

Persistent Organochlorine Pesticides and Polychlorinated Biphenyls in Intensive Agricultural Soils from North India

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Abstract: Organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) were determined in agricultural soils from the northern states of India. The average concentration of OCPs was 37.67 ± 0.33 ng/g (dry weight – DW) while HCHs alone accounted for 93% followed by DDT (4.27%) and endosulphan (2.51%). The α/γ ratio of HCH (< 0.01 –8.64) reflects the use of technical as well as lindane formulations. The ratio of p,p' -DDT/ p,p' -DDE (0.16) and o,p' -DDT/ p,p' -DDT (< 0.01) indicates the contamination of soils with the past use of technical DDT. The mean concentrations of endosulphan and dieldrin were 0.95 ± 0.53 ng/g (DW) and 0.16 ± 0.07 ng/g (DW), respectively. The average concentration of PCBs was 13.44 ± 0.06 ng/g (DW). The toxic equivalency (TEQ) calculated using WHO 2005-TEFs ranged from 0.01 to 105.40 pg WHO 2005-TEQ/g (DW) with the mean of 13.78 ± 0.11 pg WHO 2005-TEQ/g (DW). PCB-105 (25%), PCB-114 (18%), and PCB-118 (18%) were the dominant congeners and accounted for 61% while a non ortho PCBs contributed only 18% to total DL-PCBs. The contamination of soils is a matter of concern but is not alarming because the observed levels were lower than those given by the Canadian soil quality guidelines.

Keywords: accelerated solvent extraction (ASE); agricultural soil; organochlorine pesticides (OCPs), polychlorinated biphenyls (PCBs)

Widespread attention has long been given to the pollution by persistent organochlorine pesticides and polychlorinated biphenyls. Pesticides and polychlorinated biphenyls (PCBs) are ubiquitous chemicals and can persist in soils for decades (HUSSEN *et al.* 2007; SHEGUNOVA *et al.* 2007). Their characteristics, including hydrophobicity and resistance to degradation, enable them to accumulate in soils, sediments, biota (COVACI *et al.* 2005; PANDELOVA *et al.* 2008; HAO *et al.* 2009; SHELEPCHIKOV *et al.* 2009), and in human body through dietary intake, inhalation, and other indirect exposure (NANDITA *et al.* 2009).

Organochlorine pesticides and PCBs are long-range transport pollutants and can be transported to regions far from their original sources, such as the Arctic (IWATA *et al.* 1994; HALSALL *et al.*

1998). These organochlorine compounds have a wide range of acute and chronic health effects, including cancer, neurological damage, reproductive disorders, immune suppression, birth defects, and are also suspected endocrine disruptors (VAN DEN BERG *et al.* 2006; WANG *et al.* 2008).

The main reservoirs where POPs including PCBs ultimately accumulate are soils and sediments (CLAUDINE *et al.* 2009). The fate of organochlorine pollutants in soils with different cropping land use has been extensively studied in many countries. The concentrations of these pollutants were observed in many agricultural soils worldwide (KUMARI *et al.* 2008; LIU *et al.* 2009; SENTHILKUMAR *et al.* 2009; HOAI *et al.* 2010; AL-WABEL *et al.* 2011). Previous investigations dealing with various environmental matrices in India revealed OCPs and

PCBs contaminations in air, water sediment, and biota (OM PRAKASH *et al.* 2004; IPEN 2006; ZHANG *et al.* 2008; CHAKRABORTY *et al.* 2010).

However, the investigation into on organochlorine contamination in the intensive agricultural area in National Capital Region, Delhi of North India still remains to be studied. In the present study persistent organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) were determined using accelerated solvent extraction (ASE) and GC-ECD.

respectively. National Capital Territory (NCT), Delhi, accounts for 1483 km², area of the NCR Delhi region. The cultivated land occupies a large proportion of 79.53% area in the NCR, Delhi region. The main crops grown in Delhi area are wheat, mustard, sugarcane, maize, millet (jawar), Bajra, paddy, and commercial agricultural crops such as vegetables, flowers, mushrooms, etc.

Sampling

MATERIAL AND METHODS

Description of study area

The study area, National Capital Region (NCR), Delhi, spreads over an area of around 30 242 km². Out of this, the Haryana, Uttar Pradesh and Rajasthan account for 13 413, 10 853, and 4493 km²,

The sampling locations of the agricultural field of NCR, Delhi, are shown in Figure 1. The sampling locations were selected in the agricultural area with different cropping pattern. 49 samples were randomly collected in duplicate during April to December, 2009. Approximately 1 kg of the soil sample was collected using a stainless steel auger and, after removing pebbles and wood sticks, the

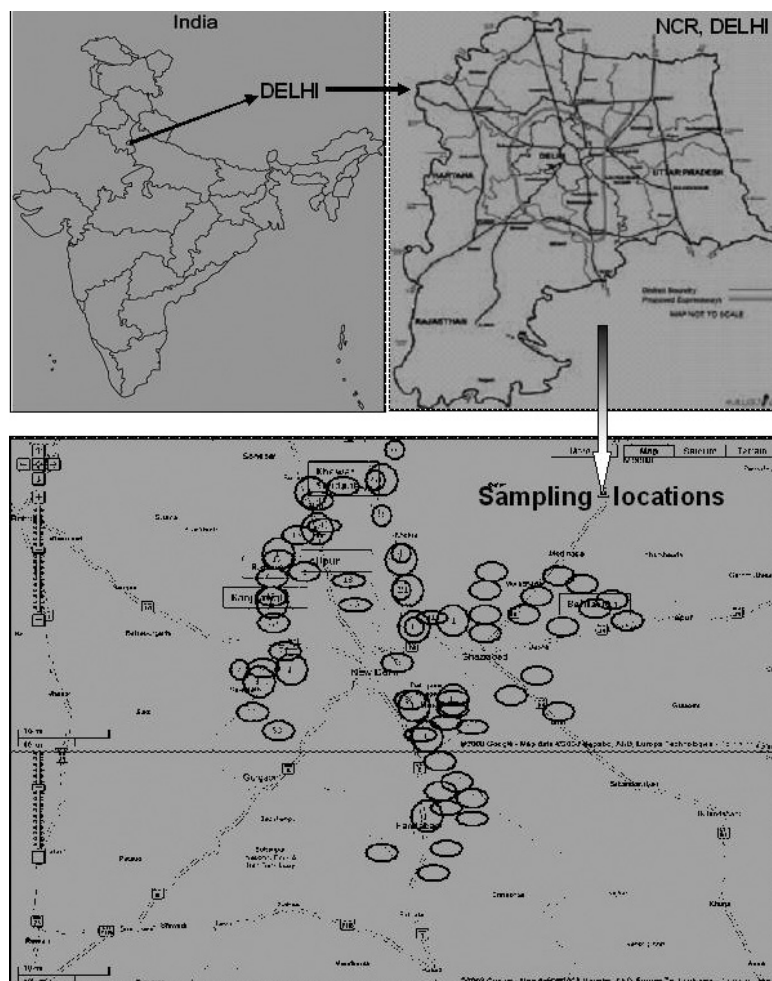


Figure1. Map showing sampling locations in National Capital Region, Delhi

sample was thoroughly homogenised, then an aliquot was transferred to a clean wide mouth amber glass bottle. After proper labelling, the sample bottles were transported to the laboratory and kept at -4°C until further chemical treatment.

Sample extraction and clean-up

The soil samples were extracted for OCPs and PCBs analysis using the extraction procedure as per SW-846 Method 3545 (USEPA 1995). Briefly, 10–15 g of the wet sample was homogenised and dried by mixing with diatomaceous earth until a free-flowing powder was obtained. The extraction was carried out with an accelerated solvent extractor (Dionex, Sunnyvale, USA) using acetone: hexane (v/v, 1:1) in two cycles with 5 min static time. The ASE was operated at 1500 psi and the oven was heated to 100°C . The extracts were concentrated to 2.0 ml using a Rotatory Vacuum evaporator (Eyela, Tokyo, Japan). The moisture content was determined to report the data on the dry weight basis.

The multilayered silica gel column chromatography was performed to remove the interfering organic and polar species. PCBs and *p,p'*-DDE were eluted as the first fraction with hexane. Other pesticides were eluted as the second fraction with 20% (v/v) dichloromethane in hexane (VIET *et al.* 2000). The eluted extract was concentrated using the Rotatory Vacuum evaporator and under gentle stream of pure nitrogen using Turbo Vap (Caliper, Hopkinton, USA) to 1.0 ml. The extract was transferred to an auto sampler vial and 1 μl was injected onto a gas chromatograph equipped with an electron capture detector (GC-ECD) for quantification.

Sample analysis and quality control

The separation and quantification of OCPs and PCBs were carried out using Gas Chromatograph attached to an auto sampler and equipped with an Electron Capture Detector (ECD, ^{63}Ni). Elite-1 and HP-5MS columns were used during the chromatographic separation of OCPs and PCBs, respectively.

Certified reference standards from Sigma-Aldrich (Sleeze, Germany) and from Dr. Ehrenstorfer (GmbH, Augsburg, Germany) were used for the quantification of OCPs and PCBs, respectively. The analytes were identified in the sample extract by comparing the retention times of the standard

mixture and quantified using the response factors obtained from five level calibration curves of the standards. Appropriate quality assurance quality control (QA/QC) analysis was performed. Each sample was analysed in duplicate and the average was used in the calculations.

Toxic equivalent quotients (TEQ) for dioxin-like PCBs were calculated by multiplying the concentration of the individual DL-PCB congener with the corresponding toxicity equivalent factors (TEFs) (VAN DEN BERG *et al.* 2006).

The results of the analysis are reported in ng/g dry weight (DW) basis. A reporting limit of $> 0.01 \mu\text{g/kg}$ was taken for the calculation and the values below MDL ($< 0.01 \text{ ng/g}$) were taken as zero (0) in the calculations.

Soil quality guidelines for environmental health

Pesticides and PCBs in soil originate primarily from the application of wastes to the land, particulate deposition, wet deposition, and also from the application of municipal sewage sludges contaminated at trace levels (WEBER & WANG 1995). The adsorption to soil particles is thought to be hydrophobic sorption, which is the partitioning of a nonpolar solute from the polar aqueous phase onto the hydrophobic surfaces of the earth materials (GAN & BERTHOUEX 1994).

Soil quality guidelines (SQGs) are usually based on the soil contact toxicity with plants and invertebrates leading to bioaccumulation and biomagnifications in the food chain. For agricultural lands, the SQGs, which are based on the soil and food ingestion guidelines, are 0.7, 0.01, and $0.50 \mu\text{g/g}$ for total DDT, lindane, and PCBs, respectively (CCME 1999). No maximal values are specified for endosulphans. Since, India does not have specified guidelines for OCPs and PCBs in agriculture soils, the observed concentrations of OCPs and PCBs in this study were compared with the guideline values and were found to be lower than those values.

RESULTS AND DISCUSSION

Organochlorine pesticides (OCPs)

The analytical results for organochlorine pesticides are presented (Table 1). The average con-

centration of OCPs in agricultural soils was 37.67 ± 5.88 ng/g DW. Among the OCPs, HCHs alone amounted to 92.79% and were the dominant pollutants with 34.96 ng/g (DW) mean concentration, followed by DDTs (1.61 ng/g DW), endosulphan (0.95 ng/g DW), and dieldrin (0.16 ng/g DW), respectively. Among HCHs isomers, α -HCH isomer was the predominant contaminant in all the pesticide compounds. The composition of HCH isomers in the present study revealed $\alpha = 69\%$; $\beta = 7\%$; $\gamma = 12\%$; $\delta = 5\%$ (Table 1) which was similar to the technical grade composition, showing the technical HCH use in the study area. The Σ HCH concentrations ranged between 3.59–157.02 ng/g DW, with a mean of 34.96 ± 5.87 ng/g DW. In this study, the ratio of α -HCH to γ -HCH isomers (α/γ ratio) ranged between < 0.01 and 8.64, with the pooled mean value of 3.59. The ratio reflects the use of technical HCH as well as the lindane formulation. Similar ratios of α/γ were reported for Indian environment by ZHANG *et al.* (2008), KUMAR *et al.* (2008), and CHAKRABORTY *et al.* (2010). The technical mixture of HCH was produced and used in India until it was banned in 1997, and, lindane formulations are registered

for use in public health practices to control the vector borne diseases and for the pest control in selected crops.

Since 1989, DDT has been banned in India for use in the agricultural sector but it is permitted for use up to 10 000 t/year under the Stockholm Convention, until an alternative can be found to combat vector borne diseases which are particularly prevalent in the monsoon season (PAPIYA 2004). Elevated concentrations of p,p' -DDT (0.87 ng/g DW) and p,p' -DDE (0.71 ng/g DW) as compared to o,p' -DDT (0.03 ng/g DW) were observed in the analysed agricultural soils (Table 1). The higher concentration of p,p' -DDT, and concurrently lower levels of p,p' -DDE were observed at all the sampling locations which reflects the use of aged DDT mixture. In this study, the ratio of p,p' -DDT/ p,p' -DDE was 0.16 (mean) indicating inputs of an aged mixture in the study area. o,p' -DDT/ p,p' -DDT ratio in this study (less than unity), is different from the scenario in China where dicofol use is a major source of DDT (LIU *et al.* 2006). Thus, the DDT contaminations in this region may come from technical DDT and obviously not from dicofol use.

Table 1. Range and mean $\pm$ SE (standard error)* concentrations (ng/g DW) of organochlorine pesticides in agricultural soils from north India ($n = 49$)

Congener name	Range		Mean $\pm$ SE	%
	minimum	maximum		
α -HCH	3.10	121.89	26.14 ± 4.43	69.39
β -HCH	< 0.01	20.32	2.72 ± 0.57	7.22
γ -HCH	< 0.01	22.36	4.35 ± 0.84	11.54
δ -HCH	< 0.01	9.17	1.75 ± 0.28	4.65
Σ HCH	3.59	157.02	34.96 ± 5.87	92.79
Aldrin	< 0.01	< 0.01	< 0.01	< 0.01
Dieldrin	< 0.01	2.07	0.16 ± 0.07	0.43
α -Endosulphan	< 0.01	6.45	0.18 ± 0.14	0.49
β -Endosulphan	< 0.01	20.48	0.76 ± 0.43	2.03
Σ Endosulphan	< 0.01	22.99	0.95 ± 0.53	2.51
p,p' -DDE	< 0.01	11.97	0.71 ± 0.27	1.88
o,p' -DDT	< 0.01	1.46	0.03 ± 0.03	0.08
p,p' -DDT	< 0.01	4.58	0.87 ± 0.18	2.31
Σ DDTs	< 0.01	11.97	1.61 ± 0.33	4.27
Σ OCPs	3.59	160.30	37.67 ± 5.88	100

< 0.01 = below detection limit; *SE = $SD/\sqrt{n}$

India is one of the major producers of endosulphan, with an average annual consumption of 3600 t from 1995 to 2000 (USHA *et al.* 2005). Endosulphan alone accounts for over 10% of the total insecticide consumption in India. In the present study, the sum of endosulphan ranged from < 0.01 to 22.99 ng/g DW, with the mean of 0.95 ± 0.53 ng/g DW. The α - and β -isomers of endosulphan were detected in the selected soil samples. Average values of α -endosulphan and α -endosulphan isomers were found to be 0.18 ± 0.14 ng/g DW and 0.76 ± 0.43 ng/g DW, respectively (Table 1). Since β -endosulphan is less soluble in water and more strongly bound to the soil particles than α -isomer (BEYERS *et al.* 1965), α -isomer is carried by run off to the aquatic environment. Thus the presence of β -endosulphan seems to be a useful indicator of the time period since the exposure to endosulphan.

Polychlorinated biphenyls (PCBs)

The concentrations (range and mean) of PCBs, DL-PCBs, and Σ PCBs in the soils are presented in Table 2. The average concentration of Σ PCBs + Σ DL-PCBs was 13.44 ± 0.06 ng/g and the range was between < 0.01 – 99.40 ng/g (DW). The DL-PCBs contributed 20% (average, 6.26 ± 0.03 ng/g) to the total PCBs, and the contribution by other DL-PCBs was 80% (average, 18.33 ± 0.08 ng/g) to the total PCBs contamination. Comparatively, higher concentrations of PCBs were detected in the samples from the vicinities of industries. This indicates that PCB has been used in these industries and found its way to the environments. Among the studied PCB congeners, PCB-74 manifested the highest concentration (2.03 ± 0.18 ng/g), followed by PCB-151 (0.85 ± 0.19 ng/g), PCB-37 (0.77 ± 0.15 ng/g), PCB-187 (0.53 ± 0.09 ng/g), PCB-18 (0.46 ± 0.15 ng/g), and PCB-52 (0.45 ± 0.17 ng/g); other congener concentrations were comparatively low (< 0.3 ng/g DW). The results of this study are in agreement with the observations by AICHNER *et al.* (2007) for Kathmandu soils, and the soil from Tibetan Plateau, China, by WANG *et al.* (2009). However, the concentrations of PCBs were lower than those of PCBs in soils from Vietnam (VIET *et al.* 2000; KISHIDA *et al.* 2007), Romania (DOINA *et al.* 2006), China (LIU *et al.* 2009), San Felipe, Nuevo Mercurio, Zacatecas, Mexico (ROGELIO *et al.* 2011) but higher than in soils from Turkey (SALIHOGU *et al.* 2011).

Table 2. Range and mean $\pm$ SE (standard error)* of Σ PCBs concentrations (ng/g DW) in agricultural soils from Delhi region, India

Σ PCBs + Σ DL-PCBs	
Range	mean $\pm$ SE
< 0.01 – 99.40	13.44 ± 0.06
Σ DL-PCBs (pg WHO ₂₀₀₅ -TEQ g ⁻¹ DW)	
Range	mean $\pm$ SE
0.01 – 105.40	13.78 ± 0.11

< 0.01 = below detection limit, *SE = $SD/\sqrt{n}$

The concentrations of DL-PCBs in this study ranged from 0.37 ± 19.09 ng/g (DW). Total TEQ levels of DL-PCBs ranged from 0.01 to 105.40 pg WHO 2005-TEQ/g (DW) with the mean of 13.78 ± 0.11 pg WHO 2005-TEQ/g (DW) (Table 3). Mono ortho PCB-105 (25%), PCB-114 (18%) and PCB-118 (18%), were the dominant congeners and accounted for 61% while, on the other hand, non ortho PCBs contributed only 18% to total DL-PCBs. Congener specific concentrations of DL-PCBs in the Delhi region were in agreement with those given by ZHANG *et al.* (2007), LIU *et al.* (2009), CLAUDINE *et al.* (2009). The observed concentrations of Σ DL-PCBs in agricultural soils from North India were lower than those from the Tailake region and Southern Jiangsu region of China (ZHANG *et al.* 2007; WANG *et al.* 2010). Municipal solid waste incinerations typically release PCB-118 into the atmosphere, however, the emissions from coal combustion and industrial waste incineration sources contributed non ortho PCBs (DYKE *et al.* 2003).

It seems likely that the main source of DL-PCBs in the soils of agricultural fields from north India is the open biomass burning which is common in agricultural fields after crop harvesting, and the depositions of emissions from wood processing, paint and dying materials, chemicals and transformer manufacturing units, and electrical and electronic waste recycling units. These PCB sources also include off gassing from closed systems such as older equipment (e.g. transformers that contain large quantities of PCB fluids), and PVC (polyvinylchloride) manufacture.

The analytical data indicates fresh use of technical DDT but no dicofol type Inputs; however, besides lindane, technical HCH mixtures are also used in the study area. The PCBs contamination of soils is a matter of concern but it is not alarming because

Table 3. Range and mean $\pm$ SE (standard error)* of PCB congeners concentrations (ng/g DW) in agricultural soils from Delhi region, India

PCBs congeners			DL-PCBs congeners		
No.	range	mean $\pm$ SE	No.	range	mean $\pm$ SE
PCB-18	< 0.01–5.56	0.46 $\pm$ 0.15	PCB-77	< 0.01–1.94	0.11 $\pm$ 0.04
PCB-37	< 0.01–3.81	0.77 $\pm$ 0.15	PCB-81	< 0.01–2.09	0.12 $\pm$ 0.06
PCB-44	< 0.01–0.91	0.10 $\pm$ 0.03	PCB-105	< 0.01–1.54	0.39 $\pm$ 0.05
PCB-49	< 0.01–1.08	0.08 $\pm$ 0.03	PCB-114	< 0.01–1.73	0.27 $\pm$ 0.07
PCB-52	< 0.01–5.49	0.45 $\pm$ 0.17	PCB-118	< 0.01–0.99	0.27 $\pm$ 0.03
PCB-70	< 0.01		PCB-123	< 0.01–0.25	0.01 $\pm$ 0.01
PCB-74	< 0.01–5.01	2.03 $\pm$ 0.18	PCB-126	< 0.01–0.17	0.02 $\pm$ 0.01
PCB-119	< 0.01		PCB-156	< 0.01–0.80	0.08 $\pm$ 0.02
PCB-128	< 0.01–0.95	0.10 $\pm$ 0.04	PCB-157	< 0.01–0.28	0.02 $\pm$ 0.01
PCB-138	< 0.01–1.44	0.15 $\pm$ 0.03	PCB-167	< 0.01–1.07	0.15 $\pm$ 0.03
PCB-151	< 0.01–4.64	0.85 $\pm$ 0.19	PCB-169	< 0.01–0.51	0.04 $\pm$ 0.02
PCB-168	< 0.01–0.75	0.02 $\pm$ 0.02	PCB-189	< 0.01–0.36	0.06 $\pm$ 0.01
PCB-170	< 0.01–0.76	0.17 $\pm$ 0.03	–	–	–
PCB-177	< 0.01–3.17	0.37 $\pm$ 0.10	–	–	–
PCB-187	< 0.01–2.86	0.53 $\pm$ 0.09	–	–	–
PCB-207	< 0.01–1.02	0.08 $\pm$ 0.03	–	–	–
Σ PCBs	< 0.01–99.40	18.83 $\pm$ 0.08	Σ DL-PCBs	0.37–19.09	6.26 $\pm$ 0.03

< 0.01 = below detection limit, *SE = SD/ $\sqrt{n}$

the observed levels were lower than those given by the soil quality guidelines. It is recommended that more intensive assessment of national Capital Region for persistent organic pollutants (POPs) be conducted, due to the human health and environment concerns.

Acknowledgements. The authors express their sincere gratitude to the Member Secretary and Chairman of Central Pollution Control Board, Ministry of Environment & Forest Government of India for enabling to conduct the study.

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Received for publication March 29, 2011
Accepted after corrections August 2, 2011

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