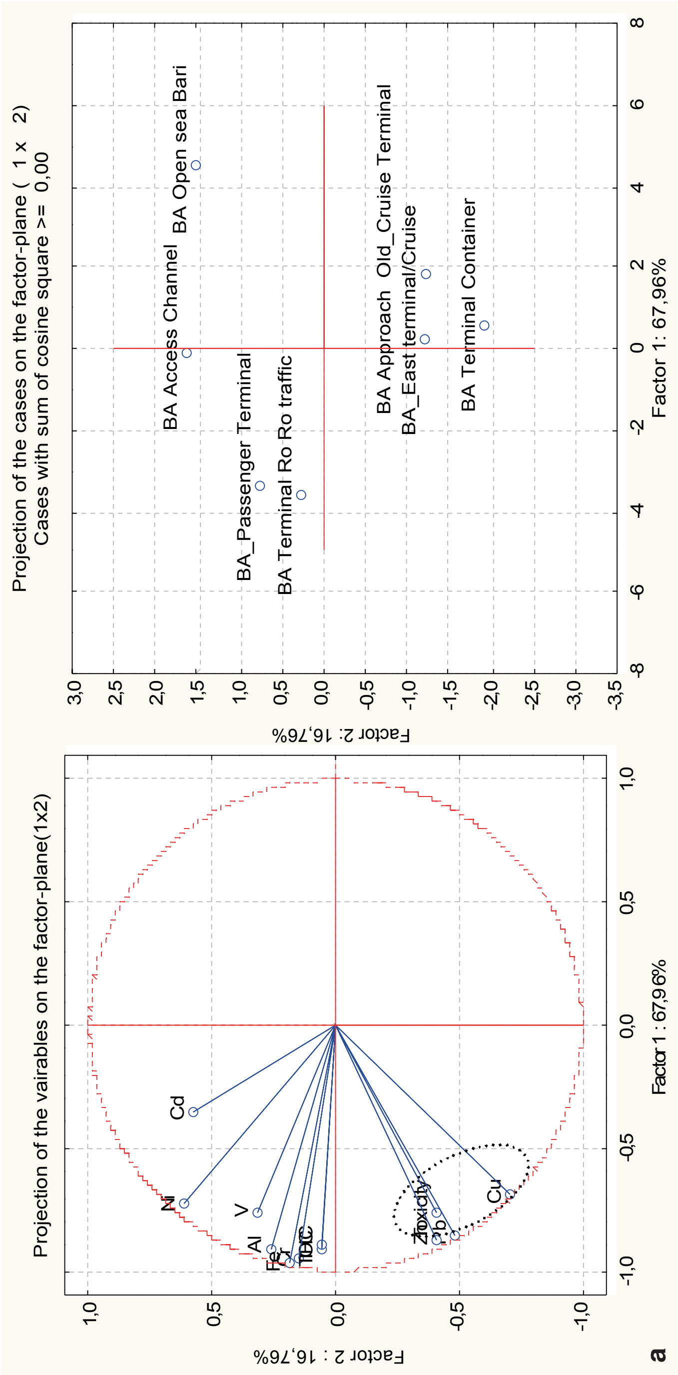

Fig. 1.8 Factor loading projection on factor plans for Bari port. **a** Projection of score and loading plot in the 2D on PC1–PC2. **b** Projection of score and loading plot in the 2D on PC1–PC3. *TOC* total organic carbon

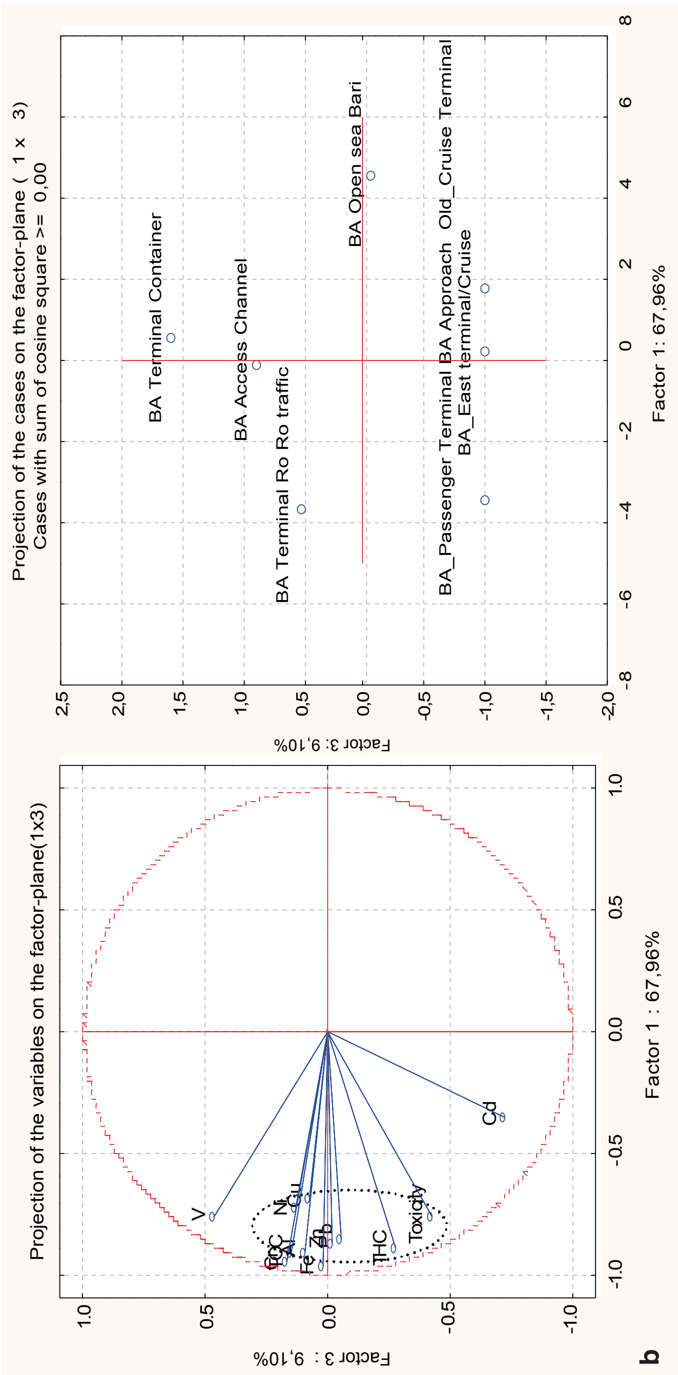

Fig. 1.8 (continued)

1.5 Conclusions

Harbor sediments are both source and sink of persistent contaminants in the port aquatorium area. It is difficult to determine cause-effect relationships resulting from mixtures of chemical contaminants found in harbor sediments, so the development of sediment quality values is moving forward on integrated approach that combines the potential cause (chemistry) and effect measurements (biology) providing a complete and powerful tools available today to determine the extent and significance of pollution-induced degradation. The integration of environmental data can be performed through different univariate and multivariate techniques: Multivariate analysis permits the integration of data of different natures (chemical, biological, toxicity endpoints, or benthic descriptors) resulting in a wider analysis that allows deeper and robust interpretation of the data. In this way, an internationally harmonized protocol would be developed available to standardize the procedures for the assessment and managing of contaminated sediment among countries allowing thus an inter-comparison of the techniques and the obtained results.

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