



Arsenic in marine sediments from French Mediterranean ports: Geochemical partitioning, bioavailability and ecotoxicology



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ABSTRACT

This work investigates arsenic mobility, bioavailability and toxicity in marine port sediments using chemical sequential extraction and laboratory toxicity tests. Sediment samples were collected from two different Mediterranean ports, one highly polluted with arsenic and other inorganic and organic pollutants (Estaque port (EST)), and the other one, less polluted, with a low arsenic content (Saint Mandrier port (SM)). Arsenic distribution in the solid phase was studied using a sequential extraction procedure specifically developed for appraising arsenic mobility in sediments. Toxicity assessment was performed on sediment elutriates, solid phases and aqueous arsenic species as single substance using the embryo-toxicity test on oyster larvae (*Crassostrea gigas*) and the Microtox test with *Vibrio fischeri*. Toxicity results showed that all sediment samples presented acute and sub-chronic toxic effects on oyster larvae and bacteria, respectively. The Microtox solid phase test allow to discriminate As-contaminated samples from the less contaminated ones, suggesting that toxicity of whole sediment samples is related to arsenic content. Toxicity of dissolved arsenic species as single substance showed that *Vibrio fischeri* and oyster larvae are most sensitive to As(V) than As(III). The distribution coefficient (Kd) of arsenic in sediment samples was estimated using results obtained in chemical sequential extractions. The Kd value is greater in SM (450 L kg⁻¹) than in EST (55 L kg⁻¹), indicating that arsenic availability is higher for the most toxic sediment sample (Estaque port). This study demonstrates that arsenic speciation play an important role on arsenic mobility and its bioavailability in marine port sediments.

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1. Introduction

Sediments in estuaries and coastal areas constitute important sinks of contaminants and represent potential sources of pollution to the marine environment. A number of studies carried out in recent years has shown the presence of a wide range of inorganic (As, Cd, Cu, Ni, Pb, and Zn) and organic contaminants (PAHs and PCBs) in Mediterranean port sediments (Andral et al., 2004; Aplitz et al., 2007; Mamindy-Pajany et al., 2010a). Remobilisation of sediment-associated contaminants can occur during natural events (i.e. tidal movement and storms) or anthropogenic activities (i.e. dredging, marine disposal of dredged materials and capping) and

increase the bioavailability of pollutants in water column (Eggleton and Thomas, 2004). Due to its high concentration in marine sediments, arsenic can cause acute and chronic toxicity to marine organisms via ingestion of particulate matter (As associated with particles), through membrane-facilitated transport or passive diffusion (As dissolved in water) (Bhattacharya et al., 2007).

Within the framework for the management of dredged sediment, a French expert group proposed sediment quality guidelines (N1 and N2) for arsenic and metals in marine sediments (Alzieu and Quiniou, 2001). Below the level N1, the ecological impact is view as negligible. Between N1 and N2, chemical analyses must be supplemented with toxicity tests. When their contamination level is higher than N2, dredged sediments cannot be discharged into the sea and must be treated or stored on terrestrial environment. In French Mediterranean ports, elevated arsenic concentrations can be found in whole sediments and they often exceed national sediment quality guidelines (Level 1 = 25 mg kg⁻¹ and Level 2 = 50 mg kg⁻¹) (Mamindy-Pajany et al., 2010b). For example, in

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the military port of Toulon arsenic level is 4-fold higher than the level 2, suggesting that a significant ecological risk is expected for marine ecosystems and living organisms (Mamindy-Pajany et al., 2010b). The distribution and transport of arsenic in sediment is a complex process, depending on oxidation state, water composition, native bacteria, and sediment type (Guo et al., 1997; Bhattacharya et al., 2007). It has become evident that total concentration alone is not sufficient for a full assessment of arsenic bioavailability and its potential mobility in sediments, thus solid phase speciation studies were developed to characterize arsenic bioavailability in ecosystems and explain its mobility. Published data are focused on the distribution of arsenic within undisturbed sediment and its affinities to the various solid-phase fractions of sediment (Bhattacharya et al., 2006; Baig et al., 2009). Many sequential extractions used for As are based on conventional schemes used to extract metals and other elements that form cations. However, several works highlighted the anionic behaviour of As in sediments, and specific schemes were developed for this metalloid (Gleyzes et al., 2002). In all sequential extraction schemes, arsenic can be extracted in an easily sorbed phase, an Al-, Fe-and/or Mn-oxyhydroxide phase and a residual phase. Other operationally defined phases extracted include water or easily soluble, acid volatile sulphide, organic matter, acid-soluble, As oxide and silicate and As sulphide. In oxic and iron-reducing conditions, arsenic species can form strong complexes with iron hydroxides or may be present in oxide minerals. In sulfidic environments, As may adsorb or coprecipitate with pyrite, or form As (III) sulphide minerals (Hudson-Edwards et al., 2004; Wilkin and Ford, 2006).

In the present paper, chemical and toxicity tests were performed to characterize mobility, bioavailability and toxicity of arsenic in marine sediments. Embryo-toxicity and Microtox solid phase tests were selected as bioassays since they provide ecologically relevant information and are rapid and cost-effective screening tools. The oyster (*Crassostrea gigas*) embryo–larval development test is one of the most sensitive and rapid bioassay to check sediment samples considering sub-chronic effects (Geffard et al., 2002; Libralato et al., 2008). Microtox test is an acute toxicity test based on the inhibition of bioluminescence of the marine bacterium *Vibrio fischeri* (Volpi Ghirardini et al., 2009). Microtox solid phase test (MSPT) allows an evaluation of the toxicity of resuspended sediments; the luminescent bacteria can be in contact with toxicants adsorbed on the particles or dissolved in the water. The geochemical partitioning of As was studied using a sequential extraction procedure specifically developed for appraising arsenic mobility in sediments (Keon et al., 2001). This procedure intends to differentiate the following pools of solid phase arsenic: weakly

and strongly adsorbed As; As co-precipitated with metal oxides or amorphous monosulfides; As co-precipitated with crystalline Fe (oxyhydr)oxides; As oxides; As co-precipitated with pyrite and As sulphides (Keon et al., 2001). All experiments were performed with two different Mediterranean marine sediments, one highly polluted with arsenic and other inorganic and organic pollutants (Estaque port (EST)), and the other one, less polluted, with a low arsenic content (Saint Mandrier port (SM)). These sediment samples were well characterized in previous work for mineralogy and physico-chemical composition of solid phases and pore waters (Battaglia-Brunet et al., 2010).

2. Materials and methods

2.1. Sediment sampling

Sediment samples were collected on Mediterranean coast in two pleasure ports: Estaque port (EST), highly contaminated with arsenic by several industrial activities and Saint Mandrier port (SM), chosen as a reference site for its low arsenic concentration.

Two different types of samples were collected for each site: surface sediments (0–10 cm) and depth sediments (10–20 cm). In EST port, two sampling campaigns were performed: one in cold season (March 2009) and another in hot season (September 2009) whereas SM port sediments were considered only in March 2009. The physico-chemical characterization of sediment samples was described in previous work (Battaglia-Brunet et al., 2010). The global risk was estimated in previous work, for each site, using Q_{PEC} (risk quotient) values (Battaglia-Brunet et al., 2010). Results showed that all sediment samples may present a risk for marine environment since their Q_{PEC} values are higher than 1. The ecological risk is two times higher at EST site, and it is mainly driven by arsenic and three other elements (Cu, Pb, Zn). In SM sample, the ecological risk is low and is driven by Cu and Pb (Battaglia-Brunet et al., 2010). However, this ecological risk parameter does not take into account organic and organo-metallic compounds.

Pollutant concentrations (expressed as $mg\ kg^{-1}$ dry weight) are reported in Table 1 for EST_S1 (Estaque surface, March 2009), EST_S2 (Estaque surface, September 2009), EST_D1 (Estaque depth, March 2009), EST_D2 (Estaque depth, September 2009), SM_S1 (Saint Mandrier surface, March 2009) and SM_D1 (Saint Mandrier depth, March 2009). Arsenic speciation is also reported in Table 1 for all sediment samples and results show that As(III) is the predominant form in Estaque sediments whereas As(V) is the major arsenic specie in Saint Mandrier sediments. No methylated arsenic

Table 1

Chemical concentrations (expressed as $mg\ kg^{-1}$ dry weight) in sediment samples from Saint Mandrier and Estaque (surface and depth) collected in March and September 2009. French sediment quality guidelines (N1 and N2 levels) are also reported (Alzieu and Quiniou, 2001).

Pollutants	Units	EST_S1	EST_D1	EST_S2	EST_D2	SM_S1	SM_D1	N1	N2
As(III)	$mg\ kg^{-1}$	148	170	73	186	1.4	4	–	–
As(V)	$mg\ kg^{-1}$	46	29	34	34	8.6	8.1	–	–
Total As	$mg\ kg^{-1}$	194	199	107	220	10	12.1	25	50
Cd	$mg\ kg^{-1}$	0.3	0.4	–	–	–	–	1.2	2.4
Cu	$mg\ kg^{-1}$	278	273	–	–	220	194	45	90
Ni	$mg\ kg^{-1}$	20	24	–	–	15	19	37	74
Pb	$mg\ kg^{-1}$	329	412	–	–	110	108	100	200
Zn	$mg\ kg^{-1}$	375	463	538	631	221	209	276	552
Mineral oils	$mg\ kg^{-1}$	1141	1646	–	–	588	250	–	–
\sum PAHs	$mg\ kg^{-1}$	2.3	5.9	–	–	1.2	0.3	–	–
\sum PCBs	$mg\ kg^{-1}$	0.12	0.16	–	–	0.10	0.10	–	–
TBT	$\mu g\ Sn\ kg^{-1}$	374	59	–	–	179	43	400	1000
DBT	$\mu g\ Sn\ kg^{-1}$	224	63	–	–	142	32	–	–
MBT	$\mu g\ Sn\ kg^{-1}$	347	39	–	–	197	37	–	–

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