



## Geochemical assessment and fractionation of trace metals in estuarine sedimentary sub-environments, in Mumbai, India



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### ABSTRACT

The distribution and concentrations of selected metals (Fe, Mn, Cu, Pb, Co, Zn and Cr) in intertidal regions (mudflat and mangrove) of Ulhas estuary, located in Mumbai, India were studied. The sediment metal contamination was assessed by applying Enrichment Factor (EF), Anthropogenic factor (AF), Contamination factor (CF) and Pollution load Index (PLI). Also, Sediment Quality Guidelines (SQGs) by Environmental Protection Agency was used to determine the risk of increased metal levels to biota. Amongst the two sub-environments studied, the mudflat region exhibited higher metal contamination than the mangrove region necessitating fractionation analysis of the core. The geochemical fractionation analysis showed higher metal percentage in the residual fraction, except for Mn and Zn. The fractionation results, in general, indicated that contamination due to the metals- Mn and Zn might pose a potential risk to the estuarine system if the sediment gets disturbed with time.

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### 1. Introduction

Metals are indestructible with most of them having toxic effects on organisms. Unpolluted soils/sediments exhibit low concentrations of metals mostly derived from rock and soil weathering (Reza and Singh, 2010; Varol and Sen, 2012), while in urban-rural areas, anthropogenic inputs, such as industry and agricultural emissions, may become predominant metal sources (Li and Davis, 2008; Tang et al., 2014). The pollution of the aquatic environment with metals has become a worldwide problem during recent years (Guo et al., 2012; Ndimele and Kumolu-Johnson, 2012). Sediments can act as a sink for various pollutants such as pesticides and trace metals and also as a source by playing a significant role in the remobilization of contaminants (Yao and Gao, 2007). The contamination of aquatic systems by heavy metals, especially in sediments, has become one of the most challenging pollution issues owing to the toxicity, abundance, persistence and subsequent bio-accumulation of these materials (Gielar et al., 2012; Varol and Sen, 2012). Numerous studies, have demonstrated that marine sediments from industrialised coastal areas are greatly contaminated by heavy metals (Li et al., 2013; Singh et al., 2013; Fernandes et al., 2011). Elevated levels of heavy metals in aquatic sediments, may pose a risk to human health

due to their transfer in aquatic media and uptake by living organisms, thereby entering the food chain (Satarug et al., 2010; Hussein et al., 2012; Tang et al., 2014). The knowledge of the total concentration of an element in specific environmental compartment is often inadequate to explain its roles and properties. Mobility and toxicity of metals associated with sediments are generally affected by metal speciation and sediment composition. Sequential extraction analysis is a technique developed for the overall assessment of the speciation and potential mobility of metals in sediments (Calmano et al., 1996; Hass and Fine, 2010). There are many such schemes (Forstner, 1982; Tessier et al., 1979) amongst which the most widely used is the one proposed by Tessier et al. (1979).

Continuous industrial growth and rapid economic development of Mumbai, in India, has resulted in dramatic increase in percentage of population. Consequently, the environmental quality of the soil is becoming more and more important with regards to human health. Rathod et al. (2002) reported decline in fin fish and shell fish fauna along Mumbai due to industrial and domestic activities. In Ulhas estuary, pollution adversely affected the distribution and abundance of fish species such as *Bhing* and *Pala* (BMRDA, 1983). Relative high concentration of Cr reported in the tissue of fish from the Ulhas estuary reflected anthropogenically added Cr in the estuary (Srinivasan and Mahajan, 1987). Singh et al. (2007) reported presence of heavy metals (Cd, Fe, Pb, Zn and Cu) in water, sediment and body tissues of Red worms (*Tubifex* spp.) collected from natural habitats around Mumbai. Therefore, there is a need to deter-

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mine the chemical form of metals in the soil to know its toxicity, mobilization capacity and behaviour in the environment. There is not much information available in the published literatures about the geochemical assessment and fractionation of heavy metals in the Mumbai area especially the estuarine region. Knowledge of the chemical forms of heavy metals as well as their complexes will help to better evaluate the environmental impact of contaminated sediments. Therefore, the aim of this study was to investigate the metal sources, distributions, concentrations and contamination status of selected elements (Fe, Mn, Cu, Pb, Co, Zn and Cr) in the sediments and compare it with standards for sediment quality as well as determination of mobility of the metals in the sediments.

## 2. Materials and methods

### 2.1. Study area

The study area is located in Mumbai (Table 1), on the west coast of India, as shown in Fig. 1. The Ulhas River estuary is one of the inward waters distinctive in its environmental conditions due to the shallow depth, tidal currents, mangrove vegetation, salinity gradient, diurnal temperature variation, etc. It is lined with vast mudflats and extensive mangrove areas. An outlet of the river touches the head of Thane creek south of Mumbra. Dredging activities carried out in the Mumbra region resuspend the finer sediments and disturb the normal distribution of particulate and dissolved materials along its course. The region receives an annual mean rainfall of 2600–2800 mm during the monsoon season (June–September). The estuary is strongly influenced by tides, with spring tidal range varying from about 4.5 m at lower estuary to about 2.8 m at the upstream end. The river has barrages constructed in the upstream region which permit only limited river discharge into the estuary during the early dry season. The estuarine flushing time has been estimated to vary between 73 and 211 tidal cycles during the dry season. The estuary is subjected to large quantities of untreated industrial wastes situated in Kalyan-Ulhasnagar belt and domestic sewage from surrounding localities. The fuel gathering, reclamation, industrial and domestic pollutants and overfishing are reported to be the main factors involved in the deterioration of the estuary (Qamrul and Savant, 1981; Baig, 1990; Athalye et al., 2007). Besides this, activities such as solid waste disposal, mangrove cutting, reclamation, construction, etc are also observed to be influencing the estuarine environment of the ambient water body (Athalye et al., 2007).

### 2.2. Sampling and methodology

Two sediment cores, one from the mudflat region (28 cm long) and the other from the mangrove region (58 cm long), were collected using PVC pipes and sub-sampled at 2 cm intervals (Fig. 1 and Table 1). All the sub-samples were dried in oven at 50 °C temperature and used for further analysis. The pipette analysis technique (Folk, 1974; ISRIC, 2002) was used for separation of sand, silt and clay components, to illustrate the sediment types. Total organic carbon (TOC) was determined by Walkley and Black method (Sato et al., 2014). For the analyses of Total Phosphorus (TP) and Total Nitrogen (TN) in sediments, standard procedure of Grasshoff (1999) was employed. For total elemental concentration, the sediments were digested using a 7:3:1 mixture of HNO<sub>3</sub>, HClO<sub>4</sub> and HF at 100 °C (Jarvis and Jarvis, 1985). Trace metal concentration of the digested solutions were determined using Varian SpectraAA240 Atomic Absorption Spectrophotometer (AAS). Each sediment sample for metal determination was analyzed in triplicates and the relative standard deviation was found to be <5%. In addition to blanks and replicates, a certified reference material

(BCSS- Marine Sediment Reference Material for Trace Metals and Other constituents) from National Research Council, Canada was included in the analytical procedure, to evaluate the effectiveness of the digestion procedure. The sequential extraction scheme proposed by Tessier et al. (1979) was adopted to partition the heavy metals into five fractions given below;

*Exchangeable (F1)* - 1 g of sediment sample was extracted with 10 ml of 1 M MgCl<sub>2</sub>, adjusted to pH 7 and shaken for 1 h using a mechanical shaker and the filtrate analyzed for metals.

*Carbonate Bound (F2)* - The residue from F1 fraction was leached with 10 ml NaOAc, adjusted to pH 5.0 with acetic acid and shaken for 5 h and the filtrate analyzed.

*Fe-Mn-oxide bound (F3)* - The residue from the F2 fraction was leached with 20 ml 0.04 M NH<sub>2</sub>OH·HCl in 25% (v/v) HOAc for 5 h with continuous shaking at 96 °C. The filtrate was analyzed for metals.

*Organic matter and sulphide bound (F4)* - The residue from F3 fraction was extracted with 10 ml 30% H<sub>2</sub>O<sub>2</sub> adjusted to pH 2.0 with HNO<sub>3</sub> and then extracted with 3.2 M NH<sub>4</sub>OAc at room temperature. The metals were determined in the filtrate.

*Residual (F5)* - The residue from F4 fraction was digested with 7:3:1 of HNO<sub>3</sub>, HClO<sub>4</sub> and HF mixture. The heavy metals (Mn, Co, Zn, Pb, Cr, Cu and Fe) from the extracts were measured by flame AAS.

The quality control and accuracy of the sequential extraction technique was checked from the difference between the total concentrations of the metals and sum of the five fractions. In general, the recovery of the metals ranged between 83 and 110%. Statistical analysis comprising of factor analysis was employed, using Statistica 6.0 software, to determine the metal sources.

### 2.3. Assessment of sediment contamination with metals

Enrichment Factor (EF): To differentiate between the metals originating from anthropogenic (non-crustal) and geogenic (crustal) sources, and to assess the degree of metal contamination, EF values were calculated for each metal of both the cores using Al as the reference element (Feng et al., 2004). The EFs for each element was calculated from the formula

$$EF = (C_n/C_{Al})_{\text{sediment}} / (C_n/C_{Al})_{\text{background}}$$

where,  $(C_n/C_{Al})_{\text{sediment}}$  is the ratio of concentration of the element ( $C_n$ ) to that of Aluminium ( $C_{Al}$ ) in the sediment sample and  $(C_n/C_{Al})_{\text{background}}$  is the same ratio with background value taken of average shale (Turekian and Wedepohl, 1961; Li et al., 2016). The EF values are interpreted as:  $EF < 1$  indicates depletion,  $EF \geq 1$  indicates enrichment while  $EF \geq 1.5$  is considered to indicate that an important proportion of trace metals is delivered from non-crustal materials or non-natural weathering processes, instead from biota and/or point and non-point pollution sources,  $EF > 2$  means significantly enriched.

Anthropogenic Factor (AF): AF was calculated with metal surface concentration and concentration at a depth which enables differentiation between geogenic and anthropogenic input of heavy metals, the results of which would indicate the extent of anthropogenic influence on metal deposition. AF was calculated (Szefer et al., 1998), using the following formula,

$$AF = C_s/C_d$$

where,  $C_s$  and  $C_d$  refer to the concentrations of the elements in the surface sediments and at depth in the sediment column. If AF is >1 for a particular metal, it means contamination exists; otherwise, if AF is  $\leq 1$ , there is no metal enrichment of anthropogenic origin.

Contamination Factors (CF) and Pollution Load Index (PLI): CF was computed utilizing the metal content in sediment and coastal

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