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Residues of Pesticides and Herbicides in Soils from Agriculture Areas of Delhi Region, India

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Abstract

This paper presents the residue levels of organochlorine, organophosphate pesticides and herbicides in agricultural soils from Delhi region. Among OCPs, HCH, DDT endosulphan and dieldrin ranged between <0.01-104.14 ng g^{-1}, <0.01-15.79 ng g^{-1}, <0.01-7.57 ng g^{-1} and <0.01-2.38 ng g^{-1}, respectively. The concentration of OPPs ranged from <0.01-20.95 ng g^{-1}, ND-3.92 ng g^{-1}, ND-31.73 ng g^{-1}, ND-6.46 ng g^{-1} and ND-6.46 ng g^{-1} for phosphomidon, monocrotophos, chlorpyriphos, quinolphos and ethion, respectively. Pendimethalin (0.27 ng g^{-1}) was the dominant herbicides followed by butachlor (0.19 ng g^{-1}), and fluchloralin (0.05 ng g^{-1}). Data showed the region was contaminated by technical DDT and technical HCH mixture. The study reveals that the level of some organochlorine pesticides in agricultural soils is a matter of concern for future food chain accumulation and human health so; regular investigation of pesticide residues is recommended on soil health and contamination levels.

Keywords: pesticides, herbicides, agricultural soil, Delhi, India

1. Introduction

According to the Food and Agriculture Organization (FAO), agricultural lands comprise 50% of all useable land worldwide (FAO 2001). About 60 percent population in India is involved in agricultural sector which contributed to a very important part of Indian economy. Several pesticides including organochlorine, organophosphate, carbamate, fungicides, herbicides and synthetic pyrethroids are used in modern agricultural production to meet the need for abundant, safe and affordable food and fiber. Although the use of pesticides has led to increased agricultural production but, their use has also been associated with several concern, including risk to human health and environment (Ejaz *et al*. 2004). These compounds have a wide range of both acute and chronic health effects, including cancer, neurological damage, reproductive effects, immune suppression, birth defects, and are also suspected endocrine disruptors (Calvert *et al*. 2001; Wang *et al*. 2008).

During the last few decades, widespread contamination and toxic effects of organic chemicals have becomes a serious environmental problem. They enter the soil by direct treatment or being washed off from the plant surface during rainfall. Their physico-chemical characteristics, which include hydrophobicity and resistance to degradation, make these chemicals to accumulate in soils and sediments Hong *et al.* 2008; Hu *et al.* 2010). Soil and sediments can act as a contributor of organic pollutants to the atmosphere, especially of semi volatile compound in warm climates. The fate of pesticides in soils with different cropping land use has been extensively studied worldwide including India (Pillai 1986; Viet *et al.* 2000; Om Prakash *et al.* 2004; Oldal *et al*. 2006; Shegunova *et al.* 2007; Senthil Kumar *et al.* 2009).

In India, there are 165 pesticides registered for use and there is a sequential rise in the production and consumption of pesticides during last three decades. India is the fourth largest pesticide producer in the world after the US, Japan and China. During 2003-2004, the domestic production of pesticides was approximately 85 TMT (thousand metric tons), and about 60 TMT used annually (Anonymous 2005), against 182.5 million hectare of land where 70% accounts for DDTs, HCHs and organophosphate pesticides Bhattacharyya *et al*. 2009; Nirula and Upadhyay 2010). The domestic consumption of pesticides in agriculture is comparatively low (0.5 kg/ha), (only 3.75% of global consumption) against 12.0, 7.0, 6.6, and 3.0 kg.ha^{-1} in Japan, USA, Korea and Germany, respectively (Chauhan and Singhal 2006). The present study was undertaken to investigate the residue of organochlorine (OC), organophosphate (OP) pesticides and herbicides, in agriculture soils of national capital region (NCR) Delhi, India.

2. Materials And Methods

2.1 Description of study area

The National Capital Region (NCR), Delhi comprises by the National Capital Territory (NCT) Delhi and the delineated area of the surrounding states of Haryana, Uttar Pradesh, and Rajasthan. The entire NCR Delhi region spread over an area of around 30,242 sq. kms. The main crops grown in Delhi area are wheat, mustard, sugarcane, maize, jawar, bajra, paddy and commercial agricultural crops, such as, vegetables, flowers, mushroom etc. Sampling locations were in Uttar Pradesh (Ghaziabad, Guatam Budh Nagar & Bagpat district), Haryana (Sonepat, Faridabad & Ballabhgarh district) and in Delhi (Alipur Block, Kanjhawala Block, Najafgarh & Nizamuddin Yamuna Bridge area).

2.2 Sampling

Agricultural area with different cropping pattern was selected for the study of organochlorine, organophosphate pesticides and herbicides in soils. 50 samples were collected during 2009 from different locations in National Capital Region Delhi, India. Approximately 1 Kg. soil of upper layer collected using stainless steel auger in clean wide mouth amber glass bottle. 4-5 samples from each location were thoroughly mixed to ensure that the soil collected was truly representative of each location. Sub-samples of the soil were subsequently taken and stored in labeled wide mouth amber glass bottles and transported ice-preserved to the laboratory and kept at -20^{0}C until further chemical processing.

2.3 Extraction and cleanup

Ground sieved soil sample (15-20 g) was thoroughly mixed in a glass beaker with 0.5 g activated charcoal, 0.5 g Florisil and 10 gm anhydrous Sodium Sulphate. The mixed sample was packed compactly in glass column (25x250 mm) in between two layers of anhydrous sodium sulphate. The elution of pesticide residues were carried out with 150 ml of Hexane-Acetone mixture (1:1v/v) at a flow rate of 2-3 ml/min. The elute was concentrated to 2.0 ml using Rotatory Vacuum evaporator (Eyela, Japan) and finally divided into two portions 1.0 ml for OCP and OPP analysis by GC and 1.0 ml was solvent exchanged to acetonitrile for herbicide determination by HPLC.

2.4 Instrumental analysis

Separation and Quantification of organochlorines (OCPs) was carried out using GC (Perkin Elmer, Clarus 500) with autosampler equipped with an Electron Capture Detector (ECD, ^{63}Ni), on fused silica column 25 m x 0.20 mm id Elite-1 (0.33 µm particle of 5% diphenylpolysiloxane and 95% dimethylpolysiloxane). The column oven temperature was initially maintained at 170^o C and programmed to 220^{o}C (7^{0}C min^{-1}) and again ramped to 250^{o}C at 5^{o}C min^{-1} and held for 6.86 min. The injector and detector temperature were maintained at 250^{o}C and 350^{o}C respectively. Purified nitrogen gas was used as carrier at the flow rate of 1.0 ml/min.

The organophosphate pesticides (OPPs) were separated and quantified by Shimadzu 2010 series gas chromatograph equipped with auto sampler and Flame Photometric Detector (FPD). A fused silica capillary column (J&W Scientific) of 30mm x 0.25mm id with 0.25 µm particle coating (14% cyanopropylphenyl and 86% dimethylpolysiloxane) was used for all the separation. The column oven temperature was performed as: initial temperature was 150^{o}C, and increased to 220^{0}C at the rate of 10^{o}C min^{-1} then ramped to 260^{o}C at the rate of 15^{o}C min^{-1}, keeping the final temperature for 4 min. The carrier gas was nitrogen gas at the flow rate 1.2 ml.min^{-1}. The injector and detector temperature were maintained at 250^{o}C and 290^{o}C respectively.

Separations of herbicides were performed with HPLC system (Agilent 1100 Series) using UV Diode Array Detector (DAD, λ=210 nm). Isocratic mobile phase was Acetonitrile: water (80:20 v/v) at flow rate of 0.5 ml.min^{-1}. Extract injection with 20 µl sample loop were chromatographed on a 150 mm x 4.6 mm id, C18 reversed phase *ZORBAX* eclipse XDB (Agilent) column with 5.0 µm particle.

2.5 Analytical quality control

Certified reference standards (Sigma Aldrich ,USA) were used for calibration of the instruments. The concentrations of analyte were determined by comparing the peak area of the samples and five level calibration curves of the standards. The correlation coefficient of calibration curves were ranged from 0.9980 to 0.9990. The peak identification was conducted by the accurate retention time of each standard. Appropriate quality assurance/ quality control (QA/QC) analysis was performed including a method blank processing along with the samples to check any loss or cross contamination during the sample processing (analyte concentrations were <MDL 'method detection limit'), random duplicate samples (Standard deviation <10), calibration curves with the r^2 value of 0.999, and matrix spike recovery 100±22%. The recoveries assumed to be satisfactory and the results were not corrected for the recovery. Each sample was analysed in duplicate and the average was used in calculations. The results of the analysis are reported in µg kg^{-1} wet -weight (wet wt.) basis. A reporting limit of > 0.01 µg kg^{-1} wet wt was taken for calculation. Levels below reporting limit or below MDL (<0.01 µg kg^{-1} wet wt) were taken as zero (0) in the calculations.

3. Results And Discussion

The observed concentration profiles of ∑OCPs, ∑OPPs and ∑herbicides in soils from national capital region of India are presented in Table 1. The pattern of contamination was observed as OCPs > OPPs > herbicides. The total concentrations of studied pesticides were ranged <0.01-104.14 ng g^{-1} (mean 14.04 ng g^{-1}), <0.01-31.73 ng g^{-1} (mean 1.68 ng g^{-1}) and <0.01-1.46 ng g^{-1} (mean 0.21 ng g^{-1}) for organochlorine, organophosphate and herbicides, respectively.

Among the OCPs, HCHs alone 89% were the dominant pollutants with a mean of 12.48 ng g^{-1} concentration followed by DDTs (1.15 ng g^{-1}), endosulphan (0.27 ng g^{-1}) and dieldrin (0.14 ng g^{-1}). α-HCH is the predominant contaminant among all organochlorine isomers followed by γ-HCH, *p,p'*-DDE, δ-HCH, β-endosulphan and β-HCH.

It has been widely recognized that HCH is available in two formulations: technical HCH and lindane. Technical HCH contains isomers in the following percentage: α, 55-80%; β, 5-14%; γ, 8-15%; δ, 2-16%; ε, 3-5% (Nhan *et al.* 2001; Fu *et al.* 2001; Qui *et al.* 2004), and Lindane contains >90% of γ-HCH. The composition of HCH isomers in the present study reveals α=65.29%; β=4.37%; γ=14.40% and δ=4.82% (Table 2) is similar to the technical grade composition, which reflects the technical HCH usage in this area. The ∑HCH concentrations ranged between <0.01 to 104.14 ng g^{-1} with mean of 12.48 ng g^{-1}. The ratio of α–HCH to γ–HCH has been used to identify the possible HCH source. The ratio of α-HCH to γ-HCH between 3 and 7 is indicative of fresh input of technical HCH (Yang *et al.* 2008). However, a lindane source will show the reduced ratio close or <1 (Willet *et al.* 1998). In this study the ratio of α-HCH to γ-HCH isomers (α/γ ratio) ranged <0.01 to 8.83 with pooled mean value of 4.0. This reflects the regular usage of technical HCH and lindane in this area. It is to note that, in the mixture of technical HCH and lindane application, a significant correlation between α–HCH and γ–HCH was observed in the soils (Figure 1). Moreover, the α–HCH in higher concentration indicated a fresh source of α–HCH, to the past application of technical HCH. The technical mixture of HCH produced and used in India until it was banned in 1997, and lindane formulation are registered for use in public health practices to control vector borne diseases and for pest control in selected crops (Gupta 2005; Zhang *et al.* 2008; CAPE 2005).

Since 1996, DDT was banned as an agricultural pesticide (Battu *et al.* 2004); however, nearly 85% of the DDT produced in India is used for public health practices for residual spray (Sharma 2003). The possible sources of DDTs are the combined effect of past and ongoing use in vector control or from sprays on open dumping sites. The concentration of DDT isomers was, *p,p*'-DDE (0.61 ng g^{-1}), *o,p'*-DDT (0.08 ng g^{-1}) and *p,p'*-DDT (0.47 ng g^{-1}) (Table 2). The relatively high concentration of DDE than DDT in this study area indicated that there was maximum degradation of DDT or there has been volatilization of DDT. The ratios between the parent compound of DDT and DDE can be used to identify the possible sources in the environment.

The vapor pressure of *o,p'*-DDT is 7.5 times greater than *p,p'*-DDT, and *p,p'*-DDT metabolize much faster in soils (Talekar *et al*. 1977). In the present study the amount of *p,p'*-DDT volatilized from the soil surface may be relatively small compared to *o,p'*-DDT. After the DDT applications were discontinued, much of the DDT may be converted to *p,p'*-DDE (Baxter 1990). Higher concentration of *p'p*-DDE has been interpreted as a result of DDT conversion to *p,p'*-DDE by UV radiation after prolong exposure in the environment (Atlas and Giam 1988). The residence time of *p,p'*-DDT could be estimated using the ratio of *p,p'*-DDT to ∑DDTs. The *p,p'*-DDT/∑DDTs ratio for technical DDTs was reported to be 0.77 (WHO 1989). The mean ratio of *p,p'*-DDT to ∑DDTs in present study was 0.44, which indicates that these areas have not been sprayed with DDTs more recently. The ratio of *p,p'*-DDT and *p,p'*-DDE can be used to estimate whether recent inputs of technical DDT exist. Generally, a ratio of 0.33 or less is considered as aged mixture, while a relatively high *p,p*'-DDT/*p,p'*-DDE ratio implies a recent input. In this study the ratio of *p,p'*-DDT/*p,p'*-DDE was 0.22 (mean) so, it is anticipated that the inputs of aged mixture of DDTs existed in the study area. The ratio of *o,p'*-DDT/*p,p'*-DDT can be used to distinguish technical DDT from "Dicofol-type DDT". The *o,p'*-DDT/*p,p'*-DDT ratio was reported to be 0.2~0.26 in technical DDT and ~7.5 in Dicofol products (Qui *et al.* 2005). In our study the ratio of *o,p'*-DDT/*p,p'*-DDT was 0.25, against China where dicofol usage is a major source of DDT (Liu *et al*. 2006). Thus the DDTs contaminations in this region were from usage of aged technical DDT mixture and obviously not from Dicofol type DDT.

The endosulphan production in India is approximately 8000 t annually (Chakraborty *et al*. 2010). Endosulphan alone accounts for over 10% of the total insecticide consumption in India. Endosulphan consists in two isomers, α and β, in the ratio of 7:3. Endosulphan is not considered as environmentally persistent compound (WHO 184). However, it is toxic to aquatic organisms particularly fishes (USEPA 1980) and classified as a class II component (moderately hazardous) by the World Health Organization. In the present study the sum of endosulphan ranged from <0.01 to 7.57 ng g^{-1} with the mean of 0.27 ng g^{-1} (Table 2). α- isomer of endosulphan was not detected at all the locations while β-endosulphan was detected at selected locations with the mean concentration of 0.27 ng g^{-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 time since exposure to endosulphan (Barbara *et al.* 1995). Similarly contamination of soils by drins cannot be ignored, where aldrin was absent at all locations and dieldrin was found to be 0.14 ng g^{-1}.

Ten commonly used organophosphate pesticides (OPPs) were analyzed in soils of Delhi region. The total OPP was ranged between <0.01 to 31.73 ng g^{-1} (dry wt.) with the mean concentration 1.68 ng g^{-1} (Table 3). Among OPPs, Chloropyriphos, phosphomidon, quinolphos, ethion and monocrotophos were the most frequent detected pesticides. Phorate, dimethoate and profenophos were not detected in any of the fifty samples analyzed. Phosphomidon (10.47 ng g^{-1}) and Chlorpyriphos (8.86 ng g^{-1}) concentrations were the highest followed by monocrotophos (3.45 ng g^{-1}), quinolphos (3.26 ng g^{-1}) and ethion (3.23 ng g^{-1}) respectively. Similar observations of contamination of soil with OPPs have been reported from northern India (Kumari *et al.* 2008).

In India, about 6000 tons of herbicides been used for weed control, mainly in irrigated crops and on plantations. Among the four analyzed herbicides butachlor alone contributes 61% followed by pendimethalin (36%), fluchloralin (3%). Alachlor was not detected at all the locations. The total range of herbicides was <0.01 to 1.46 ng g^{-1} with a mean of 0.21 ng g^{-1} (Table 4). The individual concentration of herbicides ranged 0.03-1.28 ng g^{-1} (pendimethalin), 0.02-1.22 ng g^{-1} (butachlor), 0.01-0.25 ng g^{-1}

(fluchloralin) and <0.01 (alachlor). Butachlor and pendimethalin either alone or in combination with one hoeing in Delhi areas has shown their significant levels (Arora and Gopal 2004; Kumar *et al.* 2008, 2011).

4. Conclusion

The analysis showed that there is a fresh input of technical DDT but no Dicofol; however HCH data shows, lindane and technical HCH mixtures usage. Other insecticide like organophosphates and herbicides were not in alarming concentrations. The observed pesticides in agricultural soils may be a matter of concern for future food chain accumulation and human health so; regular investigation of pesticide residues is required on soil health and contamination levels.

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Table 1: ∑OCPs, ∑OPPs and ∑herbicide at different locations in agricultural are of Delhi region (ng.g^{-1} dw).

Location	∑OCP	∑OPP	∑Herb.	Location	∑OCP	∑OPP	∑Herb
1	1.36	<0.01	0.03	26	44.92	<0.01	0.00
2	12.64	<0.01	0.23	27	84.15	<0.01	0.00
3	14.81	<0.01	0.07	28	9.12	<0.01	0.00
4	3.42	<0.01	0.03	29	0.64	<0.01	0.00
5	4.93	<0.01	0.05	30	<0.01	<0.01	0.00
6	<0.01	7.37	0.22	31	2.16	<0.01	0.00
7	14.71	<0.01	0.05	32	0.75	2.88	0.10
8	16.73	6.00	0.18	33	<0.01	31.73	0.10
9	19.85	<0.01	0.42	34	2.45	<0.01	0.32
10	5.48	<0.01	0.33	35	0.96	<0.01	0.00
11	15.47	<0.01	0.09	36	0.68	<0.01	0.03
12	<0.01	<0.01	0.07	37	0.86	<0.01	0.05
13	12.84	<0.01	0.16	38	7.96	<0.01	1.24
14	1.86	<0.01	1.46	39	0.79	<0.01	0.02
15	14.93	2.26	0.55	40	<0.01	27.40	0.00
16	1.31	<0.01	1.22	41	1.11	<0.01	0.05
17	1.03	<0.01	0.73	42	15.79	<0.01	0.00
18	104.14	<0.01	0.97	43	4.08	<0.01	0.24
19	20.07	<0.01	0.51	44	1.22	<0.01	0.00
20	11.80	<0.01	0.17	45	2.10	<0.01	0.00
21	13.15	<0.01	0.36	46	13.27	6.46	0.06
22	77.94	<0.01	0.00	47	1.00	<0.01	0.04
23	71.41	<0.01	0.00	48	6.11	<0.01	0.02
24	43.78	<0.01	0.00	49	3.10	<0.01	0.03
25	14.21	<0.01	0.00	50	1.02	<0.01	0.23

Table 2: Organochlorine pesticides in agricultural soils from Delhi region (ng.g^{-1} dw).

Compounds	Range		Mean	%
	Minimum	Maximum		
α-HCH	<0.01	76.22	9.17	65.29
β-HCH	<0.01	5.77	0.61	4.37
γ-HCH	<0.01	18.51	2.02	14.40
δ-HCH	<0.01	9.41	0.68	4.82
∑HCH	<0.01	104.14	12.48	88.88
Aldrin	--------------------------- <0.01 ------------------------------			
Dieldrin	<0.01	2.38	0.14	1.01
α-endosulphan	-------------------------- <0.01 ------------------------------			
β-endosulphan	<0.01	7.57	0.27	1.89
∑endosulphan	<0.01	7.57	0.27	1.89
p,p'-DDE	<0.01	12.26	0.61	4.33
o,p'-DDT	<0.01	1.41	0.08	0.53
p,p'-DDT	<0.01	3.94	0.47	3.36
∑DDTs	<0.01	15.79	1.15	8.22
∑OCPs	<0.01	104.14	14.04	100

Table 3: Organophosphate pesticides in agricultural soils from Delhi region (ng.g^{-1} dw).

Compounds	Range		Mean	%
	Minimum	Maximum		
Phorate	------------------------- <0.01 ------------------------------			
Dimethoate	------------------------- <0.01 ------------------------------			
Phosphomidon	<0.01	20.95	10.47	24.90
Monocrotophos	<0.01	3.92	3.45	8.21
Malathion	------------------------- <0.01 ------------------------------			
Chloropyriphos	<0.01	31.73	8.67	51.53
Quinolphos	<0.01	6.46	3.26	7.68
Profenophos	------------------------- <0.01 ------------------------------			
Ethion	<0.01	6.46	3.23	7.68
∑OPPs	<0.01	31.73	1.68	100

Table 4: Herbicides in agricultural soils from Delhi region (ng.g^{-1} d w).

Compounds	Range		Mean	%
	Minimum	Maximum		
Alachlor	------------------------ <0.01 --------------------------------			
Fluchloralin	0.01	0.25	0.05	3.01
Butachlor	0.02	1.22	0.19	36.04
Pendimethalin	0.03	1.28	0.27	60.95
∑Herbicides	<0.01	1.46	0.21	100

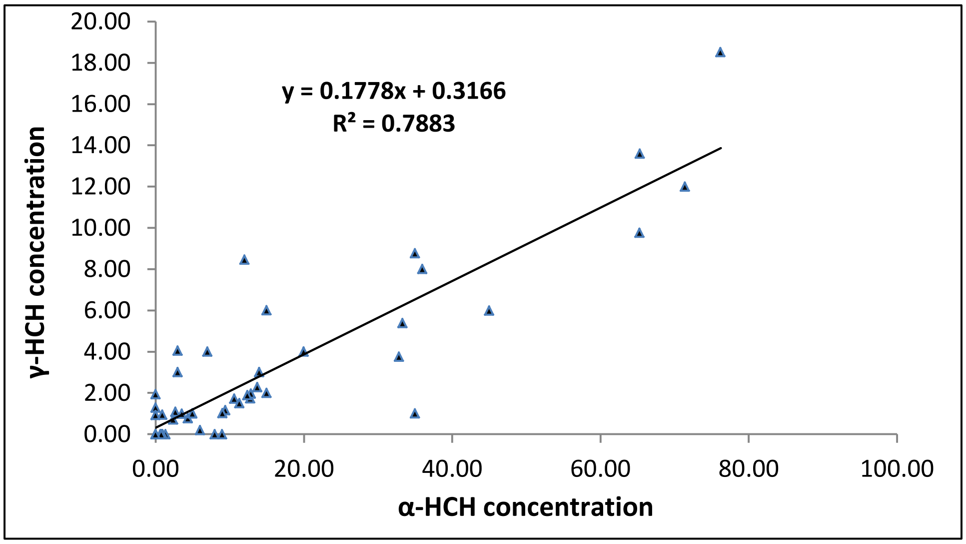

Figure 1: Relationship of α–HCH and γ–HCH in soil samples.

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