



Legacy persistent organochlorine pollutants and polycyclic aromatic hydrocarbons in the surface soil from the industrial corridor of South India: occurrence, sources and risk assessment

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Abstract Conversion of agricultural fields into the industrial corridor under the State Industries Promotion Corporation of Tamil Nadu Limited (SIPCOT) necessitated the investigation of soil-borne organic contaminants. This study is the first attempt to evaluate the occurrence of organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) in soils from Mambakkam and Cheyyar SIPCOT belt, along the residential, industrial and agricultural transects. Concentrations of Σ_{28} PCBs, Σ_{16} PAHs and OCPs were in the range 0.3–9 ng/g, 33–2934 ng/g and nd–81.4 ng/g, respectively. Residential areas showed higher OCP concentrations than other site types, probably due to their frequent use in vector control programmes. DDT

isomers and α -isomer of endosulfan showed low concentrations indicating past usage of these OCPs. Principal component analysis indicated that high-temperature combustion and industrial processes might be the major sources of high molecular weight PAHs, while low-temperature combustion processes might be responsible for low molecular weight PAHs. PCBs in soil were probably attributed to unaccounted combustion processes of e-waste in the region. Carcinogenic PAHs and Σ_{28} PCBs were higher in the industrial sites. Mean Σ_{28} PCBs at Mambakkam (4.8 ng/g) was significantly higher ($p < 0.05$) than that at the incipient industrial corridor Cheyyar (2.7 ng/g). Lower chlorinated PCBs (3-Cl and 4-Cl) amounted to more than half of Σ_{28} PCBs in 75% of the sites. Total toxic equivalents (TEQs) of PAHs (total BaP_{eq}) were found to be maximum in industrial areas. Maximum contribution to TEQs due to dioxin-like-PCBs was from PCB-157, followed by PCB-189.

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Introduction

In India, organochlorine pesticides (OCPs) were used mainly for agricultural pest control purposes due to low cost and insecticidal properties (Jayaraj et al. 2016; Sarkar et al. 2008). Although OCPs have been banned or restricted for agricultural use, including in India, some of these OCPs are still used for public health purposes, especially to control vectors that spread malaria and other insect-borne diseases (PPQS 2019; Wilson et al. 2020). OCPs belong to legacy persistent organic pollutants (POPs) and have the ability of long-range transport (Jones and De Voogt 1999). Other legacy POPs such as polychlorinated biphenyls (PCBs) have been widely used in electric transformers and capacitors as heat exchange fluids and in paints, carbonless copy papers, sealants and plastics as additives (Haddaoui et al. 2016; Jiang et al. 2015). The transboundary movement of hazardous waste and its disposal, particularly e-waste, has increased the PCB load in India, China and Africa (Heacock et al. 2016). These legacy POPs continue to be frequently detected in the environment despite their ban by many countries, due to their historical usage and global movement (Breivik et al. 2011; Prithiviraj and Chakraborty 2019; Szlinder-Richert et al. 2008). On the other hand, polycyclic aromatic hydrocarbons (PAHs) are ubiquitous, contributed largely by petroleum products (petrogenic sources) and anthropogenic activities such as incomplete combustion of coal, petroleum products and biomass (pyrogenic sources) (ATSDR 1995; Wilson and Jones 1993; Yan et al. 2005). Due to their potential effects on human and ecological health, 16 PAHs have been classified by the United States Environment Protection Agency (USEPA) and the European Commission as priority pollutants and have to be analysed in various environmental matrices (EC 2001; USEPA 2015). After these chemicals are released into the environment, they are suspected of causing carcinogenic and non-carcinogenic effects on the immune, nervous, endocrine and reproductive systems when their exposure concentrations exceed threshold limits (Tongo et al. 2017; Yahaya et al. 2017).

Soil plays an important role in the global fate and distribution of POPs because soil not only act as a sink but also re-emit these pollutants into the air, thus acting as a secondary source (Wild and Jones 1995). It is important to study soil contamination by POPs

(Manz et al. 2001) because of their greater affinity to soil organic matter (SOM) and the cumulative impact of atmospheric deposition. The concentration of POPs in soils is a good indicator of the proximity of pollution sources and atmospheric pollution levels as these compounds reach soil by dry and wet deposition. Further, these compounds undergo substantial soil adsorption, paving their persistence (Kumar et al. 2011). In addition to temperature-aided circulation that is typical of tropical and subtropical climate observed in different parts of India, soil organic carbon has been observed to play an important role in the fate of OCPs (Chakraborty et al. 2015), PAHs (Sakshi et al. 2019) and PCBs (Chakraborty et al. 2016a). Moreover, suburban and rural areas of India are developing, particularly leading to the expansion of industries, thereby giving rise to elevated levels of industrial POPs such as PCBs (Chakraborty et al. 2016b).

The State Industrial Promotion Corporation of Tamil Nadu (SIPCOT) was established during the late 1990s as part of the economic development programme in Tamil Nadu. Mambakkam and Cheyyar expanded together to 3000 acres hosting various industries such as electronics, automobiles, glass, plastic, metal forging and engineering. In addition to informal e-waste recycling and open burning in dumpsites, the expansion of industries in suburban and rural areas of Indian cities has been found to be an important contributor to soil-borne PCBs and PAHs (Chakraborty et al. 2016a, 2018; Zuo et al. 2007). PCBs have also been reported in the surface dust of Cheyyar and Mambakkam within the framework of SIPCOT (Chakraborty et al. 2016b). Moreover, agricultural activities are still practised over 1000 acres of land in this region, making it the main occupation of the inhabitants. It is necessary to consider the amount of pesticide applied as well as the residues left in the soil to improve the estimation of the emission rates. Besides, it is fundamental to study these pollutants in the soil because they can contaminate the plants growing in this soil by re-emission and atmospheric deposition. Therefore, there is a serious health risk for humans and ecosystems upon which we depend due to the bioaccumulative property of most POPs (Pokhrel et al. 2018).

This study focused on three main objectives: (1) to estimate soil-borne OCPs, PCBs and PAHs in the Mambakkam and Cheyyar industrial corridor under SIPCOT along the residential, industrial and

agricultural transects; (2) to identify sources using compositional profiles, multivariate principal component analysis (PCA) and correlation with soil total organic carbon (TOC); and (3) to estimate the potential risk using toxic equivalency of PAHs and dioxin-like (dl)-PCBs.

Materials and methods

Study area

Kanchipuram is a suburban district located on the outskirts of Chennai in the north-east of Tamil Nadu, India. Several industries, including electronics, automobiles, glass, iron and steel, plastics, etc., were established in the area under the SIPCOT scheme. Two major regions, Mambakkam, which has been an industrial area for the past 15 years, and Cheyyar, which is a developing industrial corridor with past agricultural activities, in the SIPCOT belt were chosen as study areas. The sampling site locations were recorded using a global positioning system (GPS) and are reported in Table S1 of Supplementary Information (SI). The residential (RS), industrial (IS) and agricultural (AS) sites were sampled in both regions. Two control samples C-1 and C-2 were collected from the hillock located 30 km away from the SIPCOT region.

Sampling and sample extraction

Surface soil (0–20 cm) samples from Mambakkam ($n = 15$) and Cheyyar ($n = 15$) were collected using a stainless-steel scoop pre-cleaned with acetone. Each representative sample consisted of five sub-samples from a 10×10 m grid. Samples were collected in a polyethylene Ziploc bag and stored at -20 °C until extraction. Soil samples were air-dried, ground with mortar and pestle, and sieved through 1 mm before analysis. About 20 g of soil samples was extracted using Soxhlet with 150 mL of dichloromethane (DCM) for 18 h. Deuterium-labelled PAHs (Naphthalene-D8, Acenaphthene-D10, Phenanthrene-D10, Chrysene-D12 and Perylene-D12), 2,4,5,6-tetrachlorom-xylene (TCmX), decachlorobiphenyl (PCB-209), $^{13}\text{CPCB-138}$ and $^{13}\text{CPCB-180}$ were used as surrogates. Activated copper granules were added to each collection flask to remove potential elemental

sulphur. The extract was filtered through a funnel filled with anhydrous sodium sulphate and then reduced to 2 mL by rotary evaporator and solvent exchanged to *n*-hexane.

Column clean-up

The concentrated extract was divided into two separate aliquots: the first one for the analysis of OCPs and PCBs and the second for the analysis of PAHs. The aliquots were further subjected to the clean-up process as follows:

OCPs and PCBs

The sample extract was passed through an 8-mm i.d. chromatography column containing, from bottom to top, 6 cm alumina (BDH neutral alumina, 3% deactivated), 10 cm silica gel (Merck silica gel 60, 3% deactivated), 10 cm of 50% sulphuric acid silica and 1 cm of sodium sulphate (all baked at 450 °C overnight). The column was eluted with 10 mL of a 50:50 v/v *n*-hexane/DCM mixture. The samples were further reduced to a final volume of 25 μL by nitrogen blowdown. 25 μL of dodecane was added as a solvent keeper, and a known quantity of pentachloronitrobenzene (PCNB) was added as an internal standard prior to analysis.

PAHs

The sample extract was cleaned using a column packed with 10 g activated silica gel and topped with 5 g anhydrous sodium sulphate. The silica column was pre-washed with 100 mL of *n*-hexane before the sample was loaded. Target compounds were eluted with 100 mL 1:1 v/v *n*-hexane/DCM mixture. The solvent was finally blown down to 20 μL under a gentle stream of ultrapure nitrogen. Hexamethylbenzene was added to the gas chromatography (GC) vial as an internal standard before analysis.

Instrumental analysis

OCPs, PCBs and PAHs were analysed in a GC (Agilent 7890) equipped with a 5977A mass spectrometer (MS) detector. Fourteen OCPs (*o,p'*-DDT, *p,p'*-DDE, *p,p'*-DDT, *p,p'*-DDD, α -, β -, γ - and δ -HCH, aldrin, dieldrin, α -, β -endosulfan and endosulfan

sulphate), twenty-eight PCB congeners (PCB-28, -37, -44, -49, -52, -70, -74, -77, -81, -105, -114, -118, -119, -123, -126, -128, -138, -151, -156, -157, -167, -168, -169, -170, -177, -187, -189, -207) and sixteen priority PAHs [naphthalene (NPT), acenaphthene (ANE), acenaphthylene (Any), fluorene (FLE), phenanthrene (PHE), anthracene (ANT), fluoranthene (FLT), pyrene (PYR), benzo[a]anthracene (BaA), chrysene (CHR), benzo[b]fluoranthene (BbF) and benzo[k]fluoranthene (BkF), benzo[a]pyrene (BaP), dibenz[a,h]anthracene (DBA), benzo[g,h,i]perylene (BghiP) and indeno[1,2,3-c,d]pyrene (IndP)] were analysed in all the samples. HP-5MS Ultra Inert (30 m × 0.25 mm × 0.25 µm) capillary GC column was used for analysis. The flow rate of helium as the carrier gas was 1.2 mL/min at constant flow operating under single ion monitoring (SIM) mode. The solvent delay was fixed at 5 min. Injector temperature was maintained at 250 °C. The inlet degradation of DDT was checked daily and controlled within 15%. The method of internal standard was used for pesticide quantification.

For PCB quantification, the oven temperature was initially maintained at 100 °C for 1 min, then increased to 140 °C at the rate of 4 °C/min, then ramped to 180 °C at the rate of 20 °C/min and further increased to 210 °C at the rate of 3 °C/min and finally to 290 °C (10 min hold time) at 8 °C/min.

For PAHs, the injector temperature was kept at 290 °C. The transfer line was maintained at 300 °C. The oven was initially set at 60 °C for 1 min, then increased to 290 °C at the rate of 4 °C/min and held for 20 min.

Quality assurance/quality control (QA/QC)

All the glassware used in sample preparation was ultrasonicated, cleaned and dried at 150 °C in an oven for 6 h. HPLC grade solvents were used for extraction and analysis. The EPA610 PAH solution-certified reference material was purchased from Sigma-Aldrich (USA) and used for calibration. Each sample was analysed in duplicate, and the mean concentration value was used for calculations. The concentrations were reported below detection limits if the signal (S) did not exceed the specified threshold limit [three times the noise (N)]. Eight aliquots of a sample were spiked with a quantity sufficient to produce a detectable response ($S/N > 3$), and the standard

deviation thus obtained was multiplied by 3 (Student's *t* test, i.e. t_{student} value for eight replicates) to establish method detection limits (MDLs). The instrumental stability was checked daily using standard compounds, and the deviation was found to be less than 15%. MDLs were 0.01 ng/g for both OCPs and PCBs and 0.01–0.5 ng/g for PAHs in soil. The surrogate recoveries were 81–118% for TCmX, 85–105% for PCB-54 and 87–114% for deuterated PAHs.

Determination of soil total organic carbon

TOC was calculated using the loss-on-ignition method (Salehi et al. 2011). Briefly, samples were oven-dried at 105 °C overnight to remove moisture content. The dried samples were weighed to determine the initial weights and then combusted in a muffle furnace at 360 °C for 2 h. The samples were weighed again after combustion to determine the final weights. %TOC was calculated based on the difference in the weights.

Statistical analysis

All the statistical procedures, including one-way analysis of variance (ANOVA), correlation and principal component analysis (PCA), were performed using the software package SPSS 16.0 for Windows (IBM, USA). Values less than the detection limits were set to zero for statistical purposes.

Toxicity evaluation of PAHs and dioxin-like-PCBs

The International Agency for Research on Cancer (IARC) has classified some PAHs as carcinogenic (Group 1), probably or possibly carcinogenic to humans (Group 2A and 2B, respectively). A common approach in estimating the carcinogenic potency associated with the exposure of a given PAH can be obtained by calculating its BaP equivalent concentration (ng/g). The toxicity equivalent concentration (TEQ) of individual PAH (BaP_{eq}) was evaluated by multiplying the PAH concentration in the sample by the individual toxicity equivalency factor (TEF) relative to the BaP carcinogenic potency as given in Tsai et al. (2004). The total TEQs (total BaP_{eq}) were then obtained by summing the individual BaP_{eq} using the following equation:

$$\begin{aligned} \text{Total} - \text{BaP}_{\text{eq}} = & [\text{NPT}] \times 0.001 + [\text{Any}] \times 0.001 \\ & + [\text{ANE}] \times 0.001 + [\text{ANT}] \times 0.01 \\ & + [\text{FLE}] \times 0.001 + [\text{PHE}] \times 0.001 \\ & + [\text{FLT}] \times 0.001 + [\text{PYR}] \times 0.001 \\ & + [\text{BaA}] \times 0.1 + [\text{CHR}] \times 0.01 \\ & + [\text{BbF}] \times 0.1 + [\text{BkF}] \times 0.1 \\ & + [\text{BaP}] \times 1 + [\text{IndP}] \times 0.1 \\ & + [\text{DBA}] \times 1 + [\text{BghiP}] \times 0.01 \end{aligned}$$

TEFs for dl-PCBs provided by the WHO (Van den Berg et al. 2006) were used for the toxicity evaluation of dl-PCBs in each site.

Results and discussion

Organochlorine pesticides

The concentrations of all the OCPs are reported in Table 1, and the spatial distribution is shown in Fig. 1. OCP concentrations were nd–36 ng/g in Mambakkam and 2–81 ng/g in Cheyyar. Both these sites were primarily agricultural sites, later planned and established as industrial zones. Cheyyar came up as an

industrial district much later than Mambakkam, probably justifying the higher pesticidal residues in Cheyyar (mean ± SD; 15 ± 20 ng/g) with respect to Mambakkam (11 ± 12 ng/g). However, mean OCP concentrations were comparable in IS of Mambakkam and Cheyyar. In general, OCPs were found in the order HCHs > DDTs > endosulfans > dieldrin. HCHs contributed to more than 80% of OCPs, followed by DDTs (12%). This is a typical feature of OCP contamination in different environmental media from India so far, reflecting continuous usage of HCHs for public health purposes and on certain food crops in the Asia Pacific, including India (Chakraborty et al. 2010; Kumar et al. 2012; Tanabe 2007; Tanabe and Minh 2010). Higher HCH concentrations were observed in the RS of both Mambakkam and Cheyyar compared with those in the IS, while DDTs were equally distributed. Interestingly, the lowest concentrations of OCPs were observed in the AS, reflecting the strict ban of HCHs and DDTs for agricultural usage in India (PPQS 2019; UNEP 2017). Mean HCH and DDT levels determined in this study were lower than those found in Indian soil samples from Chennai, Bangalore, New Delhi and Kolkata (Chakraborty et al. 2015), few districts of Kerala and Tamil Nadu along the south-

Table 1 Concentrations of organochlorine pesticides (OCPs) in soil (ng/g)

Compound	Overall		Mambakkam		Cheyyar	
	Range	Mean ± SD	Range	Mean ± SD	Range	Mean ± SD
α-HCH	nd–45	6.4 ± 9.3	nd–19	5.5 ± 6.5	1.1–45	7.3 ± 11.5
β-HCH	nd–1.7	0.1 ± 0.3	nd–0.0	–	nd–1.7	0.2 ± 0.5
γ-HCH	nd–23.8	3.2 ± 4.6	nd–7.7	2.5 ± 2.8	nd–24	3.9 ± 5.9
δ-HCH	nd–8.4	1.2 ± 1.9	nd–4.6	1.1 ± 1.6	nd–8.4	1.4 ± 2.3
Σ-HCHs	nd–79	11 ± 16	nd–30	9 ± 11	1.1–79	12.7 ± 20
α-Endo	nd–1.8	0.1 ± 0.4	nd	–	nd–1.8	0.2 ± 0.5
β-Endo	nd–4.0	0.3 ± 0.9	nd–1.0	0.1 ± 0.3	nd–4	0.6 ± 1.2
Endo-sulphate	nd–1.7	0.1 ± 0.3	nd	–	nd–1.7	0.1 ± 0.4
Σ-Endos	nd–7.5	0.5 ± 1.5	nd–1.0	0.1 ± 0.3	nd–7.5	0.9 ± 2.0
<i>o,p'</i> -DDE	nd–2.6	0.7 ± 0.8	nd–2.6	0.9 ± 0.8	nd–1.9	0.5 ± 0.7
<i>p,p'</i> -DDE	nd–1.7	0.8 ± 0.6	nd–1.3	0.8 ± 0.4	nd–1.7	0.7 ± 0.7
<i>p,p'</i> -DDD	nd–1.4	0.1 ± 0.3	nd–1.4	0.2 ± 0.4	nd	–
<i>o,p'</i> -DDT	nd	–	nd	–	nd	–
<i>p,p'</i> -DDT	nd–1.6	0.1 ± 0.3	nd–1.6	0.1 ± 0.4	nd	–
Σ-DDTs	nd–5	1.6 ± 1.5	nd–5	2.0 ± 1.6	nd–3	1.2 ± 1.2
Aldrin	nd	–	nd	–	nd	–
Dieldrin	nd–0.09	0.09 ± 0.3	nd	–	nd–0.8	0.1 ± 0.2
Σ₁₄OCPs	nd–81.4	13.0 ± 16.4	nd–35	11.2 ± 11.9	2–80	14.8 ± 20.2

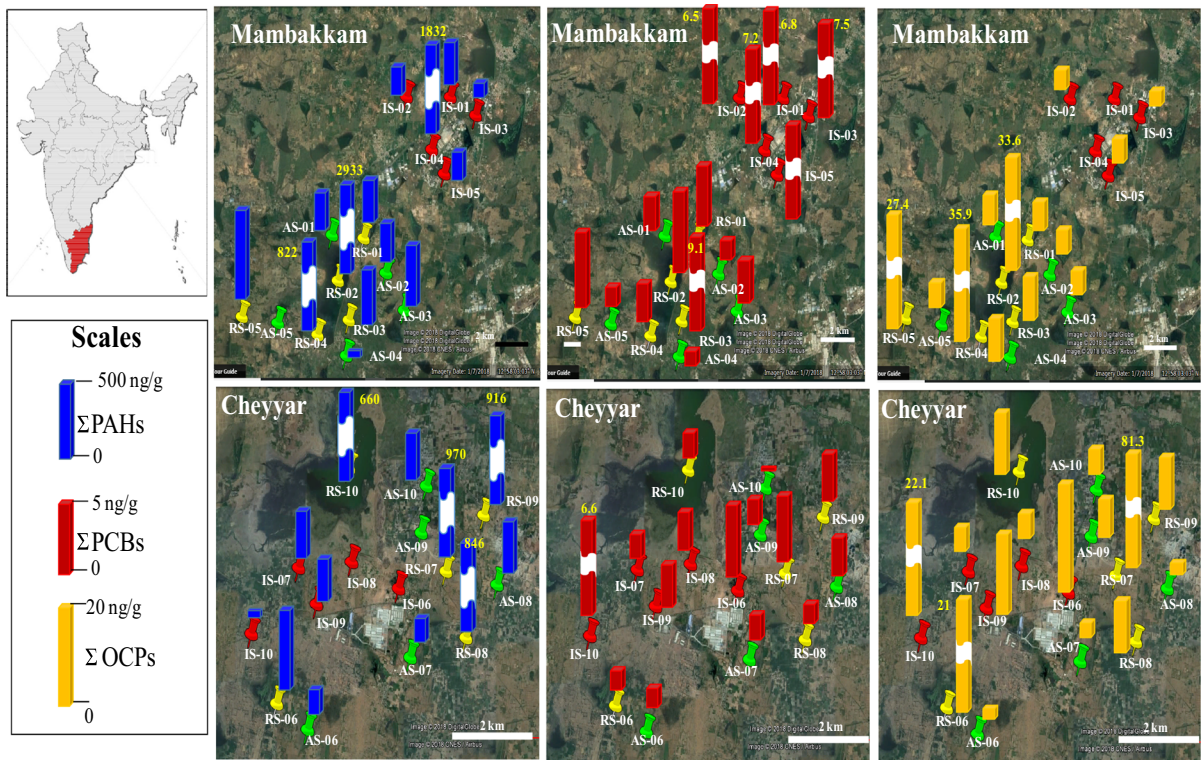


Fig. 1 Spatial distribution of total OCPs, PCBs and PAHs in the surface soil of Mambakkam and Cheyyar

west coast of India (Khuman et al. 2020a) and Hanoi region in Vietnam (Toan et al. 2007). The mean concentration of DDTs was higher than that observed in Agra (Singh 2001) in India, whereas the mean concentration of endosulfans was lower than that reported from other Indian cities (Chakraborty et al. 2015), Hissar (Haryana) in northern India (Kumari et al. 2008) and Punjab in Pakistan (Syed et al. 2013). Moreover, the mean HCH and DDT levels in IS were lower than those detected in Beijing (China) (Yang et al. 2009) and Pakistan (Syed and Malik 2011). On the other hand, the mean HCH and DDT levels in AS were lower than those reported for soils from Assam (India) (Mishra et al. 2012) and Hong Kong (Zhang et al. 2006).

HCHs

HCH (sum of four HCH isomers, i.e. α -, β -, γ - and δ -HCH) levels were the highest among all the OCPs analysed (nd–79 ng/g) and contributed 80% and 49% to OCPs in Mambakkam and Cheyyar, respectively (Fig. 2). HCH levels were in the order RS > IS > AS.

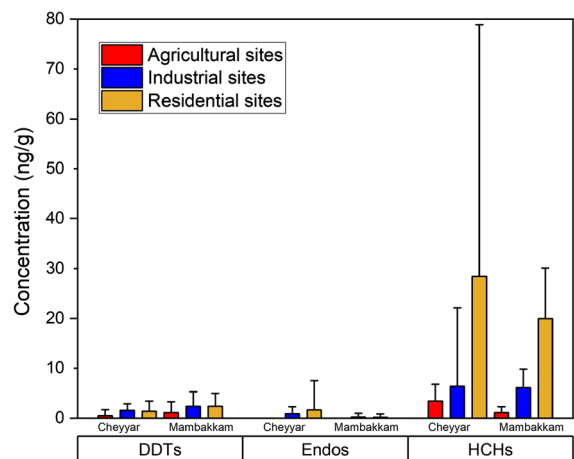


Fig. 2 Concentration of OCPs in different site types (error bars show maximum values)

HCH levels in the RS were significantly different from those in IS ($p < 0.05$) and AS ($p < 0.05$). The higher levels of HCHs in the RS can be mainly attributed to the continuous usage of HCHs for public health purposes, i.e. in vector control programmes, due to low cost and high effectiveness (Mishra et al. 2012).

The mean HCH concentration (≈ 28 ng/g) in the RS of the incipient industrial corridor of Cheyyar with a history of agricultural activities was higher than that in the RS of the established industrial corridor Mambakkam (≈ 20 ng/g). On the other hand, the mean HCH concentration was slightly higher in Cheyyar (13 ng/g) than in Mambakkam (9 ng/g), with the highest concentration observed at RS-07 in Cheyyar (79 ng/g). β -HCH was detected only at two sites in Cheyyar (RS-07 and RS-10). α -HCH was the predominant isomer, followed by γ -HCH. This trend could be explained considering that technical HCH has been extensively used in the past for paddy cultivation in the southern part of India (Courdouan et al. 2004; Ramesh et al. 1991). α -HCH was dominant, contributing about 50% to HCH levels followed by δ -HCH, γ -HCH and β -HCH in the surface soils along the south-west coast of India (Khuman et al. 2020a). However, most soil samples along the urban–suburban–rural transects of Mumbai, Kolkata, Chennai and Bangalore have shown a predominance of β -HCH, followed by γ -HCH (Chakraborty et al. 2015). Moreover, the current HCH levels are probably associated with the use of technical mixture in vector control programmes in the RS. At all sites, the α/γ HCH ratio was greater than 1, suggesting the usage of technical HCHs. The ratio of $\beta/(\alpha + \gamma)$ HCH was less than 1 in the two sites where β -HCH was detected, further confirming the ongoing usage of technical HCH (Khuman et al. 2020b). α -HCH, γ -HCH and δ -HCH concentrations were significantly correlated ($R^2 > 0.9$) with total HCH concentrations ($p < 0.01$) in both Mambakkam and Cheyyar.

DDTs

DDTs (sum of o,p' -DDE, p,p' -DDE, p,p' -DDD, o,p' -DDT and p,p' -DDT) ranged from nd to 5.3 ng/g, with a mean of 1.6 ng/g. DDT levels were in the order $RS \approx AS > IS$ for Mambakkam and Cheyyar (Fig. 2). p,p' -DDE was the most frequently detected and predominant metabolite among DDTs (67% detection frequency) followed by o,p' -DDE. DDTs contributed approximately 26% and 14% to OCPs found in Mambakkam and Cheyyar, respectively. p,p' -DDT has been observed only in one site (i.e. IS-04). In 1989, the Indian government withdrew the use of DDT in agriculture and restricted its use only in the control of vector-borne diseases. Currently, DDT is produced for domestic use and exported in accordance with the

Stockholm Convention (PPQS 2019). The presence of p,p' -DDT could be related to its ongoing use in vector control (Sahu et al. 2015), while the presence of p,p' -DDE could be due to the degradation of p,p' -DDT in the tropical climate of Chennai which is hot and humid for most of the year (Chakraborty et al. 2015). DDE, being the dominant DDT residue, indicated aerobic degradation of DDT in the past (Doong et al. 2002). o,p' -DDE and p,p' -DDE are significantly correlated with each other ($R^2 = 0.7$) and with DDTs ($R^2 = 0.97$ and $R^2 = 0.75$, respectively) ($p < 0.01$), indicating past use.

Endosulfans

Endosulfans (sum of α -endosulfan, β -endosulfan and endosulfan sulphate) ranged between nd and 7.5 ng/g, with a mean of 0.5 ng/g. α/β diagnostic ratio to determine endosulfan sources was not applicable in this study as endosulfans were detected only in 13% of sites in Mambakkam and 27% of sites in Cheyyar (Fig. 2), with β -endosulfan being the most dominant. Among the two endosulfan isomers, β -endosulfan strongly binds with the soil particles. Therefore, its soil concentration seems to be a useful indicator of the time from exposure to endosulfan (Kole et al. 2001; Kumar et al. 2011). Unlike past observations showing α -endosulfan as the dominant compound in and around Chennai city (Chakraborty et al. 2015), the predominance of β -endosulfan in this study indicates an aged source owing to past usage for agricultural purpose.

Polychlorinated biphenyls

The total concentration of PCB congeners, i.e. Σ_{28} PCBs, in soil ranged from 0.3 to 9.1 ng/g (mean \pm SD, 3.7 ± 2.4 ng/g). Table 2 lists the concentration mean and ranges of all the PCB congeners at each site. Σ_{28} PCBs in soil samples from the Mambakkam region (4.8 ± 2.5 ng/g) was almost 1.5 times significantly higher ($p < 0.05$, ANOVA) than those in the Cheyyar region (2.7 ± 1.3 ng/g). Previously, Chakraborty et al. (2016b) also observed a similar difference in the mean PCB levels in surface dust between these two regions. The major reason for this variation might be because the Mambakkam region is a two-decade-old industrial establishment under SIPCOT compared to the Cheyyar region, which is a relatively newly developed industrial corridor. In general, the geomean

Table 2 Concentrations of polychlorinated biphenyls (PCBs) in soil (ng/g)

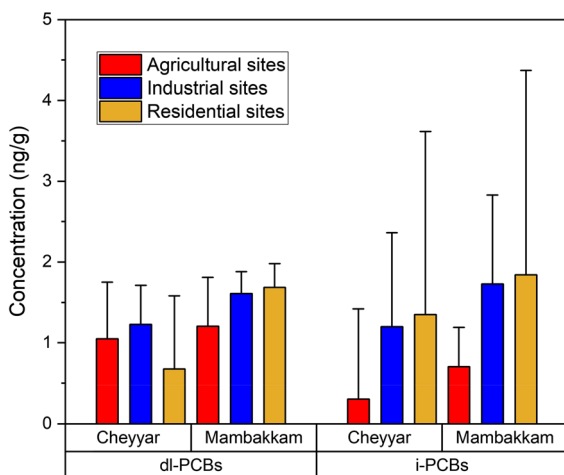
	Overall		Mambakkam		Cheyyar	
	Range	Mean \pm SD	Range	Mean \pm SD	Range	Mean \pm SD
PCB-37	nd–2.1	0.6 \pm 0.8	nd–2.1	0.8 \pm 0.8	nd–2	0.3 \pm 0.7
PCB-52	nd–2.0	1.2 \pm 0.7	nd–2	1.5 \pm 0.5	nd–1.8	1.0 \pm 0.8
PCB-74	nd–1.5	0.6 \pm 0.7	nd–1.5	0.8 \pm 0.7	nd–1.3	0.3 \pm 0.5
PCB-77	nd–1.4	0.1 \pm 0.3	nd–0.002	–	nd–1.4	0.2 \pm 0.4
PCB-114	nd–1.2	0 \pm 0.2	nd	–	nd–1.2	0.1 \pm 0.3
PCB-119	nd–1.2	0.1 \pm 0.3	nd	–	nd–1.2	0.2 \pm 0.4
PCB-118	nd–0.1	0 \pm 0.01	nd	–	nd–0.1	0.005 \pm 0.02
PCB-123	nd–0.1	0 \pm 0.01	nd	–	nd–0.1	0.007 \pm 0.02
PCB-151	nd–1.1	0 \pm 0.2	nd–1.1	0.1 \pm 0.3	nd–0.1	0.007 \pm 0.02
PCB-157	nd–2.8	0.6 \pm 0.8	nd–2.8	0.9 \pm 0.9	nd–2.4	0.4 \pm 0.8
PCB-170	nd–1.1	0.0 \pm 0.2	nd–1.1	0.1 \pm 0.3	nd–0.1	0.004 \pm 0.02
PCB-189	nd–1.5	0.4 \pm 0.6	nd–1.5	0.6 \pm 0.6	nd–1.4	0.2 \pm 0.5
PCB-207	nd–0.1	0.003 \pm 0.01	nd	–	nd–0.1	0.006 \pm 0.02
Σ_{28}PCB*	0.3–9.1	3.7 \pm 2.4	1.2–9.1	4.8 \pm 2.5	0.3–6.6	2.7 \pm 1.7

*Remaining PCB congeners were not detected (nd)

of Σ_{28} PCBs was in the order IS (4.9 ng/g) > RS (3.5 ng/g) > AS (1.5 ng/g). Further, Σ_{28} PCBs in Mambakkam and Cheyyar were found to be significantly different for IS ($p < 0.05$) and RS ($p < 0.05$), thereby indicating different source types. Although the PCBs were never manufactured in India and their usage was restricted only to power sectors (UNIDO 2011), PCBs in industrial areas can originate from incineration and as by-products from a wide variety of chemical processes. Among the indicator PCBs (i-PCBs), PCB-52 was widely observed at all sites and recorded the highest concentration except in the IS of

Mambakkam. In contrast, levels of dl-PCBs were comparable between IS and RS (Fig. 3), while a threefold to fourfold lower concentrations were observed in the AS.

The geomean concentration of i-PCBs in this study was higher in Mambakkam (1.2 ng/g) than Cheyyar (0.4 ng/g) (Fig. 3). The geomean concentrations of dl-PCBs were 0.4 ng/g and 0.2 ng/g in Mambakkam and Cheyyar, respectively. The geomean concentrations of soil PCBs previously recorded in suburban (2.5 ng/g) and rural (2.3 ng/g) transects of Chennai (Chakraborty et al. 2018) were higher when compared to that in the current study across all site types. While the concentrations of dl-PCBs were comparable to the corresponding concentrations in the Mambakkam region, dl-PCB concentrations were lower in the RS, IS and AS of Cheyyar. Moreover, the mean concentration of dl-PCBs in the present study (1.7 ng/g) was almost fourfold lower than that in the agricultural soils of Delhi (6.3 ng/g) (Kumar et al. 2011) and about 2.5 times higher than that in the residential soils (0.7 ng/g) from the industrial city of Chhattisgarh (Kumar et al. 2014), India. The mean concentration of i-PCBs (1.2 ng/g) from this study was comparable to that reported in soils from the e-waste dismantling sites (1.4 ng/g), e-waste shredding sites (1.5 ng/g) and open dumpsites (0.9 ng/g) of four major Indian cities (Chakraborty et al. 2018) but higher than that in urban

**Fig. 3** Concentration of PCBs in different site types (error bars show maximum values)

soils from an industrialized city in Turkey (0.35 ng/g) (Salihoglu et al. 2011).

Polycyclic aromatic hydrocarbons

Surface soil PAH concentrations for all the sampling sites are provided in Table 3. The highest concentration of total PAHs (Σ_{16} PAHs) was observed at RS-02 (2934 ng/g). Site RS-02 also showed the highest concentrations of low molecular weight (LMW) PAHs such as Any (953 ng/g), ANE (536 ng/g) and NPT (400 ng/g). On the other hand, the IS-04 site showed the highest levels of high molecular weight (HMW) PAHs, such as DBaH (290 ng/g) and FLT (205 ng/g). Site IS-04 also had the highest concentrations of carcinogenic PAHs.

Comparing mean concentrations of Σ_{16} PAHs, the sites were in the order RS > IS > AS. However, for carcinogenic PAHs, the sites were in the order IS > RS > AS. In Mambakkam, the sites were in the order RS (985 ng/g) > IS (517 ng/g) > AS (181 ng/g); however, the order was RS (785 ng/g) > AS (191 ng/g) > IS (126 ng/g) in Cheyyar. The mean concentration of Σ_{16} PAHs (321 ng/g) in

the IS was higher compared with that in the IS of Agra (13.22 ng/g) (Masih and Taneja 2006) and Spain (120 ng/g) (Nadal et al. 2007), but lower than that in the IS of Beijing (3917 ng/g) (Tang et al. 2005), South Korea (960 ng/g) (Kwon and Choi 2014) and South Africa (7200 ng/g) (Nieuwoudt et al. 2011). The mean concentration of Σ_{16} PAHs in the AS (186 ng/g) of this study was less when compared with that in the agricultural soils from South Korea (236 ng/g) (Nam et al. 2003) and rural sites in Delhi (886 ng/g) (Agarwal 2009). Moreover, the mean concentration of Σ_{16} PAHs in the RS (885 ng/g) was higher than that in the RS of Agra (6.73 ng/g), northern India (Masih and Taneja 2006), South Africa (360 ng/g) (Nieuwoudt et al. 2011) and Spain (207 ng/g) (Nadal et al. 2007).

Ring-wise profiling of PAHs was in the order: 2-ring (34%) > 3-ring (33%) > 5-ring (16%) > 4-ring (11%) > 6-rings (6%), indicating that LMW PAHs (2- and 3-ring) contributed about 67% to Σ_{16} PAHs, as shown in Fig. 4. The RS in Mambakkam contribute about 80–100% LMW PAHs to Σ_{16} PAHs. LMW PAHs are generated at low to moderate temperatures such as during wood and coal

Table 3 Concentrations of polyaromatic hydrocarbons (PAHs) in soil (ng/g)

	Overall		Mambakkam		Cheyyar	
	Range	Mean ± SD	Range	Mean ± SD	Range	Mean ± SD
NPT	nd–400.5	158.5 ± 119.9	nd–400.5	163.5 ± 118.6	nd–312.4	124.7 ± 116.8
Any	nd–953.8	31.8 ± 174.1	nd–953.8	63.6 ± 246.3	nd	–
ANE	nd–536.2	17.9 ± 97.9	nd–536.2	35.7 ± 138.4	nd	–
FLE	nd–179	33.6 ± 38.2	nd–179	33.9 ± 46.2	nd–77.9	28.6 ± 46.8
PHE	nd–359.8	63.0 ± 101.9	nd–270	76.7 ± 92.0	nd–359.8	48.3 ± 97.6
ANT	nd–74.5	7.8 ± 17.0	nd–74.5	10.1 ± 20.8	nd–38.0	3.2 ± 20.0
FLT	nd–204.9	22.7 ± 50.5	nd–204.9	32.3 ± 62.2	nd–107.7	6.4 ± 54.8
PYR	nd–173.1	13.6 ± 36.4	nd–173.1	19.4 ± 47.3	nd–66.6	3.6 ± 46.3
BaA	nd–116	8.2 ± 23.4	nd–116	9.9 ± 30.1	nd–51.9	6.0 ± 32.8
CHR	nd–127.7	6.7 ± 23.7	nd–127.7	12.1 ± 33.0	nd–12.7	0.6 ± 33.9
BbF	nd–171	13.5 ± 32.9	nd–171	21.5 ± 44.5	nd–29.9	5.1 ± 45.0
BkF	nd–143.4	12.3 ± 36.3	nd–129.1	15.1 ± 36.7	nd–143.4	10.2 ± 59.1
BaP	nd–176.9	20.1 ± 49.4	nd–176.9	19.2 ± 52.1	nd–143.4	19.6 ± 64.8
BghiP	nd–164.2	18.5 ± 50.1	nd–72.9	4.9 ± 18.8	nd–164.2	34.5 ± 75.9
DBA	nd–290	26.8 ± 61.7	nd–290	30.9 ± 76.4	nd–145.7	11.3 ± 77.3
IndP	nd–150.3	9.0 ± 29.7	nd–150.3	12.2 ± 39.1	nd–60.5	6.2 ± 42.0
Σ_{16} PAHs	nd–2934	464.1 ± 609.5	nd–2933.9	560.8 ± 795.9	nd–970.8	308.5 ± 752.8

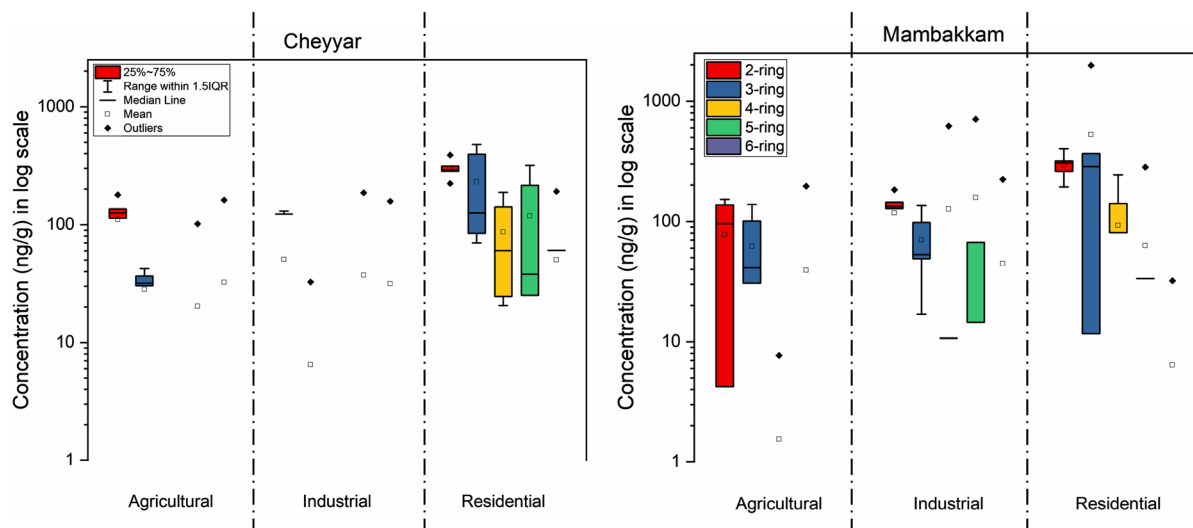


Fig. 4 Box plots showing concentration of PAHs in different site types

combustion, whereas HMW PAHs (≥ 4 rings) are generated at high temperature, particularly from vehicular emissions (Xiao et al. 2014).

AS were dominated by LMW PAHs. The majority of PAHs emitted from biomass are LMW PAHs, including NPT and PHE. The predominance of 2- and 3-ring PAHs in the agricultural soil is mainly due to the highly volatile nature of LMW PAHs. Burning of agricultural residues is a common practice at such sites (Singh et al. 2012). IS have almost equal levels of LMW and HMW PAHs, thus indicating mixed sources.

Relationship with total organic carbon

POPs tend to bind with the soil organic matter. In this study, the soil TOC was in the range 11–44%, and no correlation was found between TOC and POP concentrations in the soil. TOC distribution in the study area is reported in Table S1 in SI. A stronger correlation of POPs with TOC might indicate a historical use of POPs, and no correlation can indicate more recent contamination (Gong et al. 2004; Jiang et al. 2009). Moreover, no correlation indicates non-equilibrium conditions between POPs in the given matrix at a particular location and the corresponding TOC due to unceasing contamination (Katsoyiannis 2006). It has also been previously stated that lack of correlations between TOC and OCPs, PCBs or PAHs indicates a relatively fresh source (Chakraborty et al.

2015, 2018, 2019). This trend of no correlation between TOC and PAHs is consistent with the studies involving traffic soils of Delhi (Agarwal 2009) and urban soils, Shanghai, China (Jiang et al. 2009).

Source apportionment using principal component analysis and compositional profiles

Data sets (including OCPs, PCBs and PAHs) were used to perform PCA to evaluate chemical sources in the SIPCOT belt (Table 4). Major sources were identified using predominant compounds showing significant correlations ($R^2 > 0.5$). The three principal components, PC-1, PC-2 and PC-3, contributed 23%, 20% and 9% of the variance, respectively.

PC-1

PC-1 comprised of *p,p'*-DDT and HMW PAHs, viz. FLT, PYR, BaA, CHR, BbF, BaP, DBA and IndP ($R^2 \geq 0.7$). RS and IS contributed more than 80% of HMW PAHs, with IS contributing more than 50% except for FLT, PYR, BaA and BaP. 4- to 6-ring PAHs are primary products of incomplete combustion; in fact, FLT indicates heavy-duty diesel combustion source (Marr et al. 1999), while PYR, BaA, CHR and FLT are markers emitted from coal combustion (Tavakoly Sany et al. 2014). In general, it can be stated that HMW PAHs are generated at high temperature, particularly from vehicular emissions (Mastral

Table 4 Component matrix obtained after PCA

	Component		
	1	2	3
BbF	0.954	0.137	
CHR	0.948	0.114	
BaA	0.947		0.110
IndP	0.946		
p,p'-DDT	0.899		
DBA	0.841		0.158
BaP	0.829		
PYR	0.780	0.507	
FLT	0.680		
BkF	0.459		
PCB-37	0.442		
BghiP	0.284		
PHE		0.938	
α-HCH		0.930	0.174
ANT	0.318	0.850	
γ-HCH		0.833	0.298
δ-HCH		0.811	0.197
FLE		0.691	
PCB-114		0.652	0.504
o,p'-DDE	0.382	0.571	
NPT	0.207	0.567	0.186
β-HCH		0.559	
PCB-123		0.516	
PCB-118		0.497	
ANE	0.270	0.397	
ANY	0.270	0.397	
PCB-207		0.388	
p,p'-DDE		0.386	
PCB-119		0.426	0.582
PCB-77		0.385	0.567
α-Endo			0.540
Endo-SO ₄			0.486
PCB-52	0.191		
PCB-157	0.389		
PCB-74	0.401		
Dieldrin		0.276	
β-Endo	0.220		0.384
o,p'-DDT		0.356	
p,p'-DDD		0.241	
PCB-189		0.151	
PCB-170			
PCB-151			

and Callén 2000). One site, IS-04 in Mambakkam, contributes about 40% of the above compounds. This site has diagnostic ratios FLT/(FLT + PYR) > 0.5, BaA/(BaA + CHR) > 0.35 and IndP/(IndP + BghiP) > 0.5, all indicating coal and biomass combustion (Yunker et al. 2002). Thus, high-temperature combustion and industrial processes might be the main sources of HMW PAHs. The presence of p,p'-DDT in only one site (IS-04) could be due to its vector control usage.

PC-2

PC-2 is mainly comprised of HCH isomers (α-HCH, β-HCH, γ-HCH and δ-HCH); LMW PAHs, viz. PHE, FLE, ANT, NPT; and PCB congeners, mainly dl-PCBs, namely PCB-114 and PCB-123 (*R*² > 0.5). These compounds were predominant in the RS, which contributed about 62%, 65%, 76% and 92% of NPT, FLE, PHE and ANT, respectively, close to 99% of PCB-114 and PCB-123 and close to 75% of HCH isomers. Considering that most of these LMW PAHs were from the RS, it can be hypothesized that the domestic usage of fossil fuels is the principal source. ANT is generally a marker for domestic wood combustion or diesel and gasoline exhaust gases (Valavanidis et al. 2006). Thus, LMW PAHs in soil indicate a significant presence of combustion products from low-temperature pyrogenic processes (Jenkins et al. 1996) and light-duty vehicles (Lee 2010). About 75% and 60% of the RS in Mambakkam and Cheyyar, respectively, showed ANT/(ANT + PHE) ratio > 0.1, indicating petroleum and biomass combustion as a primary source. Two RS in Cheyyar have ANT/(ANT + PHE) ratio close to 0.1, indicating mixed sources. Moreover, the sites with higher ANT/(ANT + PHE) ratios in the residential areas also showed BaA/(BaA + CHR) > 0.35, indicating biomass and coal combustion as primary sources of PAHs (Liu et al. 2010). Most of these sites also have FLT/(FLT + PYR) > 0.5, indicating grass, wood and coal combustion. Since RS are located within the SIPCOT belt, the influence of industrial operations might also be a direct source.

Prevalence of PCB-114 was previously observed in dust from Cheyyar industrial area, and it could be due to the combustion of coal and industrial waste (Chakraborty et al. 2016b; Chi et al. 2007). Cheyyar residential region (RS-07 and RS-08) was

predominantly loaded with dl-PCBs such as PCB-114 and -123. Apart from the two RS, about 2% and 11% of PCB-114 and PCB-123 were from the AS of Cheyyar, respectively. Cheyyar is a comparatively new industrial belt compared to Mambakkam. The annual growth of this industrial area is 40% in software, 20% in the automobile industry and 10% in leather products (MSME-DI 2015). Cheyyar industrial park is mainly occupied by light engineering, automotive, footwear and plastic industries. As these PCB congeners were not observed in Mambakkam SIPCOT, it can be hypothesized that these congeners are from some newer sources in the upcoming industrial area of Cheyyar. The presence of e-waste recycling activities in the vicinity is evident from the contemporary occurrence of mono-ortho PCBs such as PCB-114 and -123 in the previous study (Chakraborty et al. 2016a). Although Kanchipuram district houses eight authorized e-waste dismantling facilities and only one authorized recycling facility (TNPCB 2020), undocumented recycling activities cannot be neglected, given the history of informal e-waste recycling units in the proximity (< 15 km) of the study area (Toxics Link 2004). Open fires, cooking and heating in the residential areas may also lead to higher concentrations of dl-PCBs. It is to be noted that the higher percentage of dl-PCBs from residential areas could be related to the burning of plastics, PVC and painted wood (Chakraborty et al. 2018). These PCB congeners have also been previously detected in residential soils of the industrial city of Korba, Chhattisgarh, in India (Kumar et al. 2014). As evident from the diagnostic ratios, the presence of the majority of HCH isomers in residential areas may be due to the usage of HCH technical mixtures in vector control programmes.

PC-3

PC-3 is mainly associated with α -Endo and PCB-77 and PCB-119. The RS and AS in Cheyyar both contribute close to 50% of these PCB congeners. PCB-77 had previously been found in dust samples from the same region, probably associated with refuse burning that includes discarded electronic parts and wires (Chakraborty et al. 2016b). Compounds segregated in PC-3 were prevalent in the agricultural and residential transects. It could be hypothesized that the levels of these congeners could be influenced by unaccounted

e-waste present in the refuse within the industrial corridor. α -Endo was present only in the RS of Cheyyar (RS-06 and RS-10), probably because endo-sulfan was used in the past in agriculture, and Cheyyar was a developing industrial corridor over agricultural land.

Toxic equivalents

BaP and DBA contribute maximum to total BaP_{eq}, as evident from the equation previously stated. Total BaP_{eq} estimated in this study (0–520 ng/g) is much lower than that in the road dust of industrial (930–16,740 ng/g) and urban (4370–68870 ng/g) areas of Ulsan, Korea (Dong and Lee 2009). The mean total BaP_{eq} of the IS (75 ng/g) was slightly higher than that of the RS (64 ng/g). Mean total BaP_{eq} (52 ng/g) of all the sites combined was lower than that in the surface soils of Dalian, China (238 ng/g) (Wang et al. 2007), and various locations across Agra city (650 ng/g) (Masih and Taneja 2006). The mean total BaP_{eq} of the AS (17 ng/g) was lower than that in the agricultural soils of Delhi, India (154 ng/g) (Agarwal et al. 2009).

In this study, TEQs for dl-PCBs (0–186 fgTEQ/g) were much lower than TEQs observed in the settled dust across urban and suburban areas of Chennai city (0–2800 fgTEQ/g) (Chakraborty et al. 2016b) and TEQs in soil samples from informal e-waste recycling sites (0–129,000 fgTEQ/g) and dumpsites (0–122,000 fgTEQ/g) of Indian cities, including Chennai (Chakraborty et al. 2018). TEQs for dl-PCBs in Mambakkam (mean \pm SD, 43 \pm 33 fgTEQ/g) and Cheyyar (40 \pm 56 fgTEQ/g) were comparable. PCB-157 (mean \pm SD, 19 \pm 25 fgTEQ/g) contributed maximum TEQ followed by PCB-189 (12 \pm 17 fgTEQ/g). Current observations are severalfold lower than the industrial contamination in mixed urban agricultural soil in north-west Italy (14,600–100,000 fgTEQ/g) (Turrio-Baldassarri et al. 2007). TEQs for dl-PCBs were in the following order: RS (mean \pm SD, 55 \pm 58 fgTEQ/g) > IS (44 \pm 27 fgTEQ/g) > AS (25 \pm 44 fgTEQ/g). TEQs were significantly lower than the TEQs in soils from South Africa (800–16,000 fgTEQ/g) (Nieuwoudt et al. 2009). Current TEQs were also lower than those in the residential soils from Chhattisgarh, India (mean 12,500 fgTEQ/g) (Kumar et al. 2014).

Conclusions

This is the first study investigating the contamination of legacy POPs (OCPs, PCBs) and PAHs in soil concurrently from the industrial belt in the suburbs of Chennai city under SIPCOT. PAH and OCP concentrations were in the order residential > industrial > agricultural sites, while PCB concentrations were in the order industrial > residential > agricultural sites. Major sources of PAHs included industrial, vehicular emission and biomass burning, while OCP residues were due to past use in agricultural activities and ongoing use for vector control. In addition, sources of PCBs were related to informal e-waste recycling and refuse burning. Lack of correlation between PAHs, PCBs and OCPs and TOC in soil reflects recent contamination. Some OCPs, such as HCHs, indicate ongoing usage despite their ban. TEQ values showed a low human health risk due to PAHs and dl-PCBs in the study area.

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