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Baseline

Organochlorine contamination (PCBs, DDTs, HCB, HCHs) in sediments from Cienfuegos bay, Cuba

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ABSTRACT

The spatial distribution of various organochlorinated compounds, e.g. PCBs, DDTs, HCB and HCHs, were investigated in sediments from Cienfuegos bay, Cuba. Their concentrations appeared to be relatively low by global standards and only sediments receiving the impact from the residual waters of the city and thermoelectrical power approached the sediment quality guidelines for PCBs and DDTs. Relatively higher ΣDDT concentrations and high DDT/DDE + DDD ratios in two sites near the outfalls of the city indicated a current DDT usage, probably linked to public health emergencies. These results contribute to the sparse regional database for organochlorinated compounds in the Caribbean marine environment.

Polychlorinated biphenyls (PCBs) and chlorinated pesticides (e.g., DDTs, HCB, HCHs, etc.), are an important group of persistent organic compounds (POPs) included in the Stockholm Convention (UNEP, 2008): they are globally distributed and their properties cause worldwide concern as toxic environmental contaminants (Gómez-Gutiérrez et al., 2007; Serrano et al., 2003; WHO, 1992) in a warming World (Noyes et al., 2009; Tanabe et al., 1994). Organochlorinated compounds have been widespread used, with both industrial and agricultural sources contributing significant amounts to the environment through leakage, disposal and evaporation. In the aquatic ecosystems, chlorinated compounds tend to accumulate in sediments and biota because of their hydrophobic character, low water solubility and persistence (Kidd et al., 2000; Paasivirta et al., 1999). As a result, sediments are usually regarded as the ultimate sink for many classes of anthropogenic contaminants to the environment, and are one of the best media for the long-term monitoring of many POPs (Jaffe, 1991; Lamon et al., 2009).

Nowadays, there is scarce information on the status of POPs in many Caribbean coastal areas. For Cuba, some old data is only available from the pesticide contamination in soils from the most agro-developed province of Cuba (Dierksmeier, 1996) and few data exist from the International Mussel Watch (Fernandez et al., 2007; Sericano et al., 1995). Cienfuegos bay, situated in the southern central part of Cuba is a semi-enclosed bay connected to the Caribbean Sea by a narrow channel of 3 km long (Fig. 1). This bay plays an important role in the industrial development of Cuba and posses the most important natural resources of the province, due to fishing activities and natural parks. The northern basin receives most of the anthropic impact from the outfall of Cienfuegos city, industrial pole in the country with a population of 150,000. The southern basin is subject to a smaller degree of anthropic pollution originating from the Caonao and Arimao rivers.

In consideration of the above, the present study measures polychlorinated biphenyls (PCBs) and organochlorine pesticides (DDTs, HCB, and lindane) in sediments to ascertain the contamination status in the Cienfuegos bay. In addition, a comparison of our results with those from other environments is included to evaluate the relative significance of the contamination.

Sediment samples were collected in March 2005 at 17 sites using a glass core (Fig. 1 and Table 1). Samples were wrapped in aluminum foil and cool transported to the laboratory where they were immediately stored in a freezer. Sediment matrices were freeze-dried, hand-sieved (250 μm) and homogenized. Approximately 8-g portion of each sediment was spiked with internal standards (PCBs 29 and 198 for the first fraction and ε-HCH for the second fraction), and extracted with a hexane: methylene chloride mixture (50/50, v/v) in a microwave at 1200 W and 115 °C for 20 min. Sulphur was removed with activated copper and two fractions were obtained from silica/alumina columns, as described in Tolosa et al. (2009). PCBs, HCB, *p*′,*p*′-DDE, aldrin, heptachlor and mirex were eluted with 20 ml hexane followed by 20 ml hexane/methylene chloride (90:10). *p*′,*p*′-DDD, *p*′,*p*′-DDT, and γ-HCH (lindane) were eluted in a second fraction with 40 ml hexane/methylene chloride (80:20).

The compounds were determined by HRGC with a ^{63}Ni electron capture detector (HRGC/ECD) on a Hewlett–Packard 6890HP equipped with a split/splitless injector, and an automatic liquid sampler (Hewlett–Packard, Palo Alto, USA). For all the analyses, a fused-silica capillary column HP-5 (30 m, 0.25 mm i.d. and film

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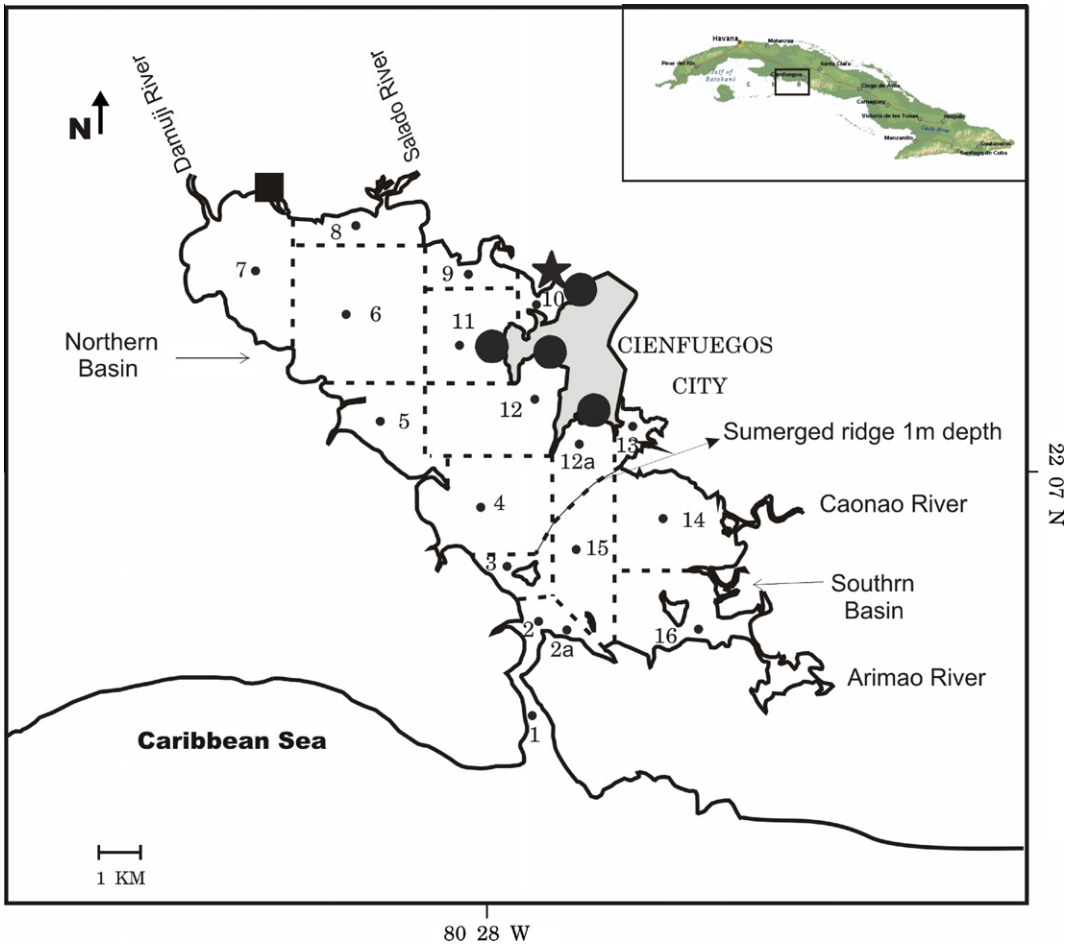

Fig. 1. Map showing the location of sediment samples in the Cienfuegos bay. In accordance with the grain size distribution and mineralogical composition, the surface sediments of Cienfuegos bay are divided in 16 sectors, which have been monitoring since 1989. The square symbol represents the location of the Cienfuegos refinery, the star is the thermoelectrical power plant and the circles are the urban waste outfalls.

Table 1
Sampling sites from the Cienfuegos bay and TOC values of the sediment samples.

Sites	Location	Latitude/longitude	Depth (m)	TOC (%)
SS-1	Entrance to the channel connecting the bay to the Caribbean Sea	22°04′03″N/80°27′35″W	31.7	3.02
SS-2	La Milpa	22°04′39″N/80°26′56″W	16	1.79
SS-2a	Caletón de Don Bruno	22°04′34″N/80°27′39″W	15.1	1.55
SS-4	Jucaral	22°06′47″N/80°28′21″W	11.6	1.91
SS-5	Calicito	22°07′44″N/80°29′31″W	12	2.86
SS-6	Center of the North basin	22°09′40″N/80°30′05″W	11.9	3.10
SS-7	Mouth of Damují river	22°09′55″N/80°31′33″W	3.7	2.74
SS-8	Mouth of Salado river	22°10′26″N/80°30′14″W	6.5	2.51
SS-9	Port, boya roja	22°09′54″N/80°28′36″W	5.1	2.83
SS-10	Termoeléctrica station power (outfall channel)	22°09′16″N/80°27′25″W	5.6	3.68
SS-11	Mielera, Boya de punta arena	22°08′42″N/80°28′29″W	9.9	2.77
SS-12	Los Pinitos	22°07′27″N/80°27′27″W	9.4	2.41
SS-12a	Entrance to the Cura lagoon	22°07′10″N/80°26′48″W	3.4	2.43
SS-13	Junco Sur, Punta La Cueva	22°07′19″N/80°25′52″W	5.7	1.77
SS-14	Mouth of Caonao river	22°05′26″N/80°26′13″W	6.9	2.26
SS-15	Center of the South basin	22°05′17″N/80°26′26″W	12.4	2.03
SS-16	South from Cayo Ocampo	22°04′30″N/80°24′49″W	2.7	2.20

thickness 0.25 μm) was used. GC parameters were: injector temperature 250 °C; detector temperature 300 °C; helium at a flow rate of 1 ml min^{-1} was used as carrier gas. The GC oven was temperature-programmed from 70 °C (2 min isothermal) to 260 C (at 3 °C min^{-1}) and held isothermally at 260 °C for 20 min. In addition, qualitative determination of organochlorine residues was confirmed by negative ion chemical ionisation (NICI) GC–MS in a HP-5890 Engine B. The ion source and mass analyser were held at 180 and 100 °C. The interface was set at 280 °C. Methane was used as reagent gas at an analyser pressure of 1.8 Torr. Masses from 34 to 550 were scanned each 0.8 s. He as carrier gas at 1.5 ml min^{-1} was used.

The quantified analytes were: 11 polychlorinated biphenyl (PCB) congeners, 28 (2,4,4′-trichlorobiphenyl); 31 (2,4′,5-trichlorobiphe-

nyl); 52 (2,2′,5,5′-tetrachlorobiphenyl); 66 (2,3′,4,4′-tetrachlorobiphenyl); 101 (2,2′,4,5,5′-pentachlorobiphenyl); 118 (2,3′,4,4′,5-pentachlorobiphenyl), 138 (2,2′,3,4,4′,5′-hexachlorobiphenyl), 153 (2,2′,4,4′,5,5′-hexachlorobiphenyl); 170 (2,2′,3,3′,4,4′,5′-heptachlorobiphenyl); 180 (2,2′,3,4,4′,5,5′-heptachlorobiphenyl); 187 (2,2′,3,4′,5,5′6-heptachlorobiphenyl), hexachlorobenzene (HCB); the DDT group (*p*,*p*′-DDT, *p*,*p*′-DDE, *o*,*p*′-DDT, *p*,*p*′-DDD, *o*,*p*′-DDD), γ-HCH, aldrin, heptachlor and mirex. The individual PCB congeners were determined by using individual standard congeners. The 11 selected congeners of PCBs are among the major 18 congeners of the NOAA NIST list, which provides at least one representative congener from every homolog group. Total PCB estimates were derived from the sum of the 11 congeners analysed, including the adjustment factor of 2.0 used by NOAA (Howell et al., 2008). Quality control was achieved by subjecting certified reference sediment (IAEA-408, International Atomic Energy Agency) and blank methods to the above procedure and analysing it by the same method as for the samples. The concentration values of the target compounds always fell within the 95% confidence interval of the assigned reference value for concentrations of the analytes (Villeneuve et al., 2000). The precision of the measurements obtained through replicates of the reference materials was better than 10% for all target compounds. The detection limit for each individual compound ranged between 10 and 30 pg g^{-1}.

The concentrations of PCBs (as sum of 11 congeners) in the 17 superficial sediments from Cienfuegos bay are presented in Table 2. They ranged from 1.9 to 16 ng g^{-1} with an average value of 6.2 ng g^{-1}, with the higher values measured in sites near the outfalls of the city of Cienfuegos (SS-11, SS-12), at the thermoelectrical "Carlos Manuel de Cespedes" (SS-10) and in the channel entrance of the bay (SS-1). In this site, polychlorinated terphenyls (PCTs) compounds, which have similar industrial applications as PCBs and HCB, were detected and identified by GC–MS–NICI. The presence of PCTs and the relatively higher levels of PCBs, TOC and pyrolytic PAHs (Tolosa et al., 2009) at site SS-1, indicates that this site is influenced by combustion sources and/or industrial processes dealing with chlorine and/or that these hydrophobic compounds are being preferentially accumulated in this deeper site of the bay (32 m depth).

According to the total PCB estimates (Table 2), sites near the outfalls of the city of Cienfuegos and the thermoelectrical power plant exceeded the minimum values reported for the sediment quality guidelines (ISQG, ERL, TEL, Table 3), but all are below the probable effect levels (PEL, ERM, Table 3). Excepting in the vicinities of Cienfuegos city, the PCB concentrations encountered in the surface sediments are probably not having an adverse effect on sediment-dwelling organisms. Compared to concentrations reported in coastal environments from other parts of the world, PCB concentrations in surface sediments of Cienfuegos bay were relatively lower than other impacted coastal areas of Adriatic (6–2203 ng g^{-1}: Picer, 2000), Vietnam (1.3–384 ng g^{-1}: Hoai et al., 2010), Narragansett Bay in USA (21–1760 ng g^{-1}: Hartmann et al., 2004) and northern Morocco (1–164 ng g^{-1}: Piazza et al., 2009). Nevertheless, they were higher than those reported for remote and pristine environments, such as Gulf of Alaska, Kara sea (Iwata et al., 1994; Sericano et al., 2001), Norwegian Artic (Jiao et al., 2009), deep Mediterranean sea and marine protected areas (Pozo et al., 2009; Tolosa et al., 1995). Overall, PCB concentrations in Cienfuegos were comparable with values reported for various fresh and coastal water ecosystems, such as Mediterranean coastal lagoons (Castro-Jiménez et al., 2008), Black Sea coastal zone (0.1–24 ng g^{-1}: (Fillmann et al., 2002), the Quingdao coastal sea located in China and Gulf of Mexico (Castro-Jiménez et al., 2008; Piazza et al., 2008).

Restrictions on the use of PCBs and their containers were placed in 2001. The inventories of PCB-containing equipment in Cuba recently identified an amount of 140 Tn of PCBs in PCB-containing equipments where the dielectric fluid called Sovol accounted for 63% (87 Tn) of the total quantity of PCB-containing dielectric fluids (Abó Balanza, 2005). Sovol formulation, which is fairly close in composition to Aroclor 1254, and various mixtures of Sovol and trichlorobenzenes distributed under the common name Sovtol (Ivanov and Sandell, 1992) were the major commercial mixtures produced in the former Soviet Union and exported to Cuba before the Soviet bloc collapsed in 1991. The potential sources of PCBs in these Cuban coastal waters are due in part to equipment/utilities still in use (e.g. old transformers and capacitors), waste incineration, accidental fires and atmospheric deposition. Once released into the water column, and due to their hydrophobic nature and high partition coefficients, PCBs tend to adsorb to the suspended particulate matter of the water column and accumulate in sediments. Typical chlorination level plots from the sediment samples and PCB commercial mixtures, such as those of Aroclor and Sovol

Table 2
Concentrations of PCBs and organochlorinated pesticides in sediments from Cienfuegos bay (pg g^{-1} dry wt.).

Compounds	SS-1	SS-2	SS-2A	SS-4	SS-5	SS-6	SS-7	SS-8	SS-9	SS-10	SS-11	SS-12	SS-12A	SS-13	SS-14	SS-15	SS-16
PCB 28 (+31)	435	554	115	445	903	1249	753	823	742	1461	940	1745	1370	564	603	750	79
PCB 52	1625	322	191	160	386	392	592	590	400	776	741	1344	238	255	122	51	51
PCB 66	727	591	517	740	842	840	601	594	809	1081	1257	1069	448	341	187	412	411
PCB 101	830	195	528	155	876	342	846	414	633	1076	1127	1313	608	209	119	530	37
PCB 118	1008	565	505	480	1082	1007	1021	809	912	1347	1219	1694	903	469	466	1182	551
PCB 138 (+163)	1886	396	1073	320	799	761	786	738	642	1627	2394	1432	838	195	343	341	318
PCB 153	1379	331	1017	384	855	888	759	686	1086	1838	3225	1793	737	284	345	407	233
PCB 170	375	64	322	63	152	175	192	39	161	543	1188	397	144	63	77	27	27
PCB 180	568	153	495	215	330	360	343	275	339	961	2127	660	482	130	194	139	113
PCB 187	205	138	363	110	220	204	150	232	240	600	1272	545	145	60	47	32	33
ΣPCBs (11 congeners)	9038	3310	5124	3071	6445	6217	6043	5200	5965	11,309	15,489	11,991	5914	2569	2504	3871	1854
Total PCBs estimates[a]	18 076	6621	10 249	6143	12 889	12 434	12 085	10 400	11 929	22 619	30 979	23 983	11 829	5139	5009	7742	3707
HCB	3536	<22	60	<19	92	<18	<15	<22	204	<27	574	241	<17	<17	<24	<17	<17
Lindane	66	78	70	41	<4	13	<7	<2	21	<6	44	50	213	55	43	76	25
p-*p*′-DDE	167	352	361	356	1091	1029	1543	1484	819	3720	1495	1474	1843	478	2035	1417	1054
p-*p*′-DDD	91	111	463	101	200	277	480	309	307	1600	723	1001	1371	204	512	254	226
p-*p*′-DDT	<14	133	2504	86	<14	<16	75	81	288	307	215	315	9580	181	142	373	556
ΣDDTs	258	596	3328	543	1291	1306	2098	1874	1415	5627	2432	2790	12,794	862	2689	2045	1836
Heptachlor	<23	<23	<24	<24	<23	<23	<19	<27	<30	<34	<33	<18	<21	<21	<30	<21	<21
Aldrin	<19	<23	<20	<20	<19	<19	<16	<22	<25	<28	<27	<15	<18	<17	<25	<17	<17
Mirex	<11	<13	<11	<11	<11	<10	<8	<12	<14	<16	<14	<8	<9	<9	<13	<9	<9

[a] Total PCBs estimated derived from the summed 11 congeners multiplied by a standard factor of two Howell et al. (2008).

Table 3
Sediment quality guidelines (SQGs) (in ng g^{-1} dry wt.) considered for organochlorine contaminants in marine sediments.

	ISQG[a] and TEL[b]	ERL[c]	ERM[d]	PEL[e]	Consensus-TEC[f]	Consensus-PEC[g]	ISQG, TEL, ERL <sites[h]< ERM, PEL and consensus-PEC
Total PCBs	21.5[a,b]	22.7	180	189	29	274	SS-10, SS-11, SS-12
p-p′-DDE	2.07[a,b]	2.2	27	374.2	2.4	55	SS-10
p-p′-DDD	1.22[a,b]	2	20	7.8	1.8	15	SS-10, SS-12A
p-p′-DDT	1.19[a,b]	1	7	4.8	1.3	7	SS-2A
Total DDTs		1.6	46.1	51.7	2.5	39	SS-2A, SS-7, SS-8, SS-10, SS-11, SS-12, SS-12A, SS-14, SS-15, SS-16
HCB						22	
Lindane	0.32[a,b]			0.99			
Heptachlor	0.60[a]			2.74			

[a] ISQG, interim sediment quality guideline after Canadian Environmental Quality guidelines.
[b] TEL, threshold effect level after MacDonald et al. (1996).
[c] ERL, effects range low after Long and Morgan (1990) and Long et al. (1995).
[d] ERM, effects range-median after Long and Morgan (1990) and Long et al. (1995).
[e] PEL, probable effect level after MacDonald et al. (1996).
[f] Consensus-TEC, consensus-based threshold effect concentration after Gómez-Gutiérrez et al. (2007).
[g] Consensus-PEC, consensus-based probable effect concentration after Gómez-Gutiérrez et al. (2007).
[h] Sites exhibiting contaminant concentrations exceeding the ISQG, ERL and TEL but below the PEL, ERM and consensus-PEC; for the *p-p′*-DDT compound, site SS-12A also exceeds the PEL, ERM and consensus-PEC.

(Takasuga et al., 2006) are illustrated in Fig. 2. All profiles indicated that the PCBs in sediments were a composite mixture derived from more than one technical formulation with different degree of chlorination (e.g., 60, 54 and 42 chlorine%). Highest levels of ΣPCBs and the predominance of higher-chlorinated biphenyls in sediments from sites close to the thermoelectrical power station (SS-10, SS-11) and SS-2A indicates a possible local contamination source from a heavy commercial mixtures (e.g., 60 chlorine%). In contrast, the highest contribution of lower chlorinated PCB congeners in the rest of the sediments from the bay probably indicates that the main source of PCB residues is atmospheric transport or that low chlorinated PCBs are transported farther from the local point sources due to their higher solubility and higher vapour pressure (Dunnivant et al., 2002).

Concentrations of *p,p′*-DDT in the sediments of Cienfuegos bay varied from <0.014 to 9.60 ng g^{-1} dry wt. with the highest values at SS-12A (9.60 ng g^{-1}) and SS-2A (2.50 ng g^{-1}); *p,p′*-DDE and *p,p′*-DDD concentrations ranged, respectively from 0.017–3.7 ng g^{-1} to 0.09–1.60 ng g^{-1} with maximum values at sites SS-10 and SS-12A. The high values of ΣDDT residue levels at site SS-12A might be accounted by the discharge of untreated effluents from the Cienfuegos Hospital and the cities of Junco Sur and Tulipán. DDT was used in Cuba since 1950 on a variety of agricultural crops and for the control of human disease vectors. The largest agricultural use of DDT has been on the rice production before its ban in 1989. Noting that the NOAA ERL for ΣDDTs is 1.60 ng g^{-1}, clearly DDT compounds exceed this quality standard at 10 sites from Cienfuegos bay but only the site SS-12A exceeds the probable

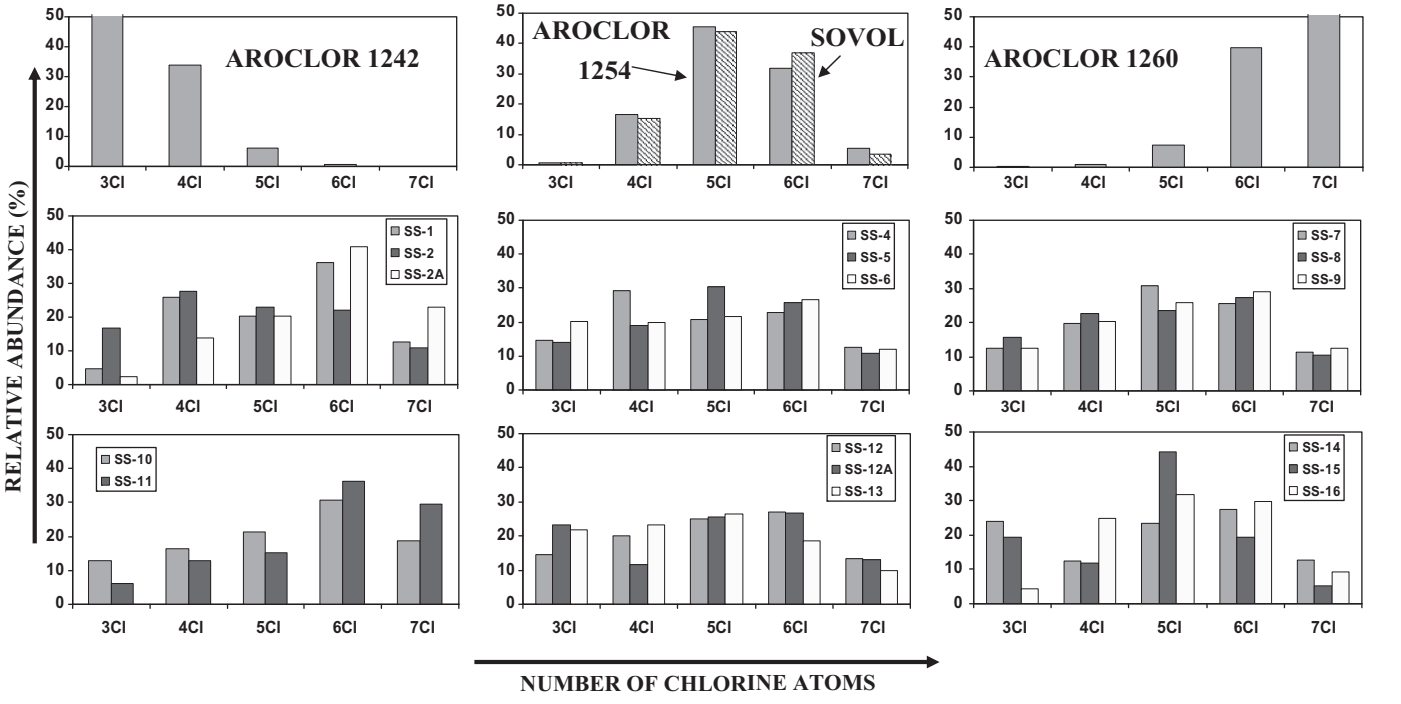

Fig. 2. Relative distribution of PCB congeners according to chlorine substitution (3Cl, trichlorobiphenyls: 28(+31); 4Cl, tetrachlorobiphenyls: 52, 66; 5Cl, pentachlorobiphenyls: 101, 118; 6Cl, hexachlorobiphenyls 138, 153; 7Cl, heptachlorobiphenyls 170, 180, 187) in the different sites. Sovol and Aroclor commercial mixtures (Takasuga et al., 2006) are also shown.

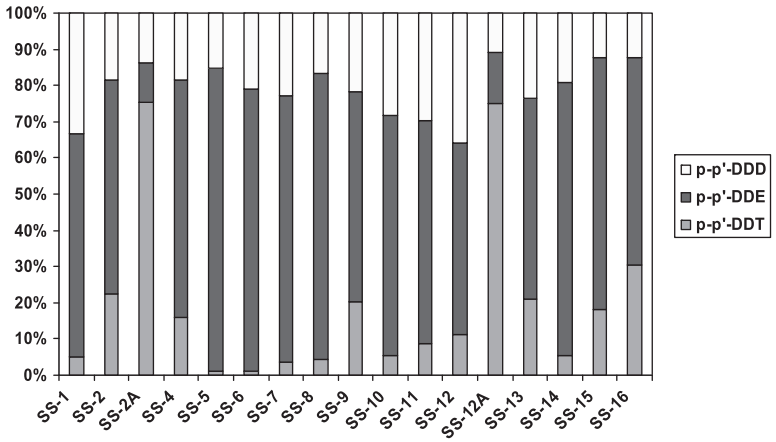

Fig. 3. Percentage of *p,p′*-DDT, *p,p′*-DDE and *p,p′*-DDD in Cienfuegos bay sediments.

effect level (PEL) and the effects range-median (ERM) values (Table 3). Because the reported environmental half-life of DDTs in tropical soils and natural environments is estimated as 6–20 years (Toan et al., 2009; Woodwell et al., 1971), its persistence is expected to decrease slowly.

The pesticide *p,p′*-DDT gradually degrades to *p,p′*-DDE and *p,p′*-DDD through biological and photochemical transformations under both aerobic and anaerobic conditions (Guenzi and Beard, 1976; Purnomo et al., 2008; Thomas et al., 2008; Yao et al., 2006). Fig. 3 showing the relative percentages of *p,p′*-DDT, *p,p′*-DDE and *p,p′*-DDD, highlights that *p,p′*-DDT exceeds 50% of the ΣDDTs at sites SS-2A and SS-12A. The *p,p′*-DDT/(*p,p′*-DDE + *p,p′*-DDD) ratio is usually used to know whether DDT input has occurred recently or not (Jiang et al., 2009; Li et al., 2006). The ratio was >1 at stations SS-2A and SS-12A where maximum ΣDDT concentrations were found. Both a high ΣDDT concentration and high DDT/DDE + DDD ratios point to a current DDT usage in these two sites, probably linked to public health emergencies. Reported soil concentrations of ΣDDTs in the most agro-developed and pesticide-consuming province of Cuba averaged ~200 ng g^{-1} within the period 1976–1983 (Dierksmeier, 1996) and ranged from 4.6 to 61 ng g^{-1} in the southwestern Cuban coast during the period of 1992–2001 (Dierksmeier, 2002). Overall, concentrations of total DDT measured in Cienfuegos bay were lower than those reported in other impacted coastal sediments, such as fishing harbors of China using DDT-containing antifouling paints (9–7350 ng g^{-1}: Lin et al., 2009) and river prodeltas of the NW Mediterranean sediments e.g. the Rhone and Ebro prodeltas (89–675 ng g^{-1}:Tolosa et al., 1995). They compared however to those reported for several coastal sediments in China (e.g. Bohai Sea, Pearl River Estuary, East China Sea, Danshui River Estuary) and some other regions over the world such as Osaka Bay, Japan, Kyeonggi Bay, Korea, coastal areas of Singapore and northern part of the Baltic Sea (Hu et al., 2009 and their citations).

Other chlorinated pesticides, such as HCB, lindane were only present in very low concentrations. HCB is a widespread contaminant that has entered the environment through its past manufacture and use as a pesticide and its formation as a by-product during the production of a variety of chlorinated compounds. HCB concentrations in all samples investigated were found to be in the range of <0.02–3.50 ng g^{-1}. Maximum HCB concentration were found at stations SS-1, SS-2A, SS-5, SS-9, SS-11 and SS-12, but these levels are much lower than the Consensus-PEC value of 22 ng g^{-1} (Table 3) and they are also relatively low compared to those indicating major industrial activities (Wang et al., 2010). Although the HCB levels in the Cienfuegos sediments are higher than those reported for the eastern Artic Sea (0.04–0.08 ng g^{-1}: Iwata et al., 1994), they compare to those from coastal areas of Western Europe (up to 6.7 ng g^{-1}), where pollution is the result of long range atmospheric transport and/or widespread regional contamination (Savinov et al., 2003).

Lindane (γ-HCH) was detected in all sediment samples excepting sites SS-5, SS-10, SS-7 and SS-8 with values ranging from 0.013–0.21 ng g^{-1}. Maximum values were found in site SS-12A. However, these levels did not exceeded the ISQG value of 0.32 ng g^{-1} (Table 3) and compare with those in surface sediment samples from the Chukchi Sea, Bering Sea and the Gulf of Alaska from 0.04 to 0.21 ng/g. (Iwata et al., 1994).

Concentrations of other pesticides, such as aldrin, mirex and heptachlor were lower than the limit of detection (<8–33 pg g^{-1}) and generally not of concern (Tables 2 and 3).

In summary, the contamination of sediments in Cienfuegos bay by PCBs, DDTs and other chlorinated pesticides appeared to be relatively low by global standards and only sediments receiving the impact from the residual waters of the city and thermoelectrical power approached the sediment quality guidelines. The present data provides a baseline to develop, target, and assess the effectiveness of environmental management activities against eventually future anthropogenic pollution of POPs.

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