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## Minerals as additives for decreasing the toxicity of Mediterranean contaminated dredged sediments

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### ABSTRACT

The management of dredged sediments is a priority issue in the Mediterranean sea where sediments are historically polluted. The aims of this study were to evaluate the toxicity of port sediment samples and the effect of three mineral additives (hematite, zerovalent iron (ZVI) and natural zeolite (NZ)) on sediment elutriate toxicity. Four sediments (A, B, C and D) were provided by port authorities after composting procedure; particle size, particulate organic carbon, metals and organic pollutants (TBT, PAHs, PCBs) were determined in whole sediments. Elutriates from these composted sediments were analyzed by determining toxicity level using oyster (*Crassostrea gigas*) larvae bioassay, metal and dissolved organic carbon concentrations. Toxicity, measured on undiluted elutriates (250 g/L), decreased as follows:  $A \geq B > C \sim D$ . The treatment of sediments with mineral additives (5%) revealed that hematite tends to decrease the elutriate toxicity in all samples, particularly in samples B and C. This effect may be related to metal concentration decrease in elutriates, in particular Cu and Zn, that have a significant toxic effect on oyster larvae. ZVI and NZ have a variable influence on elutriate toxicity. Results suggest that hematite may be a possible candidate for decreasing chemical concentration and improving the quality of elutriates. Hematite could be used for sediment stabilization prior to the deposit in a specific site or landfill.

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### 1. Introduction

The dredging operations for coastal ports and waterways consist mainly in removing sediments to improve the ship traffic. In the world 600 million m<sup>3</sup> (Boutin, 1999) of sediments and 50 million m<sup>3</sup> in France (Alzieu et al., 1999) are dredged out each year from the main maritime and commercial harbors. The management of dredged sediments is a priority issue more especially in the Mediterranean sea where sediments are historically polluted (RNO, 1998). At the international level, regulations concerning dredged material and contaminated sediments are established mainly by the international commission OSPAR in the North-East Atlantic Ocean and the Barcelona convention for the Mediterranean sea. These international guidelines are translated into national criteria for marine dredged sediment management. Thus in France, the management of dredged sediments is regulated by the ministerial order of June 14th, 2000, which sets two reference concentration levels N1 and

N2 for metals and PCBs in the whole sediment. These thresholds represent landmarks to evaluate the possible impact on marine ecosystems. Below level N1, the potential impact is theoretically considered as not significant, the concentrations being comparable to the environmental geochemical background. Between levels N1 and N2, toxicity tests are needed according to the future management. Over level N2, the discharge into the sea is forbidden and terrestrial management must be done (Alzieu and Quiniou, 2001). When contaminated sediments are discharged on terrestrial site many precautions must be taken since contaminants can be released from the deposit after waterlogging (Lions et al., 2007; Piou et al., 2009). In this context, most remediation technologies for soil or mineral ores can be used in order to limit the release of pollutants via liquid phase to aquatic ecosystems. The main stabilization process aims at enhancing metal sorption, precipitation and complexation capacity and decreasing the potential mobility or bioavailability of toxic metals in the environment. As an example, Solvay Company patented a phosphatation process (Zoubair et al., 2007) based on the formation of apatite able to trap and fix most of metals, consecutively to addition of phosphoric acid in sediment. Indeed, apatite added to a contaminated sediment immobilized almost

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metals (Pb, Mn, Co, Cu, Cd, Zn, U), but some limitations for anionic species, such as As, Mo and Cr have been highlighted (Arey et al., 1998; Seaman et al., 2001; Heinz et al., 2004).

This research investigated the possibility of reducing sediment elutriate toxicity after mixing different mineral additives with port dredged sediments, which have been already composted. Iron additives (hematite and zerovalent iron) have been studied for remediation of soils contaminated with inorganic pollutants, such as chromated copper arsenate (CCA) (Hartley et al., 2004; Kumpiene et al., 2006, 2009; Maurice et al., 2007). Iron hydroxides can be effective sorbents for both anions and cations since the surface of hydroxide particles can be positively or negatively charged depending on pH making the Fe hydroxides amphoteric (Cornell and Schwertmann, 2003). Natural zeolites, as aluminosilicate mineral, can also be considered since they have characteristics like large surface area, strong capability of ion exchange and adsorption for their particular tetrahedral pore framework. Moreover, they are one of the low-cost and easily obtaining materials, which have been used as adsorbent for removal of metals (Babel and Kurniawan, 2003; Kocaoba et al., 2007; Gupta and Rastogi, 2008; Panuccio et al., 2008).

Among the toxicity tests considered for sediment quality assessment, the oyster (*Crassostrea gigas*) embryo–larval development test was selected to test sediment elutriate since previous studies demonstrated that it is one of the most sensitive and rapid bioassays to check sediment samples providing sub-chronic effects (His et al., 1997; Galgani et al., 2006, 2009; Mamindy-Pajany et al., 2010a). Moreover, Gourmelon et al. (2003) selected oyster embryo–larval test to study the toxicity of waters from sediment terrestrial deposit. The use of elutriates is also recommended in cases of sediment dredging activities and re-suspension phenomena in aquatic environments for their ability to give information on potential effects towards sensitive species including gametes, embryos and larvae (Beiras, 2002; Nendza, 2002; Quiniou et al., 2007).

Sediments were dredged from different ports along the French Riviera from Marseilles to the Italian boarder. To date this coastal area is known to be contaminated by mineral and organic compounds (Andral et al., 2004; Damiens et al., 2007), in particular, the area of Toulon (French navy harbor). In this area, the French Réseau National d'Observation: RNO (RNO, 1998) already monitored hot spots of metals in sediments from ship chandler sites. High TriButylTin (TBT) concentrations were also reported from the French Riviera (2.9 mg Sn kg<sup>-1</sup> for sediments of Cannes port) (Cassi et al., 2008).

Sediment samples were provided in the framework of the monitoring project SEDIMARD launched in 2002 by the French public administration from the Var area in order to examine the potential issues about the management of dredged contaminated sediments in collaboration with port authorities from France and Italy. After some composting treatments, four sediment samples from Côte d'Azur area were taken into consideration. Whole composted sediments were analyzed for pH, organic carbon, metals and organic pollutants. Toxicity to oyster embryos, metals and dissolved organic carbon were measured in elutriates from composted sediments and from those treated with iron additives and natural zeolite (NZ).

## 2. Materials and methods

### 2.1. Sediment dredging and sample collection

Sediments were collected from the French Mediterranean coast using a Shipek grab and laid down over a geotextile in a terrestrial disposal site. They were actively composted for four months under sunlight. During that time, once a week sediment mechanical turn over and humidification took place in order to favor the

**Table 1**  
Composition of mineral additives.

Additive	D50 (μm)	Supplier	Composition
Hematite (H)	0.77	Alfa aesar	99.5%
Zerovalent iron (ZVI)	16.9	Sigma aldrich	99.9%
Natural Zeolite (NZ)	7.2	Zeochem (France)	Clinoptilolite, 84% Cristobalite, 8% Feldspars, 4% Illite, 4%

microbial activity, lower sediment salt content and decrease organotins concentrations (e.g. TBTs) (Grosdemange et al., 2008). After treatment, four homogeneous sub-samples were taken into consideration: two sediments from Côte d'Azur area (navy harbor, sample A and commercial port, sample B), and two other composite samples obtained after mixing A and B with several port sediments (Mamindy-Pajany et al., 2010a). All specimens were stored at 4 ± 1 °C in the dark before analyses. Composted sediment samples were treated by three mineral additives: hematite (H), zerovalent iron (ZVI) and natural zeolite (NZ) in order to evaluate the potential effect of additives on elutriate toxicity. These additives were already tested in batch and small-scale experiments at three concentrations: 5%, 10% and 15%; results generally showed a decrease in mineral pollutants in all cases (Mamindy-Pajany et al., 2010b). Therefore, the lowest concentration (5%) giving sufficient pollutant decreases was used. For this purpose, 50 g of each sediment samples (A, B, C and D) were stabilized with 5% of hematite, zerovalent iron (ZVI) and natural zeolite (NZ). The composition of each mineral additive is presented in Table 1.

### 2.2. Whole sediment physico-chemical analyses

The water content (%) was determined after gravimetric assessment by drying sediment samples in three replicates at 105 °C for 24 h. The pH of the sediment suspensions was monitored by a WTW pH meter, with a combined pH electrode, calibrated using buffer solutions at pH 7.01 and 4.00 at room temperature. Whole composted sediment chemical data were obtained from the SEDIMARD project (Grosdemange et al., 2008). Organotins (tributyltin, TBT; dibutyltin, DBT and monobutyltin, MBT) analyses were carried out by Ultra Trace Analyses Aquitaine (UT2A) Laboratory of Pau (France): after extraction in acetic acid medium, organotin derivatization was performed by adding tetraethylborate solution and after centrifugations TBT was analyzed using a quadrupole instrument and gas chromatography–tandem mass spectrometry (GC–MS–MS). PAHs were analyzed using high-pressure liquid chromatography HPLC 1100 (Agilent) combined with variable wavelength fluorometer. Total PAHs are defined by the sum of six PAHs (fluoranthene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, benzo(ghi)perylene and indeno(1,2,3-cd)pyrene). PCBs were analyzed with a gas chromatograph 6890 Agilent 5973MSD equipped with mass spectrometry detection. Particulate organic carbon was measured by thermal oxidation followed by infra-red detection. Inorganic pollutants (As, Cd, Cu, Mo, Ni, Pb, Se and Zn) were determined by inductively coupled plasma–mass spectrometry (ICP–MS). Particle size distribution of composted sediments was determined by laser granulometer (Coulter<sup>®</sup>). Granulometry size (D50) of hematite, ZVI and NZ was obtained in the same way (Mastersizer 2000, Malvern Instruments).

### 2.3. Elutriate preparation

Elutriates were prepared with seawater that was collected offshore Bastia (Corsica) and considered as reference water (Galgani et al., 2009). Reference water was filtered on 0.22 μm membrane just before use. The 1:4 (sediment:water) ratio was used for evaluating the potential effects of contaminated sediments. For this purpose, 5 g of each untreated and treated sediment samples were mixed with 20 mL of filtered reference water (i.e. 250 g/L) and shaken for 8 h before 8 h decantation.

### 2.4. Elutriate chemical analyses

The dissolved organic carbon (DOC) was determined using TOC-5050 (Shimadzu). Filtered (0.45 μm) samples were diluted 25 times to prevent salinity interference and inorganic carbon was stripped away through air bubbling for 30 min at 20 ± 5 °C after acidification with H<sub>3</sub>PO<sub>4</sub>. Inorganic pollutants (As, Cd, Cu, Mo, Ni, Pb, Se and Zn) were measured by ICP–MS (ELAN DRC II) in elutriates which were filtered (0.45 μm) and then acidified.

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