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Mercury speciation in coastal sediments from the central east coast of India by modified BCR method

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Abstract: This is the first study to describe distribution and speciation of Hg in coastal sediments from the central east coast of India. The concentrations of Hg in the studied sediments were found to be much lower than the Hg concentration recommended in the coastal sediments by the United State Environmental Protection Agency and the Canadian Council of Ministers of the Environment for the protection of aquatic life. This study suggests that the interactions between Hg and coastal sediments are influenced by the particle size (sand, silt and clay) of the sediments and the total organic carbon (TOC) content in the sediments. The availability of strong uncomplexed- Hg binding sites in the coastal sediments were observed. It is found that the coastal sediments from the central east coast of India could act as a sink for Hg.

Key words: Mercury distribution, coastal sediments, mercury speciation, Modified BCR method, Hg in coastal sediment around India

Mercury (Hg) is an environmental toxicant of concern because of its pervasiveness and adverse effects on wildlife and human health (US EPA, 1997). The high biomagnification rate of Hg in food chain, makes this metal of the most environmental concern (Fitzgerald et al., 2007; Morel et al., 1998). Global oceans, coastal zones in particular, are acting as reservoirs in the global Hg cycling and many other types of contaminants derived from various sources (Chakraborty, 2011; Chakraborty, 2012; Chakraborty et al., 2012; Chakraborty et al., 2012a; Chakraborty et al., 2012b). It has been reported that coastal sediments act both as a sink and source for toxic metals (Chakraborty et al., 2012b). Sediment contamination in the coastal areas is a major environmental issue because of its potential toxic effects on biological resources and often, indirectly, on human health (Chakraborty et al., 2013; Chakraborty et al., 2013a, Chakraborty et al., 2013b). The major research and monitoring on Hg poisoning have been undertaken mainly in coastal and estuarine sediments in different parts of the world. However, it is unfortunate that the baseline data on Hg distribution and speciation of Hg around India is scarce.

Only a few studies have been reported on the distribution of Hg in the coastal sediments around India. A detailed geochemical and mineralogical study of muddy to sandy mud estuarine sediments of the Hugli River has been reported by Sarkar et al (2004). The spatial distribution of trace element contamination (including Hg) in sediments of the Tamiraparani estuary, southeast coast of India has been reported by Magesh et al (2011). Monitoring and assessment of Hg pollution in the Rushikulya estuary, Orissa, India has been reported by Panda et al (1990). Ram et al (2009) has reported diagenesis and bioavailability of Hg in the contaminated sediments of Ulhas Estuary (west coast), India. The distribution of Hg in the estuarine and near shore sediments of the western Bay of Bengal has been reported by Sasmal et al (1987). This limited data set is old and inadequate to understand the distribution and speciation of Hg in the coastal sediment around India (with a coastline of - 7000 km). It is important to note that the available data in literature only describes the total Hg concentrations and distributions in the coastal sediments.

It has been reported that the grain size of sediments, organic matter content, and chemical composition of different kinds of sediment, as well as the amount of Hg content determine the speciation of Hg in the environment. The toxicity and bioavailability of Hg in sediments is very much dependent on its chemical speciation rather than its total concentrations in the sediments. Non-residual/dynamic complexes of Hg, methylmercury (CH_3Hg^+) in sediments are expected to have strong biological impacts. Thus, it is necessary to determine the distribution and speciation of Hg in the coastal sediments around India. In this study, an effort was made to understand the distribution

and speciation of Hg in the coastal sediments from the central east coast of India and identify the factors which control Hg speciation in the coastal sediments from the central east coast of India.

Sediment samples were collected from five different environmentally significant sites off the central east coast of India as shown in Figure 1. The sites were (1) Bheemili (BHI), (2) Vishakhapatnam (VSKP), and (3) Gangavaram (GVM) (4) Goutami Godavari Estuary (GGE) (5) Kakinada (KKD).

The Bheemili (BHI) site is located in the north of Andhra Pradesh. This city is not industrially developed. The approximate population of this city is ~50,000. Vishakhapatnam (VSKP) is the second largest city in the state of Andhra Pradesh and the third largest city (after Kolkata and Chennai) in the east coast of India. VSKP has become a hub for many heavy industries. The VSKP port, the largest in the country, is the ideal gateway contributing to the development of petroleum, steel and fertilizer industries. The approximate population of VSKP city is 2,000,000.

Gangavaram (GVM) is located close to VSKP city. India's deepest port is situated in GVM.

Sediment samples were also collected from the Goutami Godavari estuary (GGE) (A 100 km² area around Gautami Godavari River has an approximate population of 560,000) and Kakinada. Kakinada is an industrially developing city, and a branch of Godavari River joins the coastal waters through a canal that carries mostly agricultural and municipal sewage to Bay of Bengal. Kakinada has an approximate population of 300,000.

The general description, geographic location of the sampling sites, the distance from the shore, and the depth from where the sediment samples were collected are shown in Table 1.

A Van Veen stainless steel grab (with an area of 0.02 m²) was used to collect the sediments. Without emptying the grab, a sample was taken from the centre with a polyethylene spoon (acid washed) to avoid contamination by the metallic parts of the grab. Multiple sampling was done at each station. The samples were stored at -20 °C for 15 days, and then dried at 30-35°C in a forced air oven (Kadavil Electro Mechanical Industry Pvt Ltd India, Model No. KOMS. 6FD). Sediments were subsequently stored at 4 °C until needed. The texture of the studied sediments was characterized (percentage of sand, silt and clay content) and the data are presented in Table 2.

Total Hg concentrations in the sediments (dry) were determined by direct mercury analyzer. Modified BCR Extraction procedure was used to understand the distribution of Hg in the different phases of the coastal sediments. All the extraction processes were performed in Teflon containers. The reagents used in this study were of analytical grade or better (ultrapure).

The schematic diagram of the protocol is presented in Figure 2. The concentrations of Hg in each extracted solution and in residual fraction were determined by direct mercury analyzer.

All the extractions were in triplicate. Two reagent blanks were analyzed for every extraction. In all cases blank results were below the detection limit of the analytical technique ($0.001 \mu\text{g g}^{-1}$). Hg was analyzed by Direct Hg Analyzer (Tri cell DMA-80) from Milestone, Italy. The DMA-80 is fully compliant with US EPA method 7473.

Distribution of Hg content on the surface sediments from the central east coast of India is shown in Figure 3. The average concentrations of Hg in the studied sediments were relatively lower (ranging from $5.6\text{--}50.0 \mu\text{g.kg}^{-1}$) than the reported Hg concentrations in the coastal sediments from the other parts of the world (Abi-Ghanem et al., 2011; Apeti et al., 2012; Beldowski and Pempkowiak, 2007; Covelli et al., 2001; Fang and Chen, 2010; Horvat et al., 1999; Leermakers et al., 2001; Orecchio and Polizzotto, 2013). Total Hg concentrations in the studied sediments were found to increase from the north east coast to the south east coast of India (Figure 3). The lowest concentration of Hg (in the coastal sediments) was found on the northern part of Andhra Pradesh (e.g., Bheemili, Visakhapatnam). The average concentrations of Hg in the coastal sediments gradually increased (exceeded $50 \mu\text{g.kg}^{-1}$ of the sediments) in the southern part of the Andhra Pradesh (Figure 3).

A significant correlation coefficient was obtained between the total Hg content ($[\text{Hg}]_{\text{T}}$) and the total organic carbon (TOC) in the coastal sediments ($R^2=0.69$, $p \gg 0.001$). A similar significant correlation coefficient was also found between the $[\text{Hg}]_{\text{T}}$ and finer particles in the studied sediments ($R^2=0.49$, $p \gg 0.01$). This statistical analysis indicates that accumulations of Hg in the coastal sediments were probably dependent on TOC and texture of the sediments (Figures 4a and b). A strong correlation coefficient was also obtained between the sediment grain size and TOC content of the studied sediments ($R^2=0.71$, $p \gg 0.01$). The fine-grained sediments were found to have high organic carbon content and effective in scavenging Hg from the overlying water column.

According to the Canadian sediment quality guidelines for the protection of aquatic life, the interim sediment quality (ISQGs) and probable effect levels (PELs) for Hg in sediment are 130 and $700 \mu\text{g.kg}^{-1}$ respectively. The United States environmental protection agency has also suggested that effects range low (ERL) and effects range median (ERM) values for Hg in coastal sediment are 150 and $750 \mu\text{g kg}^{-1}$. Table 3 shows the average concentrations of Hg reported in the coastal sediments from the different parts of the world compared with the concentrations of Hg found in the studied sediments (from the central east coast of India). The concentrations of Hg were found to be much lower than the concentrations recommended by the United State Environmental Protection Agency and the Canadian Council of Ministers of the Environment for the protection of aquatic life.

This study suggest that the high production of atmospheric Hg (India is the second highest producer of atmospheric Hg in the world) had not elevated the concentrations of Hg in the coastal sediments of central east coast of India. The low concentrations of Hg in the coastal sediments probably indicate that high atmospheric emission of Hg from India has less impact on the total Hg content in the studied sediment.

It is well known that the total concentrations of Hg in the coastal sediments are inadequate to provide better understanding of its speciation, bioavailability, and toxicity. Further study was carried out to understand Hg-speciation in the coastal sediments by using a modified BCR sequential extraction protocol (Sahuquillo et al., 2003).

Figure 5 suggests that Hg had different affinities for the different solid-phases of the studied sediments. The concentrations of water soluble, exchangeable and carbonate forms of Hg complexes (Fr. 1) were found to be in range of 1.5 to 20% of the total Hg content ($[Hg]_T$) in the studied sediments (Table 4). This fraction of Hg-complexes (Fr.1) is expected to leach out easily from sediments and can increase the mobility and bioavailability of Hg in the overlying water column. Figure 5a represents the variation of Fraction 1 in the coastal sediments of the studied areas.

The presence of low concentrations of Hg as water soluble, exchangeable and carbonate complexes in the studied sediments (Fig. 5a) indicate that enough binding sites were present in the sediments to binds Hg. The concentrations of Hg associated with iron and manganese oxide phases (Fr. 2) were found to be in the range of 2.0–25.0% of the total Hg content in the sediments (Figure 5b). It is well known that Hg can easily associate with Fe/Mn oxide phases in sediments. Kim et al. (2004a) have reported that presence of chloride and sulphate can decrease or increase the sorption of Hg(II) on goethites (a-FeOOH), g-alumina(g-Al₂O₃),and bayerite(b-Al[OH]₃), which are useful surrogates for the natural sediments. Hg(II) sorbs strongly as a bidentate corner-sharing surface complex to the Fe(O,OH)₆ octahedra of the goethite structure and as a monodebtate, corner-sharing bidentate, and edge-sharing bidentate complexes to the Al(O,OH)₆ octahedra that compose the bayerite structure.

It was found that ~10–63% of the total Hg was complexed with organic matter present in the sediments (Table 4). Figure 5c shows that the concentration of Hg associated with total organic carbon (TOC) gradually increased with the increasing TOC content in the coastal sediments. However, maximum concentrations of Hg were found to associates with residual part of the sediments (Fr. 4) (Figure 5d). The highest concentrations of Hg in the residual fractions indicates that major part of the total Hg were not bioavailable and were present within the structure of the sediments. The data obtained from sequential extraction are presented in Table 4.

Organic matter in the surface sediments was found to play a key role in controlling Hg speciation in the sediments under oxic condition. Strong positive correlation between the total Hg content and the Hg associated with organic phases in the sediments (Figure 6) indicates that enough uncomplexed Hg binding sites were available in the organic phases of the coastal sediments. This finding encourages us to estimate the uncomplexed Hg-binding ligand concentrations (in future) in the coastal sediments from the central east coast of India.

Figure 7 shows the impact of varying Hg/TOC ratio on the distribution of Hg into the different phases within the sediments. Figure 7a) shows that the increasing $[\text{Hg}]_{\text{T}}/\text{TOC}$ ratio did not increase the concentrations of Hg present in Fr 1 (exchangeable, water soluble and carbonate forms of Hg) within the sediments. This probably indicates that there were sufficient binding sites available in the coastal sediments to form thermodynamically more stable Hg complexes. This study indicates that increasing $[\text{Hg}]_{\text{T}}/\text{TOC}$ ratio could not occupy/saturates all the strong binding sites (probably in the organic phases) present in the coastal sediments.

Figure 7 b) and c) show that the variation in concentrations of Hg associated with Fe/Mn oxide and organic phases in the studied sediments. In both cases positive correlations were obtained. These figures indicate that increasing $[\text{Hg}]_{\text{T}}/\text{TOC}$ ratio force Hg to get distributed with the strong binding sites (to form thermodynamically stable complexes) in the Fe/Mn oxide and organic phases. Fig 7 d) shows the changes in concentrations of Hg in residual fractions with varying concentration $[\text{Hg}]_{\text{T}}/\text{TOC}$ ratio in the sediments. It was found that the residual fraction gradually increased with the increasing $[\text{Hg}]_{\text{T}}/\text{TOC}$. The increasing concentrations of Hg in the residual fractions of the sediments with the increase in total Hg loading in the sediments probably suggest that more strong binding sites were available in the coastal sediments to form stable Hg-sediment complexes.

The concentrations of Hg in the coastal sediments were found to be much lower than the Hg concentrations recommended in the coastal sediments by the United State Environmental Protection Agency and the Canadian Council of Ministers of the Environment for the protection of aquatic life. It is suggested that Hg-sediment interactions are influenced by the size characteristics of coastal sediment particles, and TOC content of the coastal sediment. The sequential extraction study suggests that total organic carbon and residual phase may play an important role in Hg distribution in coastal sediments. This study suggests that the there were uncomplexed strong Hg-binding sites were available in the sediments (from the central east coast of India). It is expected that the low Hg content, high salinity on the overlying water column, high pH and oxic condition on the surface

sediments in the studied area probably decrease the possibility of methylmercury (MeHg⁺) production.

However, further investigation will be performed to map Hg and MeHg⁺ distribution in the coastal sediments around India to provide the relationship (if any) between atmospheric Hg production and accumulation and production of MeHg⁺ in coastal sediments. An effort will be made to understand the Hg-binding capacity and the uncomplexed Hg-binding ligand concentrations in coastal sediments from the central east coast of India.

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CAPTION AND LEGENDS FOR FIGURES

Figure 1: Different environmentally significant sampling sites off the central east coast of India

Figure 2: The schematic diagram of the modified BCR protocol for Hg fractionation study in coastal sediments

Figure 3: The average total Hg content in the coastal sediments from the central east coast of India

Figure 4: Variation of total Hg in the studied sediments with varying (a) silt and clay content and (b) TOC in the sediments

Figure 5: Fractionation of Hg, associated with different solid-phases of the studied sediments (by modified BCR method) (a) Fraction 1 (b) Fraction 2 (c) Fraction 3 and (d) Fraction 4.

Figure 6: Variation of Hg associated organic carbon in the studied sediments with varying TOC in the sediments

Figure 7: The impact of Hg loading (i.e. varying Hg/TOC ratio) on the distribution of Hg into the different phases within the sediments; a) Fraction 1, (b) Fraction 2, (c) Fraction 3 and (d) Fraction 4

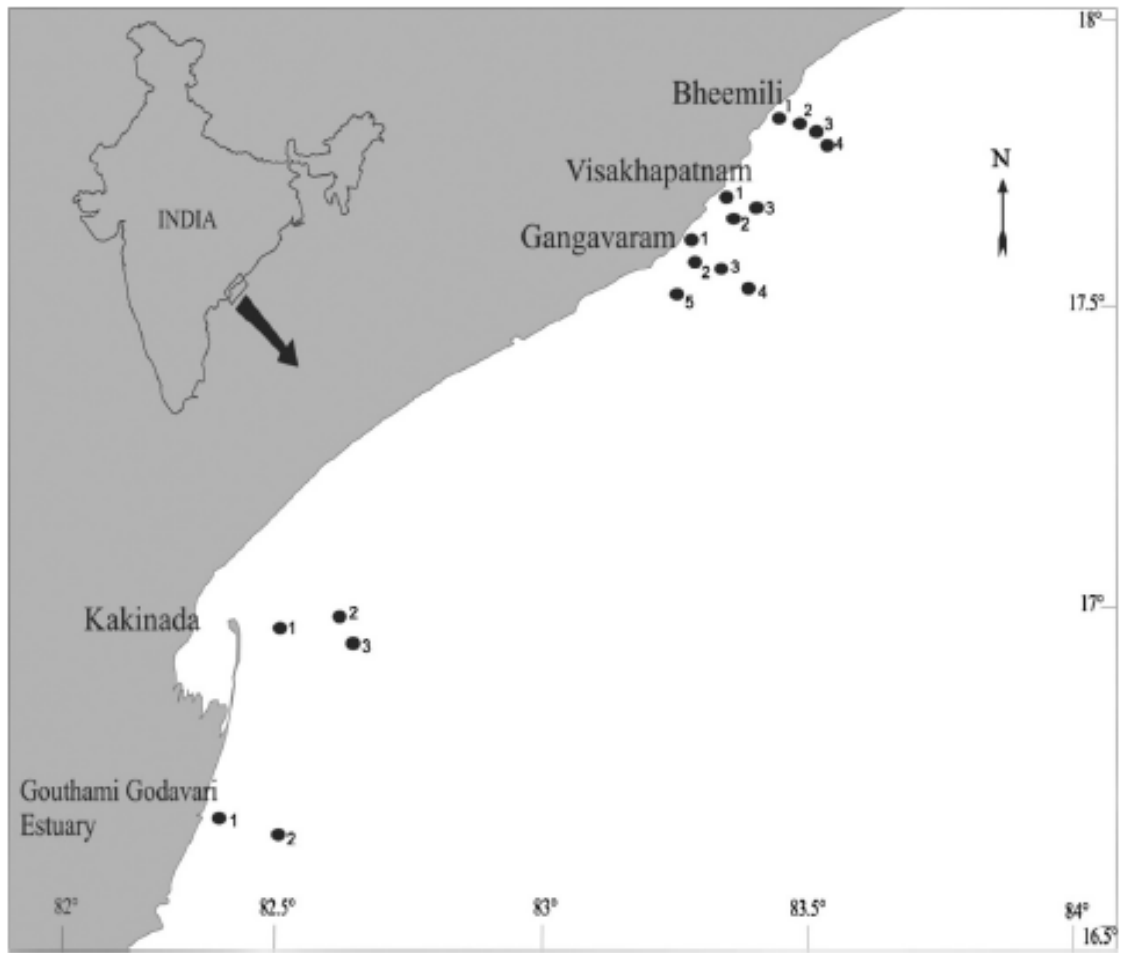


Figure 1

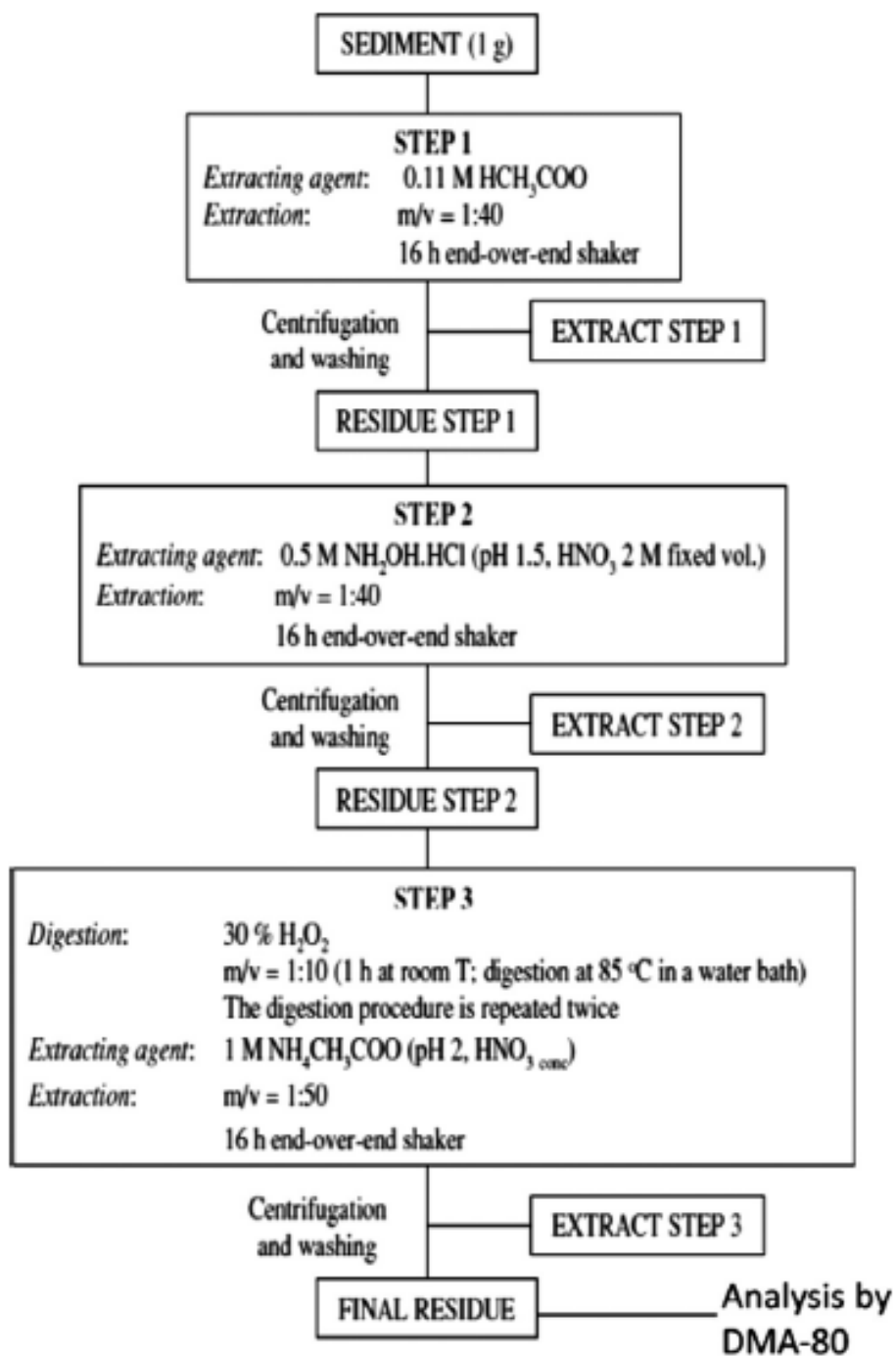


Figure 2

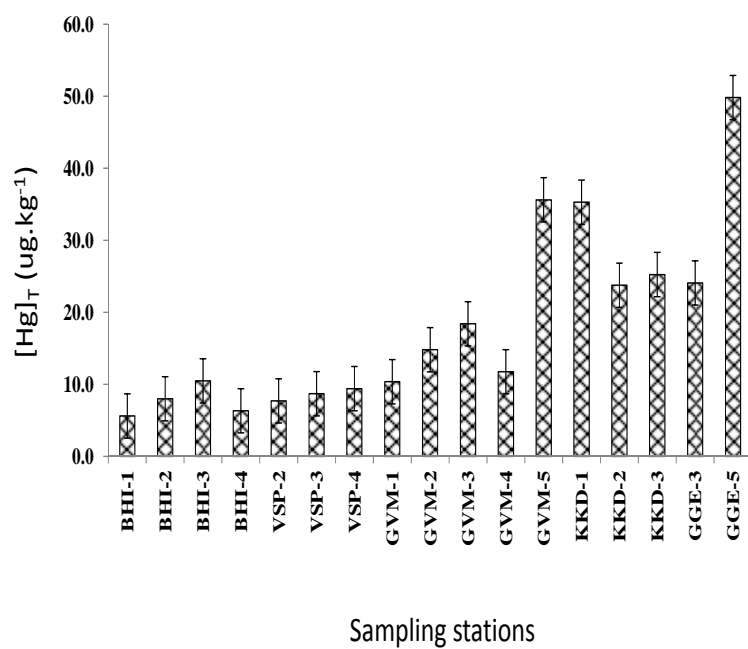


Figure 3

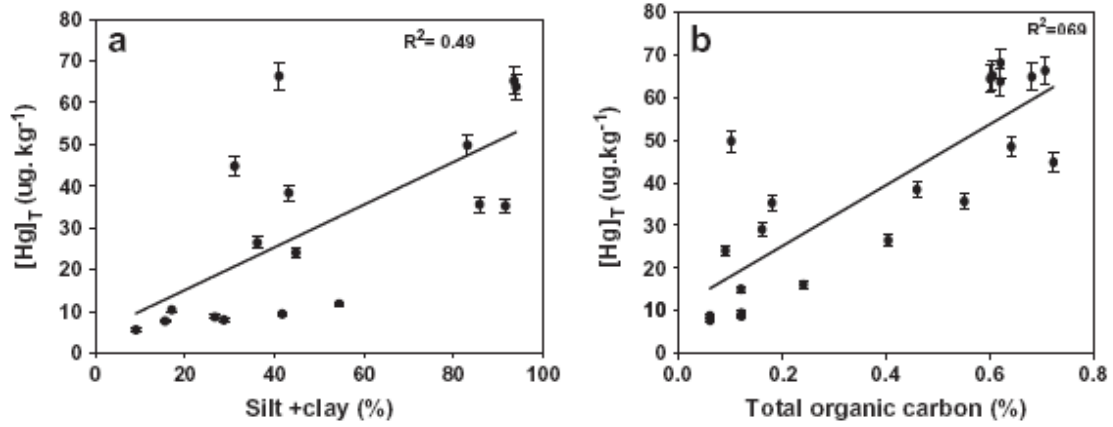


Figure 4

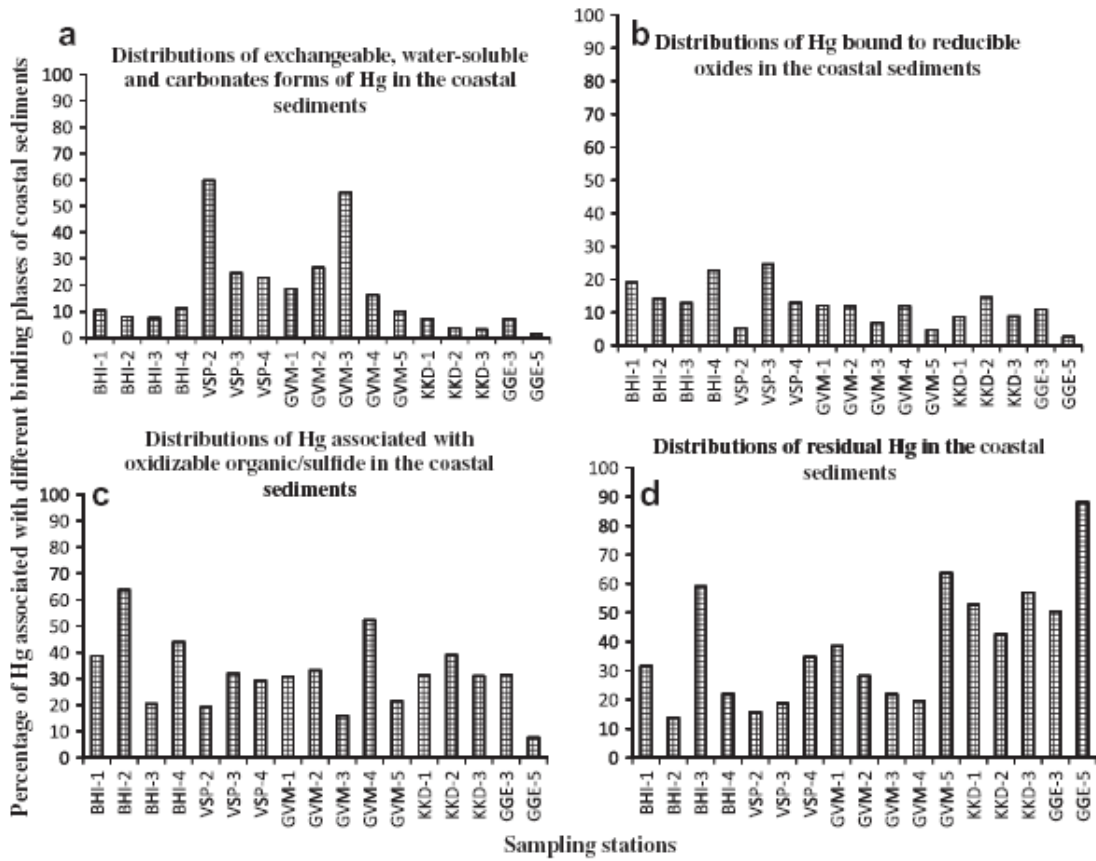


Figure 5

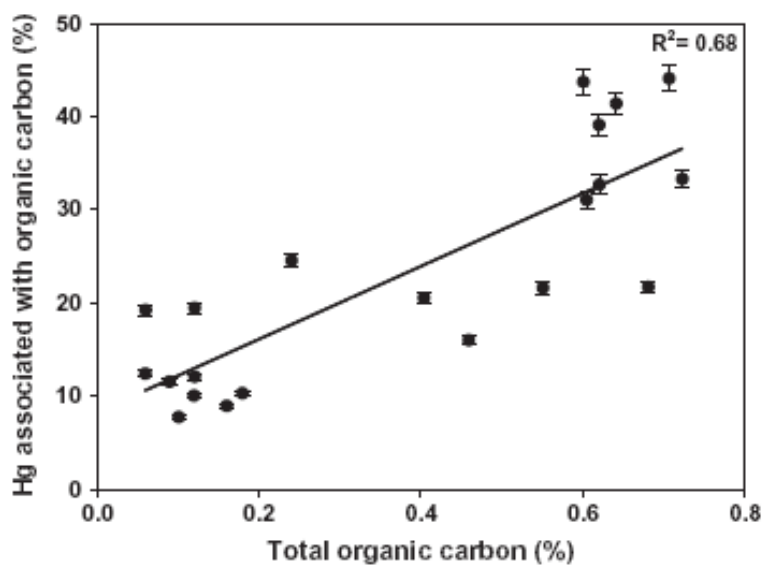


Figure 6

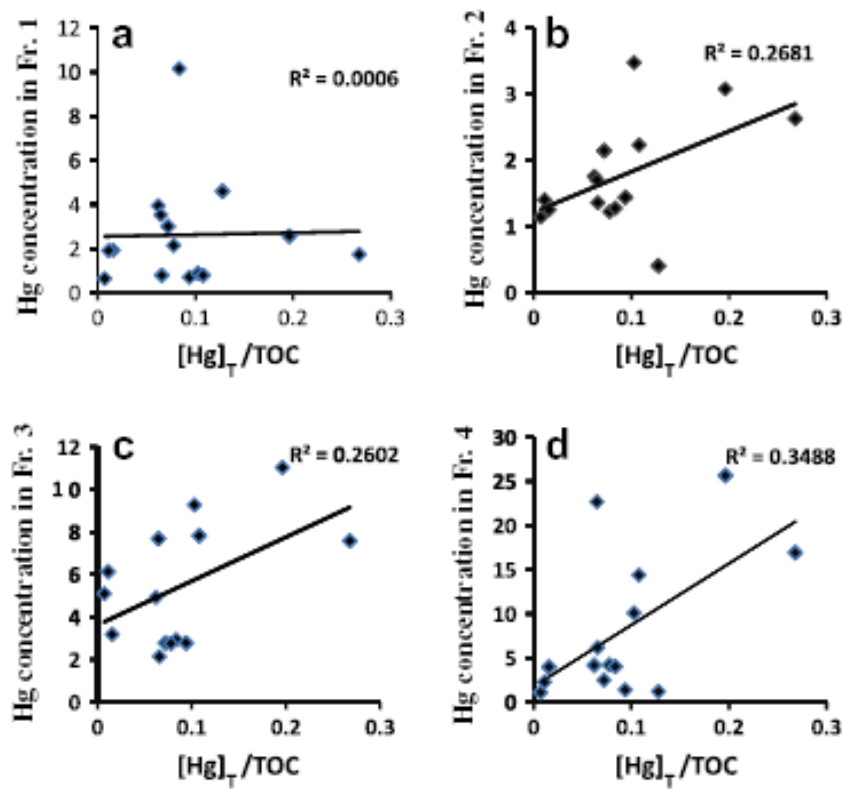


Figure 7

Table 1: Geographical locations of sampling sites

Station	Station Code	Depth (m)	Distance from Coast (Km)	Latitude	Longitude
Bheemili	BHM 1	6	0.5	17° 53.41'N	83° 27.74'E
	BHM 2	9	1	17° 53.61'N	83° 26.08'E
	BHM 3	16	3	17° 53.03'N	83° 29.21'E
	BHM 4	19	5	17° 52.42'N	83° 30.04'E
Visakhapatnam	VSP 1	12	1	17° 42.01'N	83° 14.26'E
	VSP 2	25	3	17° 41.27'N	83° 20.18'E
	VSP 3	35	5	17° 38.94'N	83° 19.11'E
Gangavaram	GVM 1	20	0.5	17° 36.83'N	83° 14.84'E
	GVM 2	23	1	17° 36.36'N	83° 15.01'E
	GVM 3	35	3	17° 35.89'N	83° 15.73'E
	GVM 4	44	5	17° 35.19'N	83° 16.67'E
	GVM 5	50	10	17° 33.94'N	83° 13.36'E
Kakinada	KKD 1	14	0.5	16° 41.75'N	82° 23.18'E
	KKD 2	21	1	16° 58.84'N	82° 23.64'E
	KKD 3	24	3	16° 58.94'N	82° 24.19'E
Gowthami Godavari Estuary	GGE 1	7	3	16° 41.50'N	82° 21.80'E
	GGE 2	36	10	16° 41.35'N	82° 25.23'E

Table 2: Texture analysis of the coastal sediments (%) from the central coast of India and the total Hg ($\mu\text{g kg}^{-1}$) concentration determined by DMA-80

Station	Sand(%)	Silt (%)	Clay (%)	Silt+clay	Hg_T
BHI1	90.9	2.7	6.4	9.1	5.6±0.5
BHI2	71.3	4.0	24.8	28.8	8.0±0.1
BHI3	63.8	15.8	20.4	36.2	26.5±0.8
BHI4	58.9	18.2	22.9	41.1	66.3±1.2
VSP2	84.4	4.8	10.8	15.6	7.7±0.3
VSP3	73.3	3.8	23.0	26.8	8.7±1.1
VSP4	58.2	39.3	2.5	41.8	9.4±0.5
GVM1	82.9	1.5	15.6	17.1	10. ±0.7
GVM2	68.9	8.3	22.8	31.1	44.8±3.5
GVM3	56.8	18.1	25.1	43.2	38.4±1.5
GVM4	45.5	24.3	30.2	54.5	11.8±0.5
GVM5	14.2	50.4	35.4	85.8	35.6±3.5
KKD1	8.4	85.2	6.4	91.6	35.3±0.5
KKD2	6.0	65.5	28.5	94.0	63.8±2.5
KKD3	6.5	85.0	8.5	93.5	65.2±1.8
GGE3	55.2	43.6	1.2	44.8	24.1±2.2
GGE5	12.4	83.1	4.5	87.6	49.8±1.8

Table 3: The average concentrations of Hg reported in the coastal sediments from the different parts of the world compared to the concentrations of Hg in the studied sediments (from the central east coast of India)

Area	Location	THg.Conc. ($\mu\text{g}/\text{kg}$)	References
China	Inner Shelf	26.5-47.6	Fang & Chen 2010
China	Middle Shelf	4.1-13.9	Fang & Chen 2010
	Gulf of Trieste, North		
Gulf of Trieste	Adriatic Sea	100.0-23000.30	Covelli et al 2001
Lebanese Coast	Akkar	10- 40	Ghanem et al 2011
Lebanese Coast	Dora	100-650	Ghanem et al 2011
Lebanese Coast	Selaata	20-60	Ghanem et al 2011
Belgian Coastal zone	Belgian Coastal zone	4-703	Leermakers et al 2001
Florida,USA	Apalachicola Bay	22-61	Apeti et al2012
Florida,USA	Choctawhatchee Bay	31-88	Apeti et al2012
Florida,USA	Cedar Key	17	Apeti et al2012
Florida,USA	Everglades	22	Apeti et al2012
Florida,USA	Florida Bay	18-25	Apeti et al2012
Florida,USA	Napes Bay	19	Apeti et al2012
Florida,USA	Pensacola Bay	53	Apeti et al2012
Florida,USA	Tampa Bay	12-187	Apeti et al2012
Louisiana, USA	Atchafalaya Bay	33	Apeti et al2012
Louisiana, USA	Barataria Bay	48-54	Apeti et al2012
Louisiana, USA	Joseph Harbor Bayou	61	Apeti et al2012
Louisiana, USA	Vermilion Bay	36	Apeti et al2012
Texas,USA	Aransas Bay	15	Apeti et al2012
Texas,USA	Copano Bay	24	Apeti et al2012
Texas,USA	Corpus Christi	35	Apeti et al2012
Texas,USA	Galveston Bay	18-109	Apeti et al2012
Texas,USA	Lower Laguna Madre	10-30	Apeti et al2012
Texas,USA	Mesquite Bay	17	Apeti et al2012
Texas,USA	Matagorda bay	10-85	Apeti et al2012
Texas,USA	San Antonio Bay	11-12	Apeti et al2012
Southern Baltic region	Gdansk Bay	6-422	Beldowski & Pempkowiak 2007
Italy, South East Sicily	Augusta Bay	0.04-21.4	Orecchio & Polizzotto2013
Central east coast of India	Bheemili	5.6-66.3	This study
Central east coast of India	Visakhapatnam	7.7-9.4	This study
Central east coast of India	Kakinada	35.3-65.2	This study
Central east coast of India	Gangavaram	10.4-38.4	This study
	Goutami Godavari		
Central east coast of India	estuary	24.1-49.8	This study

Table 4: Sequentially extractable non-residual and residual Hg species in the coastal sediments from the central east coast of India.

Stations	Fr 1 (%)	Fr 2 (%)	Fr 3 (%)	Fr 4 (%)
BHI-2	8.0±0.4	14.3±0.3	63.9±4.5	13.8±0.8
BHI-3	7.5±0.5	13.0±1.1	20.5±1.5	59.0±1.0
BHI-4	11.1±0.5	22.8±0.5	44.1±2.8	22.1±1.1
VSP-1	59.7±2.6	5.3±0.2	19.2±1.5	15.8±0.9
VSP-2	34.6±1.5	24.7±0.8	32.1±3.1	28.7±2.5
VSP-3	22.8±2.2	13.0±0.7	29.4±4.9	44.8±3.8
GVM-1	18.6±1.5	12.1±0.5	30.8±3.7	38.5±3.2
GVM-2	26.6±0.6	11.9±0.2	33.3±2.5	28.2±2.8
GVM-3	55.2±0.5	6.9±0.1	16.0±1.6	21.9±1.5
GVM-4	16.2±1.8	11.9±0.5	52.3±4.5	19.5±2.3
GVM-5	9.9±1.6	4.8±0.1	21.6±2.4	63.7±6.5
KKD-1	7.3±0.9	8.7±0.5	31.3±0.3	72.7±4.5
KKD-2	3.7±0.5	14.6±0.3	39.1±0.5	42.6±1.5
KKD-3	3.1±0.5	8.8±0.2	31.0±2.1	57.0±3.5
GGE-1	7.2±0.9	10.9±0.3	31.5±0.5	70.4±3.2
GGE-2	1.5±0.3	2.7±0.1	7.7±1.5	88.1±4.7

The quantity of sediment sample collected from BHI-1 stations was not enough to perform sequential extraction study.