

Monitoring of Pesticides Level in Agricultural Soils using Validated Method

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ABSTRACT

Agricultural soil serves as a natural medium for the growth of a variety of edible crops and vegetables. Likewise, systematic assessment of agricultural soil becomes mandatory as it reflects direct linkage with the food chain and health of living beings. In the past few decades, it has been detected with the presence of various environmental contaminants being heavy metals and pesticides. The current research focuses on the investigation of different pesticides viz., organophosphorous (OPPs) and organochlorine (OCs) in agricultural soils of Amritsar (India) using gas chromatograph. All soil samples (except south zone) were found to be contaminated with one or the other pesticide. The linearity was observed over the range from 0.1 to 1.0 mg L⁻¹ for all pesticides. The coefficient of determination (r²) was observed in the range of 0.916 to 0.999. The LODs, based on signal-to-noise (S/N) ratio of 3, ranged from 0.003 to 0.009 mg L⁻¹. The precision of the proposed method was presented and accepted with an RSD <20%. This report pertains to the presence of alarming concentrations of pesticides in soils of Amritsar (India).

Keywords: Gas chromatograph, Organochlorine, Organophosphorus, Soil pollution.

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INTRODUCTION

Pesticides are synthetic chemicals used to control weeds and harmful pests in agricultural fields and lawns. In a broader term, these substances are used to defend humans against the insect vectors of pathogens. These are also used to protect crop plants from despoliations by fungi, arthropods, and rodents (Patnaik, 1999; Onuwa *et al.*, 2017). It has been observed that in recent years, the use of various pesticides has vigorously increased in agricultural crops. Over 98% of the sprayed insecticides and 95% of the herbicides find other destination than their mark species and thereby contaminate land and water (Miller, 2004; Chiaia-Hernandez *et al.*, 2017). At high dosages, pesticides not only kill the insects and other invertebrates but birds, mammals, and floral vegetation as well.

The use of pesticides in agricultural soils is uninterruptedly increased after world war-II in order to enhance world food production. Since then, there has been a marked development of different varieties of pesticides belonging to different groups (Bai *et al.*, 2006). Among various types of pesticides, organophosphorus pesticides (OPPs) and organochlorine pesticides (OCPs) are the most widely used classes of agricultural pesticides (Toan *et al.*, 2007; Wang *et al.*, 2006; Chen *et al.*, 2009; Zhang *et al.*, 2011; Arora *et al.*, 2014).

Pesticides contain certain chemicals that are tenacious soil contaminants whose impact persists for decades and adversely affect soil conservation (USEPA, 2007). These chemicals hinder the nitrogen fixation which is required for the growth of higher plants. Insecticides like DDT, methyl parathion and pentachlorophenol interfere with legume-*Rhizobium* chemical signaling which results in a decrease rate of nitrogen fixation and reduced crop yields. The spatial distribution of these pesticides is so wide that their concentration further increases in the soil, sometimes even after the high rainfall (Vryzas *et al.*, 2009). They penetrate into deeper layers of the soil and become persistent, thus continue to show their harmful effects generation after generations by altering the normal respiration rate and CO₂ production of living organisms (Bartha *et al.*, 1967; Bento *et al.*, 2016).

DDTs, DDEs, and endosulfans are the predominant contaminant compounds in the surface soil samples. These pesticides are detected in substantial amounts in the soils which are a threat to the surrounding and downstream ecosystem as well as to the

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whole biodiversity (Gong *et al.*, 2004, Westbom *et al.*, 2008, Nishina *et al.*, 2010; Ritsema and Geissen, 2017). Pesticides present inside the soil not only affect the food crops but also show their adverse consequences in vegetables. Even after the ban to most of the OCPs like hexachlorohexanes (HCHs), these groups of pesticides are frequently applied to agricultural fields to obtain more crop yield. These pesticides enter the living systems *via* (i) inhalation while spraying (ii) by ingestion of the plants those uptake these contaminants from the soil and (iii) leaching of these contaminants from soil to the groundwater and the surface water that is the source of drinking or bathing *etc.* Different OCPs like HCHs, DDTs, aldrin, dieldrin, endrin, chlordane, heptachlor have been reported to cause cancer, nervous system damage and reproductive disorders including damage of immune system in humans (Rissato *et al.*, 2006, Wong *et al.*, 2008; Yang *et al.*, 2012). These not only contaminate the soil ecosystem but also play a predominant role in the pollution of water as well as air. Therefore, the time to time monitoring of such environmental pollutants is a crucial need of the hour.

The adverse health impacts of various pesticides have been reported worldwide, including India in which Punjab has been found to be grasped by these pesticides to the greatest extent. In the last three decades, OCPs are the most widely used class of

insecticides in soils of Punjab to control pests. The cancer reports in the Majha region of Punjab also could be directly correlated to the use of high amounts of pesticides in agricultural crops. Considering the hazardous effects of pesticides on the living system, the present study was aimed to investigate the residue level of organophosphorus and organochlorine pesticides different agricultural soils of Amritsar (India) using validated methods.

MATERIALS AND METHODS

Reagents and chemicals

Thirteen different pesticides most commonly used in the agricultural fields were selected for the study: organophosphorus (ethion and chlorpyrifos) and organochlorines (HCHs alcholor, aldrin, heptachlor, endosulfans endosulfan, and bifenthrin) (Table 1). Analytical standards of these pesticides were procured from Sigma-Aldrich. All the solvents and chemicals used for the analysis were of analytical grade purchased from E-Merck (Merck India, Mumbai).

Sampling

Soil samples were collected from agricultural fields of Amritsar divided into five zones viz. center, north, east, west, and south. To analyze the seasonal variations in the sample areas, sampling was carried out twice in a year under cultivation of two different crops viz., wheat and rice for two subsequent years. Thus the present investigation was carried out on 20 samples 10 of each under rice and wheat cultivation respectively. For each sample collection, the soil was dug to the depth of approximately 10 to 15 cm.

Standard Stock Solution

Standard stock solution (10 ppm) was prepared in acetone. A standard solution of the different concentration range of the analysts was prepared by diluting the stock solution in acetone. All standard solutions were stored at 4°C.

Extraction and Clean-up

Five grams sample was weighed and 50 ml mixture of petroleum ether: acetone (1:1 v/v) was added. The mixture was shaken for 2 hours on a mechanical shaker. The contents in conical flask were filtered using filter paper containing 2 g of anhydrous sodium sulfate (ASS) immersed in petroleum ether (PE). Residue in the flask was washed twice with 20 mL of solvent, i.e., petroleum ether. The filtrate was concentrated to 2 mL in a rotary vacuum evaporator at 35°C. The concentrated extract was relocated to a chromatography column pre-packed with glass wool plugs and activated florisil (5 g each) between the two layers of ASS. The column was pre-washed with extraction solvent. The concentrated sample extract was eluted with 100 mL of PE. The filtrate was allowed to evaporate. The residue was reconstituted in 5 mL of acetone and injected 1 µL to the gas chromatograph.

Equipments

Gas chromatograph from Shimadzu, Japan with electron capture detector (ECD) was used for the analysis. The RTX-5 column (30 m length, 0.25 µm film thickness and 0.25 mm ID diameter) was used for the analysis and the operating conditions were set as injector temperature (250°C), column temperature (220°C), detector temperature (300°C), initial oven temperature 220°C for 2 min and raised to 260°C and then held for 4 min. Nitrogen was used as the carrier gas at a flow of 30 mL min⁻¹ under pressure of 134.1 KPa.

RESULTS AND DISCUSSION

Method validation

It is necessary to validate the method for practical application with characteristic parameters like linearity, precision, accuracy, and LOD. As shown in Table 2, the linearity was detected over the range from 0.1 to 1.0 mg L⁻¹ for all pesticides. The coefficient of determination (r^2) was observed in the range of 0.916 to 0.999. The LODs, based on signal-to-noise (S/N) ration of 3, ranged from 0.003 to 0.009 mg L⁻¹. The precision of the proposed method was presented and accepted with an RSD < 20%. The International Conference of Harmonization (ICH) guideline (ICH, 1994) has recommended that the RSD less than 20% was acceptable for a concentration of < 0.1%. Accuracy of all pesticides was evaluated at different concentration viz. 0.025, 0.05 and 0.1 mg L⁻¹ (Table 3). The result revealed that accuracy (80-120%) was found under acceptable limits as per ICH guidelines (ICH, 1994).

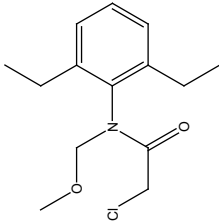
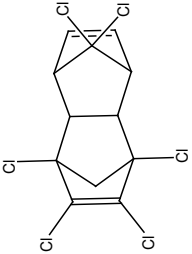
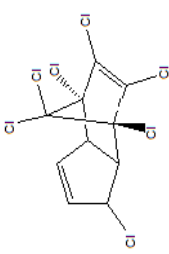
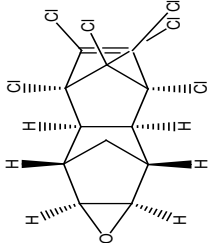
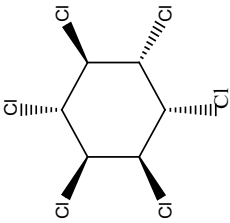
Pesticide Concentration in Soil Samples

The positive detections of pesticides in agricultural soil samples of Amritsar are presented in Table 4. In the present study, different pesticides studied in the soil samples were detected (in mg kg⁻¹) in the range of: α-HCH (0.041 and 1.091), β-HCH (0.048–6.525), γ-HCH (1.492–3.879), chlorpyrifos (0.776–6.48) alcholor (2.282 and 2.3), aldrin (0.289–2.017), heptachlor (0.059–3.602), α-endosulfan (0.024) and bifenthrin (0.047–0.491) whereas the rest of the pesticides viz. dieldrin, endrin, ethion, and β-endosulfan were found below detection limits in the soil samples studied. Heptachlor was found to be more widely distributed among all the samples studied as 65% of the total samples. It may be due to the continuous application of this insecticide to the agricultural fields. Among the detected pesticides, α-endosulfan was found to be least distributed in all the sampling zones as it was found to be present in a single sample, i.e. E-II_w. The probable reason for the absence of this pesticide in most of the samples may be its less usage in soils.

HCHs constituted about 41.37% of the total pesticides studied in the soil samples. Among HCHs, β-HCH was detected with a maximum concentration of 40.3% of the total HCHs. The very high concentration of HCHs in soil samples probably due to the direct spray of HCHs into the fields and their leniency with water. HCHs are more stable and persistent in nature than other pesticides (Feng *et al.* 2003). In the present research, about 87% samples, even crossed the MRL values for agricultural soils (0.05 mg kg⁻¹). Similar observations were documented by Zhang *et al.* (2011). Wang *et al.* (2006) also detected the alarming concentration of HCHs (86.08–93.85 mg g⁻¹) in soils of China, which was many times higher than the recommended value of HCHs by Environmental quality standards for soils of China, GB15618—1995.

Chlorpyrifos was detected in 25% of the total soil samples, and its maximum concentration reached up to 6.48 mg kg⁻¹ in the W-I_R sample which was assumed for being exposed to this pesticide for a long time. Fuentes *et al.* (2006) found agricultural soils of Chile contaminated with a high concentration of chlorpyrifos. The estimated amount of percentage of detection of all the pesticides studied was in the descending order of γ-HCH (23.02 %) > chlorpyrifos (20.28 %) > heptachlor (18.55 %) > β-HCH (16.67 %) > bifenthrin (7.139 %) > alcholor (6.756 %) > α-HCH (1.669 %) > α-endosulfan (0.035 %). Various reports have supported the high concentration of DDTs in the agricultural lands (Wang *et al.* 2006; Nishina *et al.* 2010; Arora *et al.*, 2014; Onuwa *et al.*, 2017) but the negligible concentration of DDTs was detected in the present study. It might be because of the restricted ban on the usage of DDT in soils of India. OCs constituted over 79.71 % of the total pesticides

Table 1: Details of different pesticides observed during the study.

S. no.	Pesticide	IUPAC name	Molecular formula	Chemical structure	Chemical class	Group	Health hazards
1.	Alachlor	2-Chloro-N-(2,6-diethylphenyl)-N-(methoxymethyl)acetamide	$C_{14}H_{20}ClNO_2$		Organochlorine	Herbicide	Causes potential damage to liver, kidney, spleen, lining of nose and eyelids
2.	Aldrin	1,2,3,4,10,10-Hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5:8-dimethanonaphthalene	$C_{12}H_8Cl_6$		Organochlorine	Insecticide	Headache, dizziness, nausea, vomiting, tremor, ataxia, convulsions, central nervous system depression, respiratory failure and renal damage, causes liver cancer in animals
3.	Heptachlor	1,4,5,6,7,8,8-Heptachloro-3a,4,7,7a-tetrahydro-4,7-methano-1H-indene	$C_{10}H_5Cl_7$		Organochlorine	Insecticide	Tremors, convulsions, liver necrosis and blood dyscrasia
4.	Dieldrin	(1aR,2R,2aS,3S,6R,6aR,7S,7aS)-3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-2,7:3,6-dimethanonaphtho[2,3-b]oxirene	$C_{12}H_6OCl_6$		Organochlorine	Insecticide	Affects central nervous system, liver, kidneys, and skin, causes headache, dizziness, nausea, vomiting, tremor, ataxia, chronic and tonic convulsions and respiratory failure
5.	α -HCH	α -1,2,3,4,5,6-hexachlorocyclohexane	$C_6H_6Cl_6$		Organochlorine	Insecticide	Blood disorders, dizziness, headache and changes in level of sex hormones

(Cont...)

S. no.	Pesticide	IUPAC name	Molecular formula	Chemical structure	Chemical class	Group	Health hazards
6	β -HCH	β -1,2,3,4,5,6-hexachlorocyclohexane	$C_6H_6Cl_6$		Organochlorine	Insecticide	Blood disorders, dizziness, headache and changes in level of sex hormones
7.	γ -HCH	(1r,2R,3S,4r,5R,6S)-1,2,3,4,5,6-hexachlorocyclohexane			Organochlorine	Insecticide	
8.	Chlorpyrifos	O,O-Diethyl O-3,5,6-trichloropyridin-2-yl phosphorothioate	$C_7H_9Cl_3NO_3PS$		Organophosphorus	Insecticide	Neurological disorders, lung cancer, urinal disorders
9.	Endosulfan	6,7,8,9,10-Hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepine-3-oxide	$C_9H_6Cl_6O_3S$		Organochlorine	Insecticide	Skin absorption or inhalation of its vapors can induce nausea, vomiting, abdominal pain, irritation, co
10.	Endrin	(1aR,2S,2aS,3S,6R,6aR,7R,7aS)-3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-2,7,3,6-dimethanonaphtho[2,3-b]oxirene	$C_{12}H_8OCl_6$		Organochlorine	Insecticide	Headache, dizziness, nausea, vomiting, abdominal pain, insomnia, confusion, stupor, convulsions, tremors, rise in blood pressure, fever, frothing of mouth, deafness, coma and respiratory failure, also carcinogenic to all living beings

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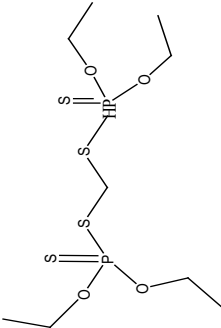
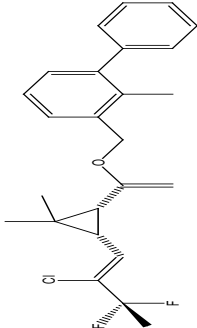
S. no.	Pesticide	IUPAC name	Molecular formula	Chemical structure	Chemical class	Group	Health hazards
11.	Ethion	O,O,O',O'-Tetraethyl S,S'-methylene bis(phosphorodithioate)	C ₉ H ₂₂ O ₄ P ₂ S ₄		Organophosphorus	Insecticide	Prevents working of neural enzyme acetylcholinesterase
12.	Bifenthrin	2-Methyl-3-phenylphenyl methyl (1S,3S)-3-[(Z)-2-chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropane-1-carboxylate	C ₂₃ H ₂₂ ClF ₃ O ₂		Organochlorine	Insecticide	Nervous tissue damage

Table 2: Validation parameter for multi pesticide residue analysis by GC-ECD for determination of pesticides in agricultural soil samples.

Validation parameter	α -HCH	β -HCH	γ -HCH	Chlorpyrifos	Alcalor	Aldrin	Heptachlor	α -Endosulfan	Dieldrin	Endrin	Ethion	β -Endosulfan	Bifenthrin
Retention Time	3.5	3.6	3.8	4.2	4.8	5.0	5.7	6.9	6.3	7.6	7.4	7.5	10.3
Linearity (r ²) (0.1-1.0 ppm)	0.981	0.973	0.999	0.993	0.995	0.993	0.895	0.999	0.999	0.995	0.916	0.960	0.987
Repeatability (RSD) ^a	4.62	3.85	9.147	26.1	10.48	5.441	5.502	3.2	2	16.41	8.45	1.5	27
Reproducibility (RSD) ^b	4.77	7.95	8.38	5.76	7.19	1.98	8.72	10.06	12.01	5.42	5.84	9.17	1.32
% recovery	96.38	108.1	105.5	97.8	118.6	84.9	108.7	110.6	119.6	102.2	101.6	119.2	88.18
LOD (ppm)	0.006	0.006	0.003	0.003	0.003	0.003	0.003	0.003	0.003	0.003	0.003	0.006	0.009

^aData represent the mean value of three replicates^bData represent the mean value of three days each day three replicates

Table 3: Validation parameter for reproducibility for determination of pesticides in agricultural soil samples.

Name of the pesticide	Recovery (%)		
	0.025 ppm	0.05 ppm	0.1 ppm
α-HCH	96.38 ± 0.045	85.5 ± 0.153	98.7 ± 0.173
β-HCH	108.1 ± 0.393	95.69 ± 0.119	103.3 ± 0.233
γ-HCH	105.5 ± 0.173	81.39 ± 0.307	85.83 ± 0.088
Chlorpyrifos	97.8 ± 0.265	85.28 ± 0.205	84.77 ± 0.176
Alcholor	118.6 ± 0.404	100.2 ± 0.153	83.1 ± 0.153
Aldrin	84.9 ± 0.115	105.2 ± 0.208	88 ± 0.058
Heptachlor	108.7 ± 0.173	100.4 ± 0.145	80.9 ± 0.153
α-Endosulfan	110.6 ± 0.208	110.2 ± 0.24	90.2 ± 0.252
Dieldrin	119.6 ± 0.338	94.9 ± 0.115	92.87 ± 0.088
Endrin	102.2 ± 0.219	111.7 ± 0.12	97.67 ± 0.145
Ethion	101.6 ± 0.176	110.9 ± 0.088	80.83 ± 0.186
β-Endosulfan	119.2 ± 0.578	105 ± 0.058	87.03 ± 0.203
Bifenthrin	88.18 ± 0.148	101.1 ± 0.12	92.03 ± 0.376

Data represent mean and standard error values of three replicates (Mean ± SE)

studied, whereas OPs was 20.28 % of the overall concentration of pesticides. In another study, Ritsema and Geissen (2019) evaluated 76 pesticides in 317 agricultural topsoils across Europe and revealed alarming concentrations of the detected pesticides which evidences the persistence of pesticides at high environmental risk across the globe.

CONCLUSION

A selective, sensitive and convenient method has been standardized to determine organochlorine and organophosphorus pesticides in agricultural soils. The fidelity of this method was proved as it was successful to detect very low concentrations of pesticides. This is an imperative study, which provides the level of contamination of agricultural soils of Punjab. In our study, even the banned pesticides like HCHs, chlorpyrifos, and endosulfan were found to be prevalent in higher concentrations much beyond the threshold limit which is alarming for the crop consumers.

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