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Baseline

Spatial budgetary evaluation of organochlorine contaminants in the sediments of Cochin Estuary, India

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ABSTRACT

This paper presents the first detailed investigation on the residual levels of organochlorine insecticide (OCI) concentrations in the Cochin estuarine sediment. It aims in elucidate their distribution and ecological impact on the aquatic system. Concentrations of persistent organochlorine compound (OC) were determined for 17 surface sediment samples which were collected from specific sites of Cochin Estuarine System (CES) over a period of November 2009 and November 2011. The contaminant levels in the CES were compared with other worldwide ecosystems. The sites bearing high concentration of organochlorine compounds are well associated with the complexities and low energy environment. Evaluation of ecotoxicological factors suggests that adverse biological effects are expected in certain areas of CES.

Pesticides are synthetic organic chemicals used to control unwanted or harmful pests, such as insects and mites that feed on crops. The environmental pollution by organochlorine insecticides are one of the most important Persistent Organic Pollutants (POPs) and have been of great concern around the world owing to their chronic toxicity, persistence and bioaccumulation. They have been extensively studied over the last 30 years, because of their resistance to degradation which has resulted in its being an almost universal contaminant of the environment (Jones and Voogt, 1999; Laabs et al., 2002; Monirith et al., 2003; Mudiam et al., 2012; Akhil and Sujatha, 2012). Due to the careless disposal practices, they have become a major pollutant in many parts of the world. In aquatic environments, OCs get removed from the water column and adsorb onto the particulate matter, and finally deposit on to sediments. The persistent nature of these compounds in soil and water, can adversely affect the health of pedospheric (Kammenga et al., 2000) and aquatic biocoenoses and the quality of drinking water (Kumar et al., 1995; Akhil and Sujatha, 2012). A greater tendency shown by OCIs for bioaccumulation and biomagnification in the food chains is due to their resistance to chemical and biological decay (Badawy and Wahaab, 1997; Yamashita et al., 2000; Abbassy et al., 2003). Although the applications of OCIs have been banned in many developed countries, some developing countries are still producing and using these persistent pesticides because of their low cost and versatility in controlling various insects (Tanabe et al., 1994; Monirith et al., 2003).

The main objective of the study is to survey the contaminant levels, distributions and other sources of these OCIs in surface sediments from CES and to assess their environmental impact in the ecosystem. Most of the earlier research contributions were based on one-time or seasonal sampling during a year, from the areas known for environmental pollution (Barakat, 2003; Khaled et al., 2004; Said et al., 2008). An approach based on the analysis of OCI residues in sediments collected over a considerable time period can provide a clue for a change in environment and such studies are limited. A few reports are available on the residue analysis of conservative pesticides in CES (Sujatha and Chacko, 1991; Sujatha et al., 1993, 1994, 1999) but no studies concerning these persistent contaminants in sediment were reported. A broad spectrum of pesticides is used in India for agriculture as well as vector control programs and hence the impact of residues of these OCs on Indian coastal environments is of considerable interest.

Cochin Estuary, one of the largest tropical estuaries of India, is facing gross pollution problems due to the release of untreated effluents from industries and domestic sectors. The major polluting industries in the region include fertilizer plant, oil refinery, rare earth processing plant, minerals and rutiles plant, zinc smelter plant, insecticide manufacturing unit and organic chemical plant. Reclamations over the past several decades have resulted in considerable shrinkage (40%) of the Cochin Estuary (Gopalan et al., 1983). Further, the construction of hydraulic barriers on the northern and southern limbs of the estuary to prevent saline intrusion into the upstream agricultural fields has imposed severe flow restrictions and increased sedimentation rate in the estuary (Menon et al., 2000). The development activities in and around Cochin Backwater System have added to the complexities and environmental dilemmas in this coastal niche. For a long period, there were no pollution control regulations and the untreated effluents continuously discharged into the backwaters.

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Study area is divided into three zones viz South, Middle, and North (Fig. 1). The south zone is situated in the fresh water region and it originates from southern bough of Moovattupuzha. Major source of pollution is from agriculture runoff and it is less affected by industrial effluents (Station No 1–6). The middle zone is well regulated by a bund (namely Thannirmukham), which was constructed in order to prevent the salt water intrusion into the paddy fields. The bund remains open during monsoon season. This zone has a perennial connection with the Arabian Sea and experiences an irregular encroachment of saline water intrusion there by making cradle grounds for diverse types of flora and fauna. With the advent of ICTT project, this area has become a backbone for the economy of State of Kerala. Due to enhanced containerization, resulting in improved trade and economic growth, widespread activities like dredging, piling, along with anthropogenic inputs are increasing frequently (Station No 7–11). Finally north zone originates from the industrial locale of Periyar – the life line of Kerala. Large scale industries on the river bank discharge effluents directly into these waterways resulting in the accumulation of varying amounts of nutrients in the Periyar River (Station No 12–17).

Surface sediments (top 0–5 cm) were collected from seventeen locations of CES over a period of November 2009 and November 2011. This was performed using a stainless steel grab sampler used repeatedly (three to five times) at each station, followed by thorough mixing of collected sediment on an aluminum tray in order to obtain a more representative sediment sample. All samples were then transferred into well labeled hexane-rinsed glass jars and kept it in the ice chest boxes on board and during transportation. The samples were then stored at −20 °C until the analysis.

About 5 gm of the sediment sample was accurately weighed and then extracted twice with 50 mL portions of 1:1 hexane-acetone mixture (HPLC grade, Glaxo, Mumbai, India). The combined extract was subjected to a cleanup procedure involving elution through a Florisil column (60 cm × 22 mm i.d) with 50 mL 1:1 hexane–acetone mixture. The extract was concentrated to about 5–6 mL by means of a rotary evaporator at 50–60 °C for further analysis. Separation and analysis of the OCIs were performed on a gas chromatograph (GC) (model 7890A, Agilent, Waldbronn, Germany) with a Ni-63 ECD and equipped with capillary column (HP-35, 30 m × 0.320 mm × 0.5 mm) using nitrogen as carrier gas (1.5 mL min^{-1}). The GC was calibrated with a standard solution of a pesticide mixture (Supelco, USA) prepared in HPLC grade n-hexane. Solvent blanks were used to confirm the absence of any pesticide residues. Analytical reproducibility was checked by replicate measurements. Identification and quantification of OCIs were accomplished by using reference solutions supplied by EPA (USA) and Supelco (USA). The following GC conditions were maintained: injection port temperature 250 °C, detector temperature 350 °C, oven temperature program: 110 °C (5 min) at 5 °C min^{-1} to 190 °C (2 min) at 15°C min^{-1} to 280 °C (10 min). 1 μL of aliquot samples were injected onto the column. All data were subjected to strict quality control procedures, including the analysis of procedural blanks and spiked samples with each set of samples analyzed. None of the target compounds were detected in the procedural blanks for sediment samples. Spiked samples (10 ng of pesticide mix standard) were determined with good precision and high recoveries. Limit of detection (LOD) and relative standard deviation (RSD) of the analytical method for OCIs is as follows. The detection limit was lowest for aldrin (0.04 ng/g) and highest for endrin and 4,4′-DDT (0.19 ng/g), while the detection limit of other analytes lies within the range. The average recoveries (n = 3) for OCIs revealed an efficiency of 87–103%. The relative standard deviations (RSD) were below 5.0% and fall within the requirement criteria of US-EPA (Recovery: 70–130%, RSD is <30%). The following organochlorine

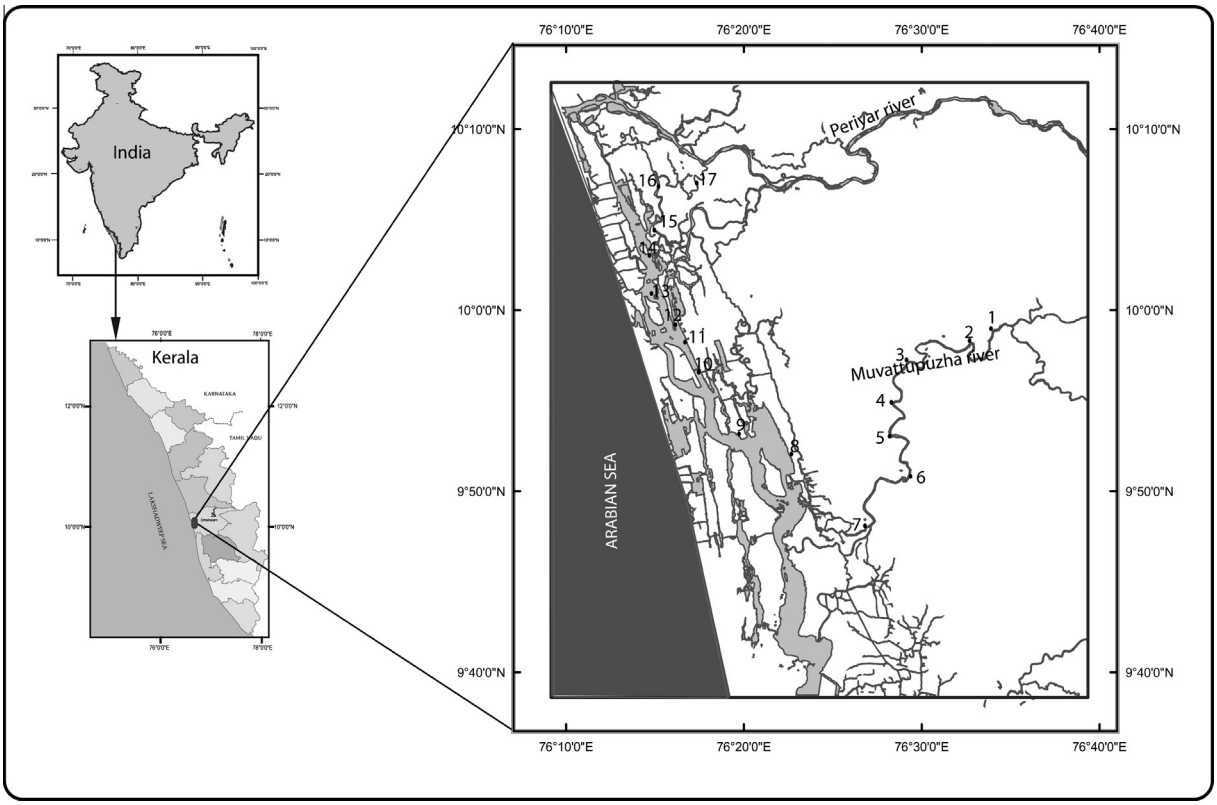

Fig. 1. Map of the study area and sampling sites.

pesticides were quantified. α-hexachlorocyclohexane (α-HCH), β-HCH, γ-HCH, heptachlor, aldrin, heptachlor epoxide (B), 4,4′-DDE, dieldrin, endrin, 2,4′-DDD, 4,4′-DDD, 2,4′-DDT, 4,4′-DDT, α-endosulfan, and β-endosulfan.

Data on the residual levels of pesticide accumulation in the marine sediments is limited, especially in the coastal belt of India. Coastal sediments serve both as temporary and long-term sink for many classes of anthropogenic contaminants and consequently act as the source of these xenobiotics into the aquatic realm. The applied and direct dumping of industrial wastes of these persistent contaminants can be transported through surface run-off, leaching ultimately accumulating and getting settled into the bottom sediments. Hence these bottom sediments represent an integrated conspicuous level of particle bound contaminants that have deposited over a longer period of time. A few studies have been reported on these contamination of OCs in the sediments from Indian coastal regions (Sarkar and SenGupta, 1987, 1988, 1991; Takeoka et al., 1991; Sarkar et al., 1997; Pandit et al., 2001) indicating the presence of their major emission sources in these regions. The findings of this research work constitute the first judicious base line data set for the OCI residues in the sediments of CES (Tables 1 and 2). Across the world, HCH is the most extensively used insecticide, particularly in Asia due to their large scale production in China and India. Being as an inexpensive industrial product, it has been commercially available for decades for varying purposes in these regions. Previously, Mathur (1993), reported that, around 25,000 MT (metric tons) of chlorinated pesticides were used annually in India and DDT accounted for 40% of this group. Among the HCH isomers, α-HCH is more likely to diffuse into the air and get transported for a long distance, while β-HCH is more resistant to hydrolysis and environmental degradation, and is the predominant isomer in the water, fluids, animal tissues and sediments (Sujatha et al., 1993; Walker et al., 1999; Lee et al., 2001). In the present research work, total HCH concentration of 423 ng/g was detected in the entire study area during 2009. Levels decreased to 23.7 ng/g in 2011. Concentrations and compositions of HCH isomers were found to be in the order β-HCH > γ-HCH > α-HCH in 2009, which implies that HCH contamination in the sediments of CES might have originated from a relatively remote source or long-term accumulation from prolonged usage or discharge from industries. In 2011, residual levels of these isomers was in the order γ-HCH > α-HCH > β-HCH showing the recent input of these isomers in the study area (Fig. 2). Levels of ∑HCH in sediments from CES were found generally higher than those of ∑DDT during 2009. However, the ∑HCH concentrations in sediments collected in 2011 is lower than that of ∑DDT, while the levels of ∑Cyclodienes were still higher in both the sampling periods, 2009 and 2011. These results reflect the fact that the contamination sources and environmental fate of degradation in these HCH's and cyclodienes are different, this clearly depict that ∑Cyclodienes followed by ∑HCH and ∑DDT compounds were the predominant OCIs in the sediments of CES. In 2009, the residue levels of endosulfan (α) ranged from BDL to 43.2 ng/g. Contamination level of endosulfan (α) was highest at Station No:16 (350.5 ng/g) during 2011. The elevated concentration of α-isomer may be due to the recent input of these pesticides in the study area.

Aldrin, the probable carcinogen is mainly used as an insecticide for the underground control of termites. The residue level of aldrin was BDL to 33.2 ng/g in 30% of the samples collected in 2009 and is quickly transferred to a more toxic persistent dieldrin, through microbial oxidation or epoxidative degradation. The abundance of aldrin relative to dieldrin implies that it is widely used in this region during the year 2009. Dieldrin concentration was high (BDL to 142.4 ng/g), with maximum value at Station No:13 in 2011. The result shows that instead of restriction in usage, excessive and indiscriminative adoptions of these are still continuing due to the low cost and popularity of the insecticide formulations among the local people. Among the chlordane compounds, heptachlor epoxide is relatively abundant with the highest contamination level (69.4 ng/g) at Station No:8 during 2009 and BDL during 2011 in the entire study region.

The relative concentrations of the parent compound and its metabolite can provide useful information on the pollution source. DDT can be biodegraded by microorganisms to DDE and DDD under aerobic and anaerobic conditions. Therefore, ratios of (DDE + DDD)/∑DDT and DDE/DDD can be used as indicative indices for assessing the long term weathering and biotransformation of DDT under various redox conditions (Zhang et al., 1999; Doong et al., 2002; Yang et al., 2005). Mostly DDE/DDD ratio were in the average value >0.5 which indicates that the collected samples were mainly under anaerobic conditions. The observed pattern of DDT and its metabolites in most sediment samples were in the order of DDD > DDE > DDT. The ratio of (DDE + DDD)/∑DDT > 1 in majority of the stations infers that the contaminant levels of DDT's in the sediments of CES may have been derived from the aged and weathered agricultural soil and retained under anaerobic conditions within the sediment phase. The dominance of DDD's over DDE's clearly supports the reductive dechlorination of DDT's to DDD

Table 1
Concentrations (ng/g, dry wt.) of selected chlorinated pesticides in the sediments of Cochin Estuary, November 2009.

Station	Heptachlor (ng/g)	Heptachlor epoxide (B) (ng/g)	4,4′-DDE (ng/g)	2,4′-DDD (ng/g)	4,4′-DDD (ng/g)	Aldrin (ng/g)	Endrin (ng/g)	α-Endosulfan (ng/g)
1	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
2	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
3	63.6	BDL	BDL	BDL	BDL	11.8	BDL	BDL
4	BDL	BDL	12.2	11.9	BDL	BDL	BDL	36.8
5	BDL	BDL	BDL	BDL	BDL	21.1	BDL	BDL
6	BDL	BDL	BDL	BDL	33.1	BDL	BDL	BDL
7	BDL	BDL	16	41	BDL	BDL	BDL	BDL
8	BDL	69.4	BDL	BDL	BDL	BDL	BDL	BDL
9	43.6	BDL	BDL	BDL	BDL	33.2	BDL	BDL
10	BDL	BDL	6	BDL	BDL	BDL	BDL	BDL
11	2.7	BDL	BDL	BDL	BDL	BDL	BDL	BDL
12	BDL	22.2	21.5	BDL	BDL	BDL	24.1	BDL
13	BDL	BDL	BDL	BDL	34.7	BDL	BDL	43.2
14	BDL	BDL	BDL	BDL	BDL	BDL	103.1	BDL
15	BDL	BDL	4.5	12.2	BDL	7.2	BDL	BDL
16	BDL	BDL	BDL	BDL	BDL	BDL	30.4	BDL
17	BDL	BDL	BDL	BDL	35.9	6.1	BDL	BDL

Notes: BDL, below detection limits.

Table 2
Concentrations (ng/g, dry wt.) of selected chlorinated pesticides in the sediments of Cochin Estuary, November 2011.

Station	Heptachlor epoxide (B) (ng/g)	4,4′-DDT (ng/g)	4,4′-DDE (ng/g)	2,4′-DDD (ng/g)	4,4′-DDD (ng/g)	Aldrin (ng/g)	Dieldrin (ng/g)	Endrin (ng/g)	α-Endosulfan (ng/g)
1	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
2	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
3	BDL	BDL	BDL	BDL	33	BDL	BDL	BDL	BDL
4	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
5	BDL	BDL	BDL	BDL	33	BDL	BDL	BDL	BDL
6	BDL	BDL	14	BDL	BDL	BDL	BDL	BDL	BDL
7	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
8	BDL	BDL	BDL	BDL	33	BDL	18.7	BDL	BDL
9	BDL	BDL	14.2	BDL	BDL	BDL	BDL	BDL	BDL
10	12.6	BDL	BDL	42.7	BDL	BDL	BDL	59.4	6
11	BDL	162.5	BDL	BDL	33.1	BDL	18.8	BDL	BDL
12	BDL	BDL	13.8	BDL	BDL	3.1	BDL	BDL	BDL
13	21.8	BDL	BDL	BDL	BDL	BDL	142.4	BDL	BDL
14	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
15	12	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
16	BDL	BDL	BDL	38.7	BDL	BDL	BDL	BDL	350.5
17	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL

Notes: BDL, below detection limits.

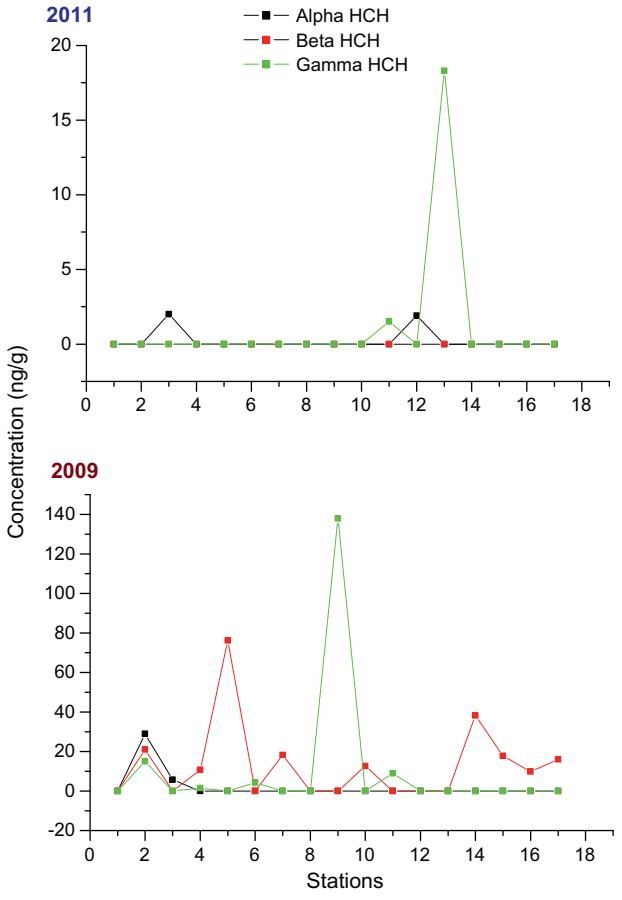

Fig. 2. The levels of HCH isomers in surface sediments of Cochin Estuary (2009–2011).

under anaerobic conditions and similar observations reported earlier (Barakat et al., 2002). The results of anoxic behavior of sediments from the study area was in good concurrence with the previous work of Akhil et al., 2013 and Manju et al., 2013.

The total ∑OCI contamination in sediment was 1170 ng/g (2009) and 1087 ng/g (2011) in the entire study region. Maximum concentration of ∑OCI were found in the northern part of the estuary (Fig. 3). Enrichment of organic carbon in association with the greater clay contents is attributed to the enhanced adsorption of organic carbon onto the clay minerals in the low salinity regimes. The weak flow and the huge input of industrial waste in the northern part of the estuary also results in high organic input in this area (Akhil et al., 2013). However the successive post depositional changes may also modify the environment niche drastically.

To determine the potential of organochlorine contaminants that cause adverse effects to biota, concentration ranges of selected analytes from the sampling location were compared to the sediment quality guidelines (SQG) sited in Table 3. Sediment quality guideline values from NOAA's National status and trends program (Long et al., 1995) designate an effects range low (ERL) and an effects range medium (ERM). ERL's represent chemical concentrations below which adverse effect would be rarely observed; ERM's represents the chemical concentration above which adverse effects would frequently occur. The Canadian sediment quality guideline (CCME, 1999), probable effect level (PEL) defines the level above

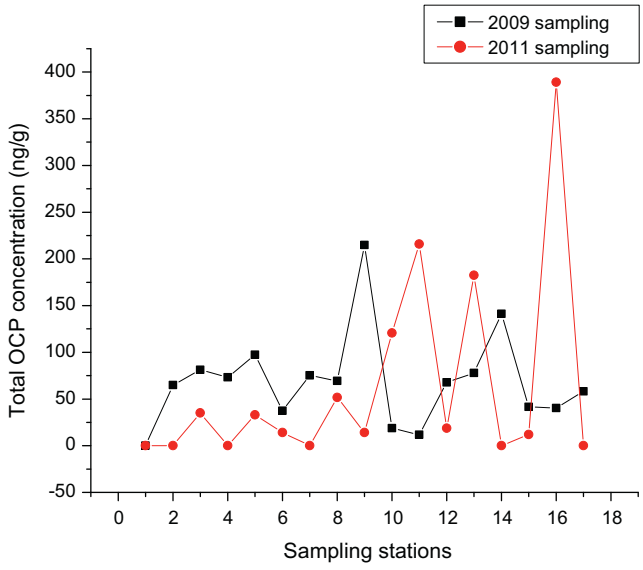

Fig. 3. Total OCP concentrations in surface sediments of Cochin Estuary (2009–2011).

Table 3
Comparison of OC concentrations in the study area (ng/g dry wt.) with toxicity guidelines.

Compound	SQGs (ng/g d.w.)			Study area	Stations (S)		
	ERL	ERM	PEL	2009–2011	S of ERL ~ ERM 2009–2011	S of >ERM 2009–2011	S of >PEL 2009–2011
∑DDTs	1.58	46.1	NA	229–418	3,4,5,6,8,9,10,12,13,15,16,17	7,11	
o,p′- and p,p′-DDD	NA	NA	8.51	168.8–213.5			3,5,6,8,11,13,17
o,p′- and p,p′-DDE	NA	NA	6.75	60.2–42			4,6,7,9,12
o,p′- and p,p′-DDT	NA	NA	4.77	BDL–162.5			11
p,p′-DDD	2	20	NA	103.7–132.1	None	3,5,6,8,11,17	
p,p′-DDE	2.2	27	NA	60.2–42	4,6,7,9,10,12,15	None	
p,p′-DDT	1	7	NA	BDL–162.5	None	11	
Endrin	0.02	45	62.4	157.6–59.4	12,16	10,14	14
Dieldrin	NA	NA	6.67	BDL–179.9			8,11,13
Lindane	NA	NA	1.38	167.8–19.8			2,4,6,9,11,13
Heptachlor epoxide	NA	NA	2.74	91.6–BDL			8,10,12,13,15
CHLs	0.5	6	8.87	201.5–46.4	11	3,9,10,13,15	3,9,10,13,15

Table 4
Comparison of organochlorine concentrations (ng/g dry wt.) in the sediments from other locations.

Area	Year	∑DDTs	∑HCHs	References
Eastern coast of India	NA	0.10–0.97	0.008–0.02	Sarkar (1994)
Rhone prodelta, France	1987–1991	62–675	NA	Tolosa et al. (1995)
Barcelona Offshore	1987–1991	4.4–76.2	NA	Tolosa et al. (1995)
Ebro Prodelta, Spain	1987–1991	0.8–89.2	NA	Tolosa et al. (1995)
Arabian Sea	1997	1.14–25.17	NA	Sarkar et al. (1997)
Ebro River, Spain	1995–1996	0.4–52	0.001–0.038	Fernàndez et al. (1999)
Manzala Lake	1993	5–640	0.97–16.1	Yamashita et al. (2000)
West and east coast of India	1995–1997	ND–364	1.5–1053	Pandit et al. (2001)
Alexandria harbour, Egypt	1998	<0.25–885	0.25–6.0	Barakat et al. (2002)
Mumbai	NA	0.5–9.6	3.8–16.2	Pandit et al. (2002)
Masan Bay, Korea	1997	0.27–89.2	NA	Hong et al. (2003)
Northeastern coast of India	2003	0.18–1.93	0.11–0.40	Guzzella et al. (2005)
Bahrain	2000	0.088–0.430	0.004–0.013	De Mora et al. (2005)
Bay of Bengal, India	1998	0.04–4.79	0.17–1.56	Babu Rajendran et al. (2005)
Gulf of Aden, Yemen	2005	ND–0.74	ND–0.06	Mostafa et al. (2007)
Estuarine and marine sediments, Vietnam	2003–2004	0.31–274	ND–1.00	Hong et al. (2008)
Remote lakes and coastal areas, Norwegian Arctic	2005	0.23–5.4	0.21–7.0	Jiao et al. (2009)
Baiyangdian Lake, North China	2007–2008	1.3–51.3	4.7–679	Hu et al. (2010)
Lower Mekong River Basin, Laos and Thailand	2005	0.027–52	0.005–9.9	Sudaryanto et al. (2011)
Lake Victoria, Uganda.	2003–2007	4.24 ± 3.83	2.80 ± 2.00	Wasswa et al. (2011)
Maryut Lake, Egypt	2005	0.07–105.6	ND–2.20	Barakat et al. (2012a,b)
Manzala Lake, Egypt	2005	ND–3.42	ND–7.25	Barakat et al. (2012a,b)
Cochin Estuary	2009–2011	229–418	423–23.7	This study

which adverse effects on aquatic biota are expected to occur frequently. Most of the pesticide residues detected from the study area were above SQG. The highest concentrations of OCs were detected at stations near the discharge point of sewage and close to the industrial areas. Based on earlier reported values, the study infers that sediment from CES are of higher toxicity than those from other worldwide locations (Table 4).

The research work allowed a comprehensive evaluation of OCIs content in the sediments of CES. Due to the excessive agriculture application, industrial pollutant discharge, atmospheric transport and its persistent nature, obviously there still exist a variety of OCI residues in the sediments of CES. Among these contaminants, cyclodienes followed by HCH and DDT compounds are the predominant OCIs in the study area. The high concentration of biological metabolite p,p′-DDD from the parent DDTs depict that OCIs contamination was mainly from aged and weathered agricultural soils and was retained under anaerobic conditions in the sediment. The results also indicate that there exists a certain potential health risk to the habitat in both the southern and northern area. The baseline data obtained from the present investigation can be used for regular ecological monitoring, considering the industrial and agricultural growth around this important estuarine system. Further work is needed to determine the bioaccumulation of POPs in the food web and the associated risks to the ecosystem and human health.

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