



Monitoring of Organochlorine Pesticides in the Soil of Bathinda City, Punjab, India

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Keywords

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Soil;
Land use;
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Gas Chromatography Electron-Capture Detector (GC-ECD)

Abstract

The concentration level of organochlorine pesticides in the soil of Bathinda, Punjab was studied using Gas Chromatography equipped with ⁶³Ni Electron-Capture Detector (GC-ECD). In February 2016, 19 soil samples were collected from different locations representing different land-use types of Bathinda i.e. agriculture, roadside, residence, commercial land, wasteland and industrial. Out of these land-use types studied, agricultural soil contributed maximum towards the OCP load followed by wasteland, residential, commercial, roadside and industrial sites. The study has shown the contamination of the soil of this region by all the nine pesticides viz. α -BHC, γ -BHC, Heptachlor, Endosulfan-I, Dieldrin, Endrin, 4,4'-DDD, 4,4'-DDT and Methoxychlor at all the sampling locations. Endosulfan was found to be the most abundant OCP followed by Heptachlor > 4,4'-DDT > Methoxychlor > α -BHC $\approx$ γ -BHC > Dieldrin > 4,4'-DDT > Endrin. The total organochlorine pesticides level ranged from 34.09 ng/g to 1407.79 ng/g. Most of the OCPs found in this study have been officially banned, but they are still present in the soil of this region. The present study would serve as a baseline data for further analyzing the pesticide usage in this cotton belt of Punjab.

1. Introduction

Pesticides are used to control the pests in home, garden, yards, food crops, agricultural crops, vector borne diseases like malaria, dengue, etc. and increase food production. In 1940s and 1950s, these chemicals were widely used to control pests in agriculture, forestry and home. Insecticidal chemicals that belong to organochlorine group are DDT, Metaoxychlor, Dicobol, HCH, Lindane, Chlordane, Aldrin, Dieldrin, Heptachlor, Endosulfan and Taxaphone. Chemical stability of organochlorine pesticides (OCPs) is responsible for their highly persistence nature. These OCPs enter into the food chain due to their lipophilic character causing contamination and are eliminated very slowly from the environment (Jones and De Voogt 1999; Nag and Raikwar 2008). The whole environment is affected by excessive use of pesticides that pollutes the air, water and soil (Bro-Rasmussen 1996). High temperature and humidity conditions in India are responsible

for fast multiplication of pests and higher crop losses (Lakshmi 1993). Further, India ranks second in Asia and 12th in the world for manufacturing of pesticide chemicals. 45% of pesticides in India are used on cotton crops, followed by paddy and wheat (Tiwana *et al.* 2009). Large amount of chemical pesticides and fertilizers are used in the agricultural region of India.

Organochlorine pesticides are known as Persistent Organic Pollutants (POPs). The main characteristic of POPs is their persistence nature, bioaccumulation and long-range transport. 20 to 70% of OCPs and their degradation products can remain in soil after their application. The presence of organic carbon in soil is responsible for the distribution and persistence of OCPs in surface soil (Devi *et al.* 2015; Wania and Mackay 1993). In 1962, Rachel Carson observed sudden deaths of birds due to spraying of pesticides (DDT) and wrote the book 'Silent Spring' (www.rachelcarson.org/SilentSpring.aspx). This forced the ban on the use of DDT in 1972. The United States and finally banned it in 1979, where 4000-5000 tons of DDT was being used annually for vector control applications (Kang and Chang 2011).

The Stockholm Convention on POPs was adopted in 2001 and entered into force in May 2004. The aim of this Convention was to eliminate the production and use of nine organochlorine pesticides (OCPs) and three industrial chemicals or by-products such as aldrin, dieldrin, DDT and its metabolites, chlordane, mirex, toxaphene, endrin, heptachlor. Lindane, chlordecone and pentachlorobenzene were added in August 2010. Many organochlorine pesticides were banned or restricted in the United States in 1970s. In India, 179 pesticides were registered for use and 30 others are banned, while seven are restricted. In September 1996, aldrin, chlordane and heptachlor were banned. In May 1990, Dieldrin was in restricted use and completely banned in July, 2003. In May 1990, Endrin was banned. In April 1997, HCH was banned.

2. Material and Methods

2.1. Study area and soil sampling

The study area is located in the Bathinda city of Malwa region of Punjab. It lies between coordinates 30.14°-30.29°E and 74.87°-74.99°N. Figure 1 shows the study area map of Bathinda city. Figure 2 shows the detailed sampling sites for OCPs. Soil samples using random sampling were collected from nineteen different sites, in the month of February, 2016. Approximately 1kg of soil was collected from a depth of 0-15 cm. (Jiang *et al.* 2009). A global positioning system (GPS, Garmin: eTrex 20) was used to record coordinates of each location of soil sampling. At room temperature, all the collected soil samples were air-dried in shade, remove all the stones and residual roots were removed from fully dried samples and powdered by pestle and mortar. To achieve the desired sample size, the crushed soil was passed through 1.18 mm sieve and homogenized by 'Quartering' method. Further, EPA 8081B method was employed for analysing the pesticides.

2.2. Extraction of soil samples

In this process, 500 ml round bottom flask and 40mm inner diameter of Soxhlet extractor was used. 10 gm of soil was placed in extraction chamber below plugs of glasswool. Then, 150 ml of n-Hexane and 150 ml of Acetone (1:1; v/v) were added into round bottom flask. Then extraction chamber was placed above the round-bottom flask having solvent and condenser above the

extraction chamber and heated using a heating mantle. The extraction process was run for 8 hours continuously at a temperature of 40°C. After extraction, the solvent was concentrated by using rotator evaporator upto 2 ml and stored in sealed vials with parafilm into a refrigerenator for further analysis.

Table 1 Description of Sampling Sites

Sample No.	Location	Latitude	Longitude	Land use type	Description
S1	Phus Mandi	30°10.118'N	74°59.414'E	Agriculture	Wheat field+fodder
S2	Jassi	30°09.376'N	74°58.113'E	Agriculture	Wheat field+fodder
S3	Giani Zail Singh College	30°10.272'N	74°55.562'E	Agriculture	Wheat field+fodder
S4	Housefed Colony	30°10.925'N	74°56.626'E	Residential	Garden soil
S5	Cantonment	30°11.944'N	74°57.772'E	Wasteland	Fallow land
S6	Mittal Mall	30°13.841'N	74°56.101'E	Commercial	In front of Mittal Mall
S7	Thermal Power Plant	30°14.067'N	74°55.807'E	Commercial	Fallow land
S8	Faridkot Road	30°16.544'N	74°56.204'E	Road side	Gill patti
S9	Joganand	30°15.746'N	74°58.094'E	Agricultural	Potatoes field
S10	Sivian	30°15.848'N	74°54.010'E	Agricultural	Wheat field
S11	Gill Patti	30°16.221'N	74°56.424'E	Agricultural	Wheat field
S12	DAV College	30°13.034'N	74°57.009'E	Commercial	Roadside
S13	Bus-Stand	30°12.357'N	74°57.739'E	Commercial	Parking area
S14	Bir-Talab	30°11.549'N	74°52.300'E	Commercial	Vegetation
S15	Mansa Road	30°10.873'N	74°57.720'E	Road side	Opposite Honda Agency
S16	Multania Pull	30°11.982'N	74°55.468'E	Agricultural	Wheat field
S17	Model Town	30°12.193'N	74°57.654'E	Residential	Garden soil
S18	Ambuja-Factory	30°14.199'N	74°54.859'E	Industrial	Roadside
S19	Central University of Punjab	30°10.358'N	74°57.973'E	Waste land	Fallow land

2.3. Sample clean-up

Cleanup was carried out by 100/200 mesh silica gel, that was activated at 130°C for 16 hours and cooled in a desiccator for 6 hours. Then the slurry of 3 gm deactivated silica gel and n-Hexane was transferred into 10 mm ID glass chromatographic column. Just prior to exposure of silica gel to air, the hexane eluate flow was stopped by closing the stopcock. 2 ml of the sample extract was added onto the column and extracted vial was rinsed two times with n-Hexane and each rinse was added to the column. Three elutions were carried out; first with 80 ml n-Hexane, second with 50ml n-Hexane and last with 15 mL of Dichloromethane. After clean-up, the fraction thus achieved was concentrated by using rotator evaporator upto 2 ml and stored in plastic vials into a refrigerator for further analysis.

2.4. Sample analysis

The samples were analysed by Gas chromatography (Shimadzu QP 2010 Ultra) with the Ni⁶³ Electron-Capture Detector. The capillary column used was Rtx-5ms, DB-5; 0.25 mm ID and 30 m length. The carrier gas and makeup gas was Helium (He) and Nitrogen (N₂) with a 1.25

ml/min and 29.30 ml/min flow-rate respectively. 2 μ l of the final extract (2ml) was injected at a temperature of 280°C. The initial oven temperature was 100°C. The oven temperature was kept at 100°C with a hold time of 2 min, then from 100°C to 160°C at a rate of 15°C/min with a hold time of 0 minute then finally from 160°C to 300°C at a rate of 5°C/min at a rate of 5 min. The detector temperature was maintained at 300°C.

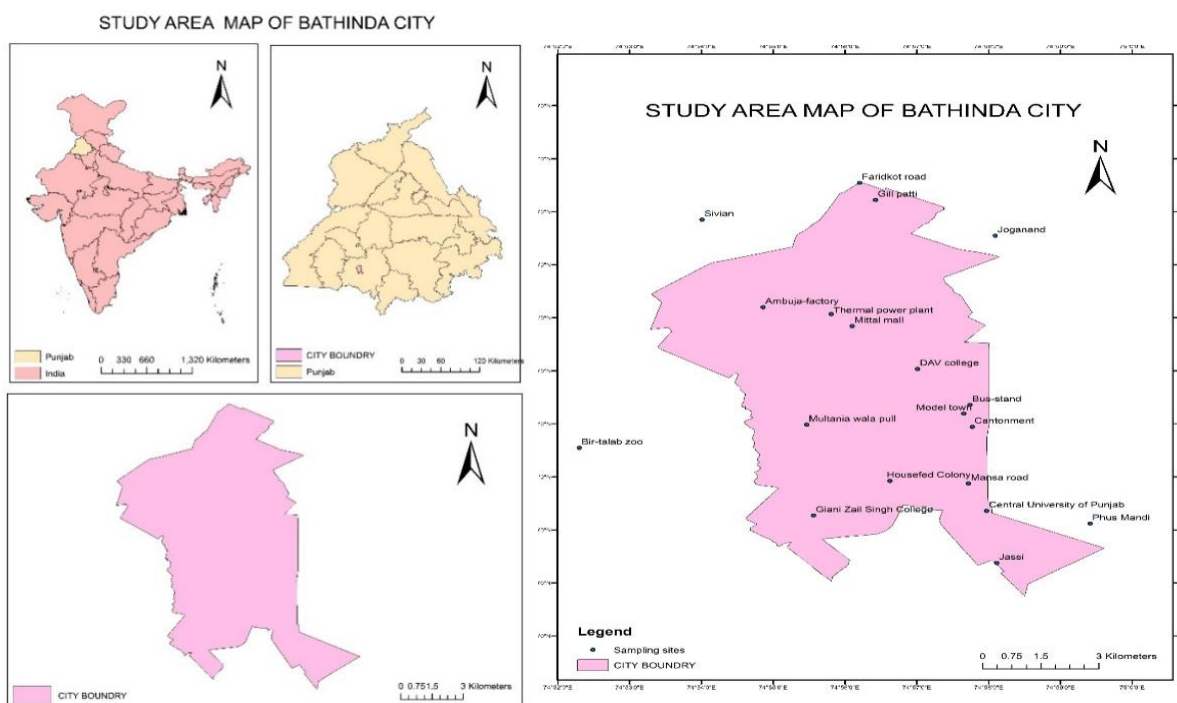


Fig 1 Map of study area of Bathinda city. Fig 2 Map showing the nineteen sampling sites of Bathinda

According to the retention time of the peaks of the standard mixture of pesticides, the identification and quantification of different organochlorine pesticides in the samples was done. The area of individual peak provides the concentration of pesticide residues in the extract. The concentration in the soil sample can be calculated using the formula given below.

$$\text{Concentration } \left(\frac{\mu\text{g}}{\text{kg}} \right) = \frac{(A_x) (V_t) (D)}{(\bar{C}\bar{F}) (V_i) (W_s)}$$

where:

A = Area of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (μ L).

D = Dilution factor.

$\bar{C}\bar{F}$ = Mean calibration factor from the initial calibration (area/ng).

V_i = Volume of the extract injected (μ L).

W = Weight of sample extracted (g).

3. Results and discussion

3.1. Spatial variation of Organochlorine Pesticides Concentration

In the present study, all the 19 soil samples analysed were found to be polluted with 9 organochlorine pesticides viz. α -BHC, γ -BHC, heptachlor, endosulfan-I, dieldrin, endrin, 4,4'-DDD, 4,4'-DDT and methoxychlor. According to the nature of pollutants they receive, all the sampling sites hold distinctive characteristics with respect.

S1, S2, S3, S9, S10, S11 and S16 representing agriculture sites where variety of pesticides and fertilizers are used to control pests and increase the crop production. S8 and S15 sample is collected from the sides of the roads besides the agricultural fields. Soil of S5 and S19 are the samples collected from the wasteland area, S5 surrounded by cantonment and road which is a fallow land and S19 is a playground of central university of Punjab, Bathinda. Soil of S18 sample is collected from the industrial area that is near the Ambuja-cement factory. S17 and S4 are the samples from the gardens of the residential areas, these areas are under constant influence of vector-control programme and the water used in the garden might also be contaminated. S6, S7, S12, S13 and S14 sites are the commercial locations, receiving pollution from the transportation sector. The spatial variation in concentration of OCPs at different locations is shown in a table 1 and figure 3.

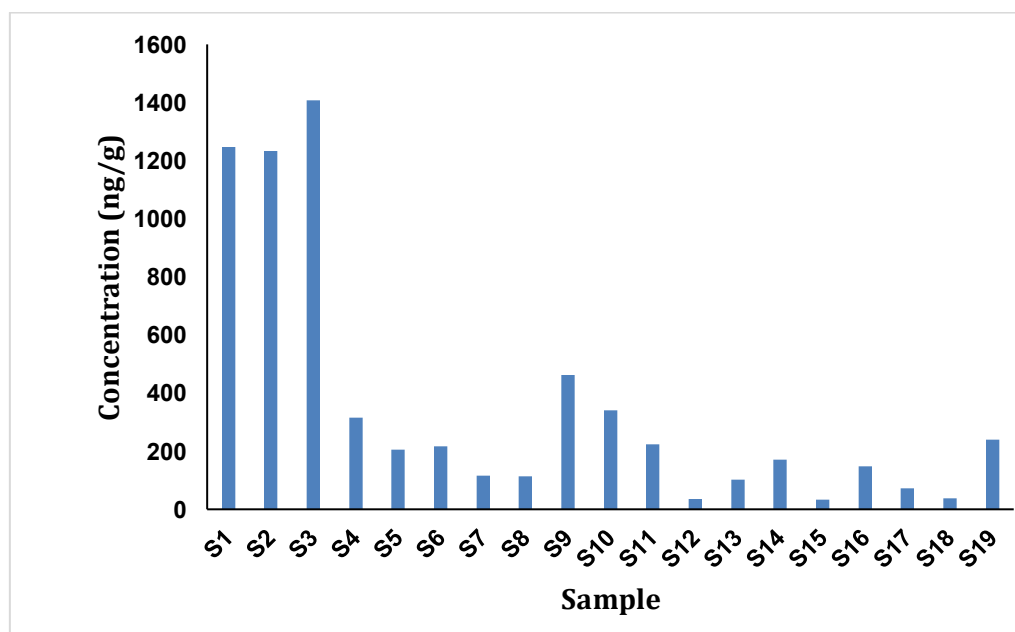


Fig 3 Total OCPs concentration

It was observed that the concentration of endosulfan-I and heptachlor were maximum than all the pesticides while endrin and 4,4'-BHC were minimum. The present result, with reference to OCPs detected in soil, is in accordance with various findings of Devi et al. 2015; Rao and Wani 2015; Parween et al. 2014; Kumar et al. 2012; Mishra et al. 2012; Kumari et al. 2008; Gbeddy et al. 2015; Akan et al. 2014; Kafilzadeh et al. 2012; Jamal, 2011.

Table 2 Concentration of pesticide residues detected in soil samples

Sample	α -BHC	γ -BHC	Heptachlor	Endosulfan-I	Dieldrin	Endrin	4,4'-DDT	4,4'-DDT	Methoxychlor	Total OCPs
S1	8.3	57.8	245.3	338.6	5.3	4.7	22.3	262.	302.4	1246.7
S2	14.5	9.2	292.0	481.5	9.9	10.9	50.1	279.	85.0	1232.7
S3	10.6	9.8	252.8	498.8	1.1	94.6	20.3	243.	276.3	1407.8
S4	3.0	6.1	38.8	66.5	7.8	7.3	56.0	88.4	40.5	315.3
S5	5.3	29.1	67.0	45.4	ND	ND	ND	22.6	35.6	205.0
S6	6.6	ND	13.8	94.9	ND	ND	ND	42.2	59.4	216.8
S7	3.4	13.6	41.4	41.3	ND	ND	ND	15.8	ND	115.5
S8	27.1	ND	49.2	9.1	ND	ND	ND	9.1	17.3	112.6
S9	42.8	54.8	71.0	47.6	218.1	ND	ND	28.4	ND	462.8
S10	50.0	59.8	116.7	71.5	ND	6.5	ND	35.7	ND	340.2
S11	8.7	37.8	44.5	56.1	16.7	3.5	ND	57.4	ND	224.7
S12	13.7	ND	7.0	8.4	ND	ND	ND	5.8	ND	35.0
S13	34.0	ND	35.7	19.7	ND	ND	ND	11.8	ND	101.1
S14	35.6	31.0	50.1	40.1	ND	ND	ND	12.8	ND	170.5
S15	ND	ND	24.1	ND	ND	ND	ND	10.0	ND	34.1
S16	29.2	17.9	66.2	22.6	ND	ND	ND	11.6	ND	147.4
S17	ND	ND	ND	48.7	ND	ND	9.5	14.1	ND	72.1
S18	11.2	ND	ND	13.1	ND	ND	ND	12.9	ND	38.0
S19	16.8	11.2	26.2	112.5	38.2	ND	ND	34.7	ND	239.8

ND- Below detection limit

Endosulfan-I was detected in all the samples except soil of S15 site and its concentration ranged from not detected (N.D.) to 489.9 ng/g. The higher concentration of endosulfan-I in all the soil may be the resultant of fresh usages of it. The present results are in accordance with the findings of Chakraborty et al. 2015 and Odukkaathil and Vasudevan 2016. Heptachlor were detected in all the samples except S17 and S18 and its concentration ranged from not detected to 292.0 ng/g. Analysis result showed that DDT were detected in all the samples and its concentration ranged from 5.8 to 279.6 ng/g. The concentration of DDE is lower than DDT. DDE was detected in lesser number of samples. The concentration of 4,4'-DDT was higher in the soil of S2 sample. Methoxychlor was detected in sample S1 to S6 and S7 while in S1 sample the concentration of methoxychlor was higher. Methoxychlor comprised 12% of total OCPs. The concentration of γ -BHC, /N was higher than α -BHC. Its concentration was lower as compare to endosulfan-I, heptachlor, 4,4'-DDT and methoxychlor. α -BHC and γ -BHC were sparingly present which could be the result of their less usage as compared to the more persistent OCPs. The concentration of dieldrin was greater than endrin. The concentration of dieldrin was higher in the soil of S9 sample

that is a potatoes field. Endrin and dieldrin were detected in less number of samples. Dieldrin and endrin comprised 4% and 2% of total OCPs.

3.2. Spatial Distribution of total OCPs

A spatial analysis of total OCPs in soils was examined using Inverse Distance Weighted (IDW) algorithm of Spatial Analyst toolbox in ArcGIS 10.3 software (Figure 4). The lowest concentration was found at station S15; while the highest concentration observed at station S3. which is resultant of agricultural activities on that land from which soil was collected. The amount of pesticides applied to the field mainly depends on the ability of a farmer to buy a variety of pesticides due to which concentrations of OCPs in agricultural fields shows extreme variations. Similar interpolation studies for concentration of pesticides maps have been carried out by Sun et al. 2016; Mishra 2012.

3.3. Percentage distribution of studied OCPs based on land use

Figure 5 shows percentage distribution of OCPs based on land-use. The concentration of endosulfan-I was highest among the pesticides at all different land uses sites except roadside site. 4,4'-DDD was present only in agricultural and residential sites; while absent in others. 4,4'-DDT was detected in all different land-uses. No endrin, dieldrin and 4,4'-DDD was found in commercial, residential and industrial sites. Figure 6 shows the spatial variation in OCPs concentration in different land-use types. Agricultural fields contributed maximum among all the land-use forms which is due to obvious reasons of pest control.

The present results are in accordance with the findings of Fang et al. 2007. and Mahugijia 2014. Wasteland was having second highest contribution among others due the volatilization of pesticides, atmospheric deposition and their transport from the other sampling locations. They travel a long distance from agricultural and residential area via air and mainly depend upon environmental conditions. The site S19, i.e., the playground of Central University of Punjab, Bathinda (CUPB) is a fallow site without any plantation. Secondly, the present CUPB campus was a Cotton thread mill industry earlier, which was abandoned and converted into an educational institute much later. This explains the presence of various OCPs in the campus due to its historical use; although no fresh input of OCPs has been provided. Further, 4,4'-DDD and endrin was not present at the site. Dieldrin and DDT were also present in small quantities; indicating subsequent volatilization and dissipation after past usage. Residential samples revealed high OCPs concentration, which could be due to the ongoing vector control programme and usage of pesticides in the kitchen gardens.

3.4. Box and Whisker plot for OCPs in soil

An analysis of Box and Whisker plot provides information on identifying outliers and comparison of distribution. Box and Whisker plot was used for the comparison of pesticide concentration in soil of Bathinda region. The data analyses of nine pesticides in soil of Bathinda region are summarized by Box and Whisker plots (Figures 7).

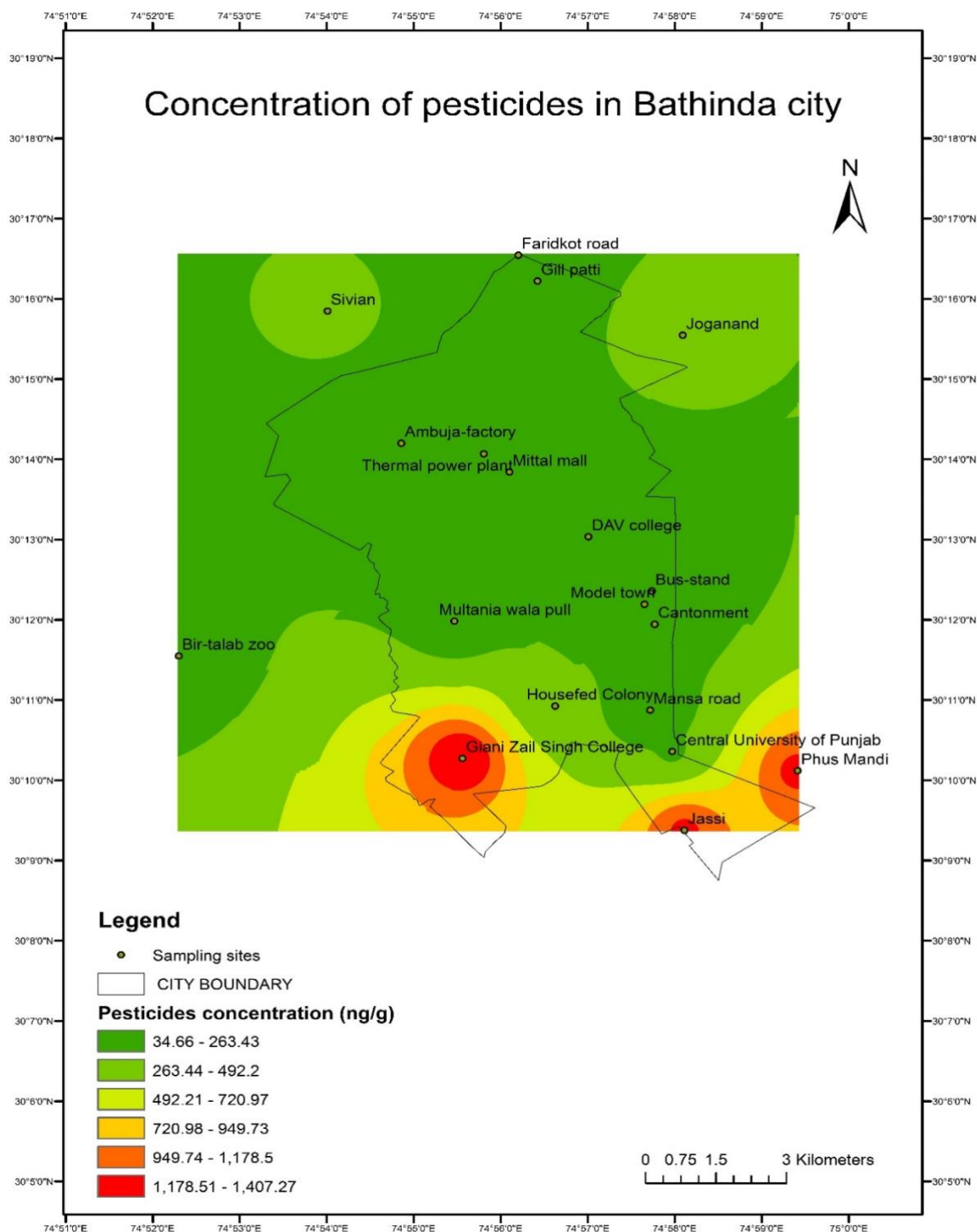


Fig 4 Spatial Distribution of total OCPs

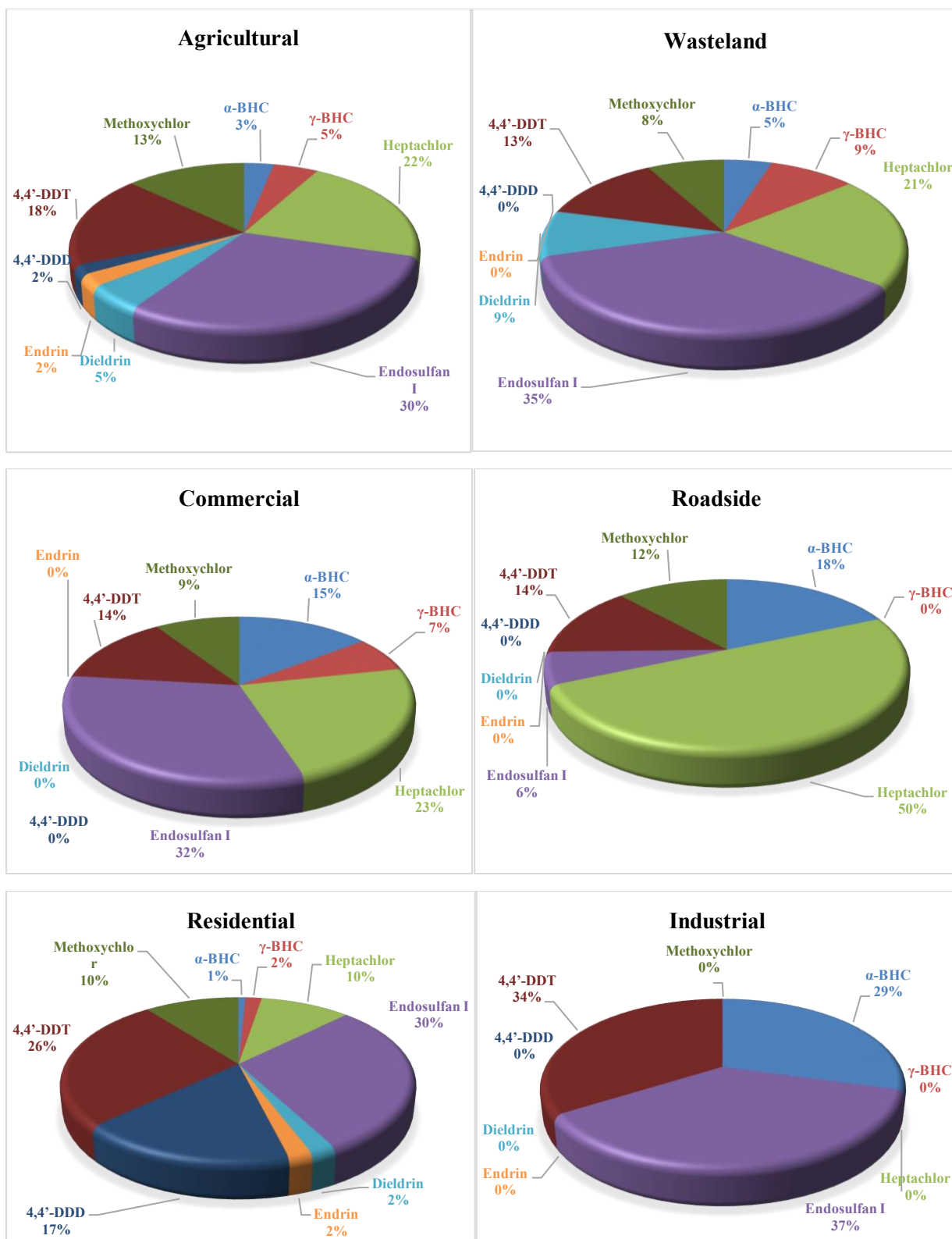


Fig 5 percentage distribution of OCPs

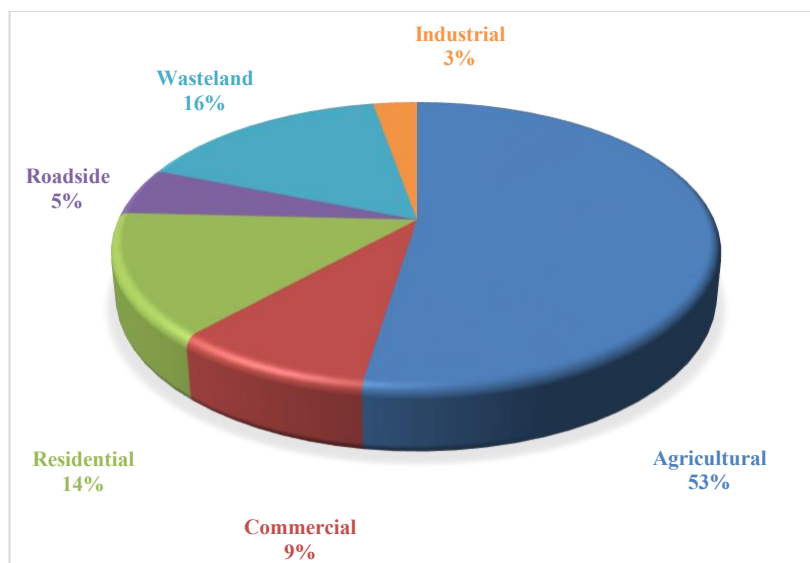


Fig 6 Spatial variation in total OCPs concentration

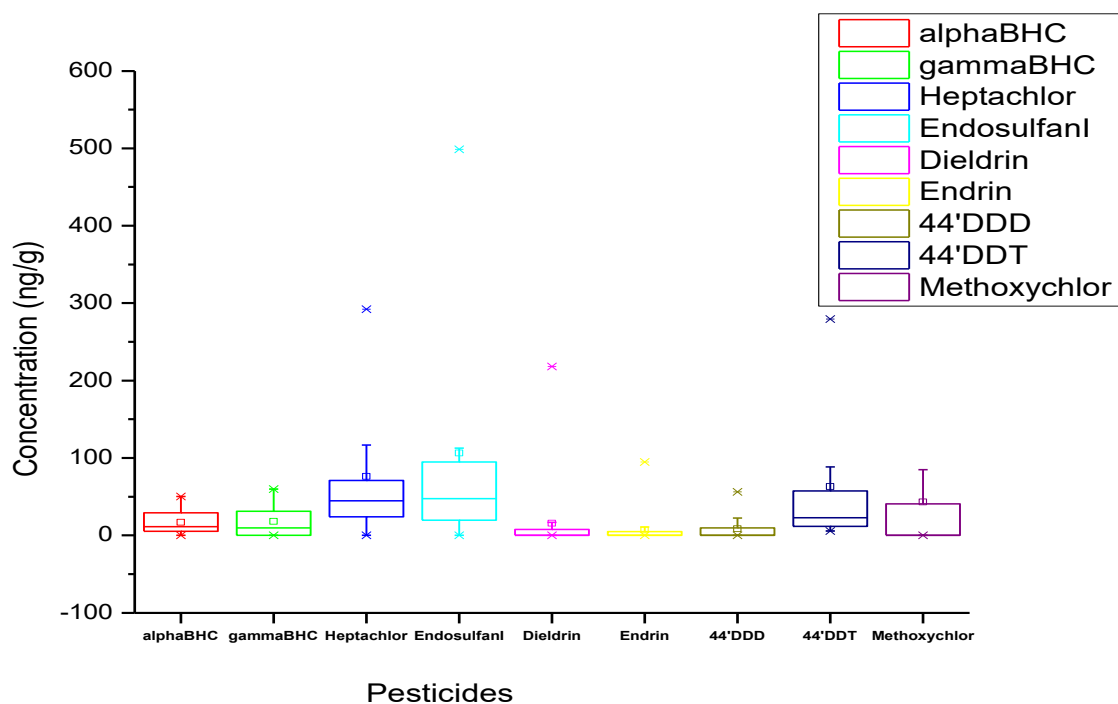


Fig 7 Box and Whisker plot of OCPs

The present study indicates that there is a major use of many organochlorine pesticides like DDT, heptachlor and endosulfan-I in the study area, which could be attributed to the inappropriate agriculture practices by the farmers where economic advantages take precedence over environmental concerns. These OCPs add greatly to the soil, either by atmospheric deposition or

agricultural and surface run-off. Soil acts as a sink of pesticides from where these chemicals are released into the environment. These pesticides persist in soil due to their unique characteristics such as, hydrophobicity, relatively low vapour pressure, long half-lives and long-range transport. Most of the OCPs found in this study have been officially banned, but they are still present in the soil of this region. Though the concentration obtained for each OCP is in the permissible concentration range of FAO as indicated in Table 3

Table 3 Permissible limit of OCPs

Pesticides	Half lives (ng/g)
α -BHC	2.5×10^5
γ -BHC	2.5×10^5
Heptachlor	5.0×10^4
Endosulfan-I	3.0×10^6
Dieldrin	5.0×10^4
Endrin	1.0×10^5
4,4'-DDD	1.0×10^7
4,4'-DDT	1.0×10^7
Methoxychlor	2.0×10^6

4. Conclusion

The present study demonstrated that the soils of the Bathinda are characterized by a high rate of contamination of OCPs, in spite of restrictions and bans on the usage of these pesticides in India. The main findings of the study are as under:

- At all the sampling sites, the observed concentrations of organochlorine pesticides could explain both continued use of these pesticides and their persistence in the environment.
- It was observed that almost all the 19 soil samples analysed were found to be contaminated with one or more of the 9 organochlorine pesticides viz. α -BHC, γ -BHC, heptachlor, endosulfan-I, dieldrin, endrin, 4,4'-DDD, 4,4'-DDT and methoxychlor. The total organochlorine pesticides level ranged from 34.1 ng/g to 1407.8 ng/g.
- Out of all the land use types, agricultural soil contributed the maximum load.
- 4,4'-DDT was found at all the samplings sites, despite its restricted use for vector control programmes.
- Endosulfan-I was having highest concentration among other pesticides in all land-uses except roadside soil.
- The mean concentration of pesticides was observed to be 723.2 ng/g, 222.4 ng/g, 194.1 ng/g 127.8 ng/g, 73.4 ng/g and 38.0 ng/g for agricultural, wasteland, residential, commercial, roadside and industrial sites respectively.
- All the value of pesticides was below the permissible limit, but their presence in the environment is a matter of great concern.
- Education of the farmers about these pesticides is necessary to manage the environmental hazards caused by these chemicals.
- In several environmental components, there is a need of fixed monitoring, assessment and reporting of contamination outline of these pesticides. Further, research, development and accomplishment of alternative malaria and pest control programmes are needed.

Author's Contribution: The author Harneet Kaur (HK) was involved in design of the research problem, the compilation of data, analysis and its interpretation, and drafting the research article. The author Puneeta Pandey (PP) was involved in conceptualization of the study, revising it critically and approving the final version.

Author's Agreement: All the authors have seen and approved the final version of the manuscript being submitted. Further, we warrant that the article is the authors' original work, hasn't received prior publication and isn't under consideration for publication elsewhere.

Declaration of interests: The authors declare no competing interests, whether financial or personal relationships with other people or organizations that could inappropriately influence or bias our work.

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