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Is the 1:4 elutriation ratio reliable? Ecotoxicological comparison of four different sediment:water proportions

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Abstract

Methodological research was carried out to evaluate the discriminatory capability of three toxicity bioassays toward different elutriation ratios (1:4, 1:20, 1:50, and 1:200 sediment:water ratios). Samples from six sampling stations of the Lagoon of Venice have been investigated. The toxicity bioassay results (sea urchin *Paracentrotus lividus* Lmk sperm cell and embryo toxicity bioassays and bivalve mollusk *Crassostrea gigas* Thunberg embryo toxicity bioassays) have shown that elutriates generated from the widely used 1:4 ratio were less toxic than those from intermediate ratios (1:20 and 1:50).

Keywords: Sediment; Elutriate; Elutriation ratios; Toxicity bioassays; Sea urchin; *Paracentrotus lividus*; Oyster; *Crassostrea gigas*; Confounding factors

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1. Introduction

Most contaminants originating from human activities and discharged into surface water are eventually deposited and concentrated in sediment. Due to its propensity to sequester both organic and inorganic contaminants, sediment can be defined as the main sink and source of pollutants. The sediments in estuaries and coastal areas thus constitute important reserves of contaminants and represents potential sources of pollution. In particular, marine disposal of dredged material, dumping of wastes, and shipping and fishing activities can play major roles in sediment particulate resuspension, a possible source of pollution (USEPA, 1977; Forstner and Wittmann, 1983). In sediment, in addition to independent actions of contaminants, the presence of complex mixtures could produce additive, synergic, and antagonistic interactions. Their concentrations in sediment may be several orders of magnitude higher than that in overlying water; however, bulk sediment concentrations are not highly correlated to bioavailability (Burton, 1992).

Elutriate is an environmental matrix that enables the replication of sediment mobilization phenomena (Shuba et al., 1978) and the prediction of the release of contaminants from the sediment to the water column (ASTM, 1990). It was first developed for evaluating the potential effects of disposing of dredged material in open water and is nowadays also applied to the quality evaluation of in situ sediment (Beiras et al., 2001; Lazorchak et al., 2003).

Briefly, the elutriation procedure consists of the vigorous shaking of a predetermined part of sediment with parts of water to release sorbed pollutants. This mixture is allowed to settle and the liquid phase is centrifuged (ASTM, 1991; Burton and MacPherson, 1995). Analyses of elutriate samples provide information on the water-soluble constituents potentially released from the sediment to the water column. The method has been proved suitable for detecting altered and toxic sediment, supplying information on water-bioavailable components (Williams et al., 1986).

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Several methods of elutriate preparation have been proposed in the literature. The main differences consist of sediment:water ratios, sediment shaking techniques, times, and temperatures, and supernatant centrifuging techniques. The 1:4 sediment:water ratio, suggested by USEPA (1991), is the most commonly employed sediment: water proportion (USACE, 1978; Burton et al., 1989; Daniels et al., 1989; Munawar et al., 1989; Long et al., 1990; Ankley et al., 1991; USEPA, 1991; Vashchenko and Zhada, 1993; Andreatta et al., 1994; Hurkey et al., 1994; Bridges et al., 1996; Schuytema et al., 1996; Da Ros et al., 1997; Sibley et al., 1997). Nonetheless, a wide series of alternative ratios have flourished: the 1:2 ratio (Meador et al., 1990; Vashchenko and Zhada, 1993), 1:3 ratio (Matthiessen et al., 1998), 1:5 ratio (Lee et al., 1978; Da Ros et al., 1997), 1:8 ratio (McFadzen, 2000), 1:10 ratio (Daniels et al., 1989; Da Ros et al., 1997), 1:20 ratio (Lee et al., 1978; Daniels et al., 1989), 1:25 (Lee et al., 1978), 1:50 ratio (Long et al., 1990; Van den Hurk, 1994; Van den Hurk et al., 1997; Da Ros et al., 1997), 1:200 ratio (Da Ros et al., 1997), 2:1 ratio (Williams et al., 1986), and 2:5 ratio (Thain et al., 1996). Moreover, Daniels et al. (1989) reported that the choice of the dry or wet weight of sediment to achieve the sediment:water proportions is questionable.

Different sediment shaking devices are also employed: rotary shaker tables (Williams et al., 1986; Ankley et al., 1991; Matthiessen et al., 1998), rotary tumblers (Daniels et al., 1989; Munawar et al., 1989), air systems (USACE, 1978; Daniels et al., 1989; McFadzen, 2000), shakers (Burton et al., 1989; Daniels et al., 1989; Long et al., 1990; Van den Hurk, 1994; Matthiessen et al., 1998; His et al., 1999), magnetic stirrers (Bridges et al., 1996), and ultrasonic baths (Andreatta et al., 1994) have all been proposed. Shaking times varied from about 30 min to 24 h (Ankley et al., 1991; ASTM, 1991; Burton and MacPherson, 1995; Liß and Ahlf, 1997).

Despite all these methodological differences, only limited studies have been conducted to highlight the procedure best able to simulate natural sediment resuspension phenomena or to render pollutants more bioavailable (Daniels et al., 1989; Da Ros et al., 1997).

The aim of this work was to assess the discriminatory capability of three toxicity bioassays toward four different elutriation ratios (1:4, 1:20, 1:50, and 1:200) to evaluate which ratio can produce greater effects for water-column bioindicators. Moreover, this research aimed to determine whether the widely used 1:4 sediment:water ratio represents the best method for a quality control sediment diagnosis.

Static nonrenewal bioassays using sea urchin sperm cells, embryos, and larvae, and oyster embryos and larvae, widely used in biomonitoring programs (ASTM 1998a, b), have been employed to evaluate elutriates toxicity. To evaluate the reliability of the results, the toxicity data are discussed in relation to possible confounding factors (ammonia and sulfide).

2. Materials and methods

2.1. Sampling sites

The sediments were sampled in late summer (August–September 1998) at six sites in the Lagoon of Venice, northeast Italy, as part of the "Orizzonte 2023 Project." The stations were located along a pollution gradient: two presumably unpolluted sites (CE(a) and CE(b)), two oil-refinery and industrial sites (BR and SA), and two estuarine sites (DE and OS) close to agricultural areas.

At Centrega Marsh (CE), in the northern Lagoon, CE(a) and CE(b) were chosen as two possible reference sites, intertidal and subtidal, respectively, because of their location characterized by a fast water renewal time (due to the vicinity of the Lido sea inlet), morphological diversity, high biodiversity, and expected minimal contaminant levels. Previous investigations in this area had reported very low levels of some heavy metals (Volpi Ghirardini et al., 1998, 1999) and organic micropollutants (Di Domenico et al., 1998).

The South Industrial Canal (SA) and Lusore-Brentelle Canal (BR) are located within the Porto Marghera industrial zone. The Lusore-Brentelle Canal samples can be expected to be extremely polluted according to Spoladori (2004). Some information on sediment contamination of both industrial canals is already available (Di Domenico et al., 1998; Volpi Ghirardini et al., 1998, 1999; MAV, 2004).

The DE and OS sampling stations are at the mouths of the Dese (DE) and Marzenego (Osellino Canal, OS) rivers, respectively. Both stations are influenced by freshwater carrying agricultural runoff. In addition, OS is affected by multifactorial contamination due to urban (treated and untreated wastewaters from the town of Mestre) and industrial (vicinity of Porto Marghera industrial zone) sources and to an uncontrolled landfill of the Campalto saltmarshes, just behind the Osellino Canal (i.e., possible release of leachates), where confining works (CVN, 1999) were started after the end of this project.

Detailed data on particle size fractions (percentage of sand, silt, and clay), total organic carbon (TOC) (percentage of dry weight), organic matter (OM) (loss on ignition at 450 °C overnight, percentage dry weight), and water content (W) (105 °C overnight; percentage of water content) (ASTM, 1990), chemical analyses of metals (in the acid extracts determined by ICPM according to USEPA 1998) and organic micropollutants concentrations (extraction at 4 °C with n-hexane/dichloromethane 50/05 at 150 °C and 1500 psi; analyses conducted using a HP 6890 plus gas chromatograph coupled to a Micromass Autospec Ultima mass spectrometer; determinations of PCDDs/PCDFs and PCBs-HCB performed according to the USEPA, 1994, 1999), and the geographical coordinates of the sampling stations (Volpi Ghirardini et al., 2005) are reported in Table 1.

2.2. Sediment collection, characterization, and storage

Samples were collected at low tide by a 5-cm-diameter Plexiglas corer. At each site, the area (a circle of approx. 30 m diameter, with a central point fixed by geographical coordinates) and sample sizes (eight sediment cores at depths of 15–20 cm) were defined, and integrated sampling (with cores equally distributed over the area) was done to take any spatial microvariability in pollution/bioavailability into account, according to the scheme reported in Volpi Ghirardini et al. (2005).

Cores from each station were pooled in 2-L glass jars and stored at 4 °C. At 1 to 2 days after sampling, each integrated sediment sample was homogenized and sieved (1 mm mesh size), and subsamples were prepared for sediment characterization and chemical analyses and processed to obtain elutriates for toxicity bioassays.

2.3. Elutriation procedure

In preparing elutriates, the USEPA (1991) standard procedure was used as reference, but some modifications based on the more recent