

Monitoring of pesticide residues in soil samples from agricultural field of Haryana, India

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Abstract

Agricultural field soil was analysed for the presence of 52 crop protection agents which are commonly used by farmers in Panipat, Karnal, Rohtak, Ambala, Sonipat and Faridabad, districts of Haryana. A total of 161 samples were screened for the presence of pesticide residues of which 51 samples showed the presence of pesticides. Sixteen samples amongst these were detected with multi class pesticides. The sampling of soil was conducted during winter (pre and post) and monsoon (pre and post) seasons in 2014-2018. The survey indicated that in various districts of Haryana, almost 32% of the agricultural filed soils were detected with pesticide residues.

Seasonal study revealed that 40% of monsoon season and 26.50% of winter season soil samples showed the presence of pesticides. 58.74% of the soil samples showed the presence of chlorpyrifos mainly and 23.5% of sample showed presence of cypermethrin. Panipat, Rohtak, Ambala and Faridabad soils mainly showed the presence of chlorpyrifos whereas in Karnal soil samples, cypermethrin was commonly detected.

Keywords: Soil, Pesticide Residue, Multi-pesticide residue, Monsoon, Winter.

Introduction

The use of pesticides in agricultural production protects the crops and increases the yield. Their use in the environment raises serious concern which will out weight the overall benefits^{4,19}. The use of organochlorine pesticides was earlier prevalent across the world to control pests and vector born diseases^{1,30}. However as per Stockholm Convention²³, they have been banned or have restricted use in health and hygiene sector. The organochlorine compounds are stable compounds with half-life range between few months to several years. DDT is one of the OCs which degrades in the soil from 4 to 30 years. Similarly, other chlorinated pesticides remain present in soil for several years².

In spite of ban in production and consumption of POP's, the pesticides have been reported in multi-environment matrices^{13,26,28,29}. According to the reports of WHO and UNEP, around 3 million people got poisoned and about 2 lakhs people die each year by pesticides all over the world. 95% cases are from developing nations¹². Soil acts as a sink for plenty of chemical pesticides through atmospheric

deposition which re-emits them into atmosphere¹⁷. POPs present in surface soil pollutes air through air soil exchange mechanism⁸. The distribution of POPs in soil assesses the level of contamination, sources of emission, human health impacts and environment risks.

India's population is about 16% as compared to the other world but its land area is only 2%. Due to rapid population growth, emphasis on achieving food self-sufficiency compelled Indian farmers to use pesticides. India has highest consumption of pesticides namely monocrotophos, endosulfan, phorate, chlorpyrifos, methyl parathion and quinalfos, mancozeb, paraquat, butachlor, isoproturon, phasphamidon⁶.

India is second largest producer of pesticides in Asia and fourth largest pesticide producing nation in the world next to USA, Japan and China. It is reported in literature that Indian soil is contaminated with persistent organic residues^{5,11,15}.

Most of the pesticides pass into the soil by missing target, surface runoff, spillage during application. Dissipation of pesticides depends on soil characteristics, cropping system, irrigation pattern and ambient climatic conditions³. Polar pesticides may pollute ground water through leaching which may affect the health of consumers of the area²¹. Pesticides residues are widely distributed in all types of soils²⁴. Literature documents report on presence of different class of pesticides in soil from several parts of India⁷ and the World¹⁰.

Location and Sampling: The monitoring study was conducted on agricultural field soil samples for the presence of 52 pesticides which are commonly applied in the area of Panipat, Karnal, Rohtak, Ambala, Sonipat and Faridabad. A total of 161 soil samples were tested for the presence of pesticide residues. The sampling of soil and analysis were conducted during 2014-2018 during winter (pre and post) and monsoon (pre and post) seasons. The reason of the soil sampling during these seasons is that almost 90% of the crops are cultivated during this season.

The areas selected for the study are most populous agriculture belt of North India. Soil samples was collected from 50 cm in depth from a minimum of 10 random locations in the sampled field and mixed together. Representative soil samples were collected in one kg polythene bags. The samples were transported to the laboratory under cold conditions and stored at 4°C. The samples were air dried, finely powdered and sieved before extraction.

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Material and Methods

Anhydrous sodium sulfate (Na_2SO_4 , AR grade), sodium chloride (Hi-Media, AR grade) methanol and n-hexane solvents of pesticide residue were grade purchased from Merck (Darmstadt, Germany). The water samples were screened for 52 pesticides viz. α -HCH, β -HCH, γ HCH, δ HCH, alachlor, aldrin, dicofol, pendimethalin, o,p DDE, α -endosulphan, heptachlor, p,p DDE, endosulphan sulphate, dieldrin, o,p DDD, β -endosulphan, p,p DDD, o,p DDT, p,p DDT, difenthrin, fenpropathrin, lambda cyhalothrin, β -cyfluthrin, fipronil, cypermethrin, fenvalarate, fluvalinate, deltamethrin, anilophos, chlorfenvinfos, chlorpyrifos, butachlor, pretilachlor, chlorpyrifos-methyl, dichlorvos, ethion, malathion, parathion methyl, monocrotofos, phorate, profenofos, quinolfos, trizofos, fenitrothion, paraxon-methyl, pretilachlor, fenamifos, edinfos, dimetoate, diazinon, fenthion, parathion phosphomidon and atrazine. The Certified Reference Material (CRM) was purchased from Sigma Aldrich (USA).

The soil samples were dried, sieved and mixed homogeneously. 20 g sample was taken in 250 ml stoppered conical flask followed by addition of 50 ml of methanol (HPLC grade) to each conical flask. Each conical flask was stoppered and subjected shaking on auto-shaker for one hour at 160 rpm. After one hour of shaking, samples were left undisturbed on flat surface for half an hour for settling of soil particles. After half an hour, 20 ml of clear supernatant was pipetted out and filtered through anhydrous sodium sulphate (Na_2SO_4), pre washed with 20 ml methanol to reduce the absorption of pesticides in the sample during clean up. All the filtrate was collected into 100 mL flat bottom round flask.

Anhydrous sodium sulphate (Na_2SO_4) bed was finally washed by 10 mL of methanol (HPLC grade) into same flat bottom round flask. The solvent was evaporated near to dryness on rotary evaporator (Buchi RP-300) under vacuum (500 mm of Hg) keeping constant temperature of water bath at 40°C. The sample was reconstituted by 2mL n-hexane for instrumental analysis using gas chromatography mass spectrometry.

Pesticide solution of approx.100 mg/L concentration was prepared. 10 mg/L of pesticides mixture standard solution was prepared from 52 individual pesticides standard stock solutions. Calibration standards of pesticide mixture 10, 50, 100, 500, 1000 and 2000 $\mu\text{g/L}$ were prepared in n hexane. The standard stock, calibration standards and working solutions were stored at 4°C. Figure 1 shows the standard mixture chromatogram of 500 $\mu\text{g/L}$ (0.5 ppm).

Gas chromatography system (Agilent 7890A) and mass spectrometer (Agilent 5975C inert XL EI/CI MSD) with DB-5MS fused silica capillary column GC Column, 30 m \times 0.25 mm \times 0.25 μm were used for identification and quantification of 52 pesticides in soil samples. 1 μl of sample was injected in splitless mode with linear flow at 1 ml/min of carrier gas helium (99.995% purity). Column oven was programmed at 50°C (hold 2 min) and then increased to 280°C (at the rate of 8°C per min) (hold 15 min). The injector was set at 280°C temperature with an interface temperature of 280°C and ions source temperature set at 250°C and quadruple temperature at 150°C. Agilent ChemStation System software was used for instrument control and data analysis. Gases were passed through purification filters which can trap oxygen and moisture before supply to the column of the instrument.

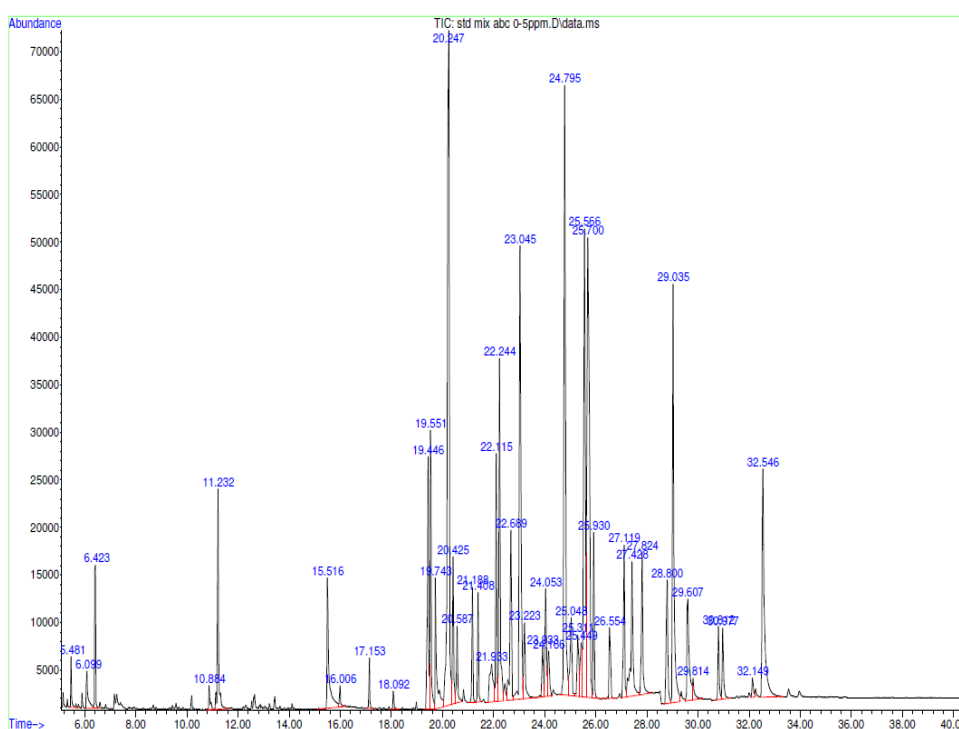


Figure 1: Standard Mixture Chromatogram (0.5 ppm)

The mass spectrometer was operated in full scan data in a mass range of m/z 35–500 in positive ion electron impact energy of 70 eV and an emission current 60 μ A. Scanning interval and SIM sampling rate were kept at 0.5 and 0.2s respectively. The presence of pesticides residues in collected soil samples was quantified by comparing the retention time, peak area and peak height of the sample with those of the standards.

The pesticides can be confirmed when mass fragments at particular RT match with the fragmentation pattern of pesticide library database (NIST) above 90 per cent. Analytical instrument and lab methods were validated as per ISO/IEC 17025 (2005). The mean recovery ranged from 80% to 95% and within the 95% confidence level with a relative standard deviation between 5-20% at 0.050 μ g/L level. The detection limit was 0.010 μ g/L.

Results and Discussion

Agricultural soil samples were screened for the presence of 52 pesticides which are commonly used for crop production and protection in cultivation of vegetables, cereals oil seeds in areas of Panipat, Karnal, Rohtak, Ambala, Sonipat and Faridabad. Total 161 samples were monitored for the presence of pesticide residues among which 51 samples

showed the presence of pesticides and out of which 16 samples showed the presence of multiple pesticide residues. From selected locations of Panipat, 64 samples were collected, out of which 14 samples showed the presence of pesticides and 3 of the samples recorded the presence of multiple pesticide residues (Table 1).

Similarly 36 samples from Karnal, 28 samples from Rohtak, 13 samples from Ambala, 10 samples from Sonipat and Faridabad were analyzed for the presence pesticide residues, among which 14, 12, 3, 2 and 6 soil samples recorded the presence of single pesticide respectively while 9 samples of Karnal and 2 samples of Faridabad were detected with multiple pesticide residue.

Seasonal impact on the presence of pesticides in the area under study was recorded. Mainly two seasons were considered (pre and post) monsoon and winter. Figures 2 and 3 showed variation of seasonal pesticides recorded in soil samples collected from Panipat, Karnal, Rohtak, Ambala, Sonipat and Faridabad during 2014-2018. Total of 59 samples were collected during monsoon season (pre and post) amongst which 24 samples recorded the presence of pesticide residues. 102 samples were collected during (pre & post) winter season in which 27 samples showed the presence of pesticide residues.

Table 1
Districtwise contamination of soil in Winter and Monsoon seasons

District	Sampling Month and year	Season	Number of samples	Total Number of Samples	Contaminated Samples	Total Contaminated Samples	Samples with Multi-residue	Total Samples with Multi-residue
Panipat	July-2014	Monsoon	10	64	1	14 (22%)	-	3
	August-2014	Monsoon	04		-		-	
	November- 2014	Winter	14		3		2	
	January-2016	Winter	19		3		1	
	September-2017	Post Monsoon	06		5		-	
	December-2017	Winter	11		2		-	
Karnal	January-2016	Winter	7	36	1	14(39%)	-	9
	January- 2016	Winter	5		-		-	
	November-2017	Pre-winter	10		-		-	
	March-2018	Post-winter	2		2		1	
	October-2018	Pre-winter	12		11		8	
Rohtak	December-2015	Winter	5	28	5	12(43%)	-	-
	September-2017	Post Monsoon	11		4		-	
	July-2018	Monsoon	8		-		-	
	October-2018	Post-Monsoon	4		3		-	
Ambala	January-2016	Winter	8	13	2	3(23%)	-	-
	January-2016	Winter	5		1		1	
Sonipat	August-2014	Monsoon	4	10		2(20%)	-	-
	February-2016	Winter	6		2		1	
Faridabad	December -2015	Winter	5	10	3	6(60%)	1	2
	Janaury- 2016	Winter	5		3		1	
Total				161		51(32%)	-	

Seasonal study revealed that 40% of the monsoon soil samples and 26.47% of winter soil samples showed the presence of pesticides. 6 samples from Panipat and 7 samples from Rohtak collected during monsoon season were recorded with pesticide residue whereas residues in soil samples collected from Ambala and Faridabad were below

the quantifiable levels (0.01 mg/kg). Similarly, 8, 3, 5, 2, 3 and 6 soil samples recorded pesticide residues collected during winter season from Panipat, Karnal, Rohtak, Ambala, Sonipat and Faridabad respectively. Out of the 27 soil samples of winter season containing pesticides, 16 soil samples were detected with multiple pesticide residue.

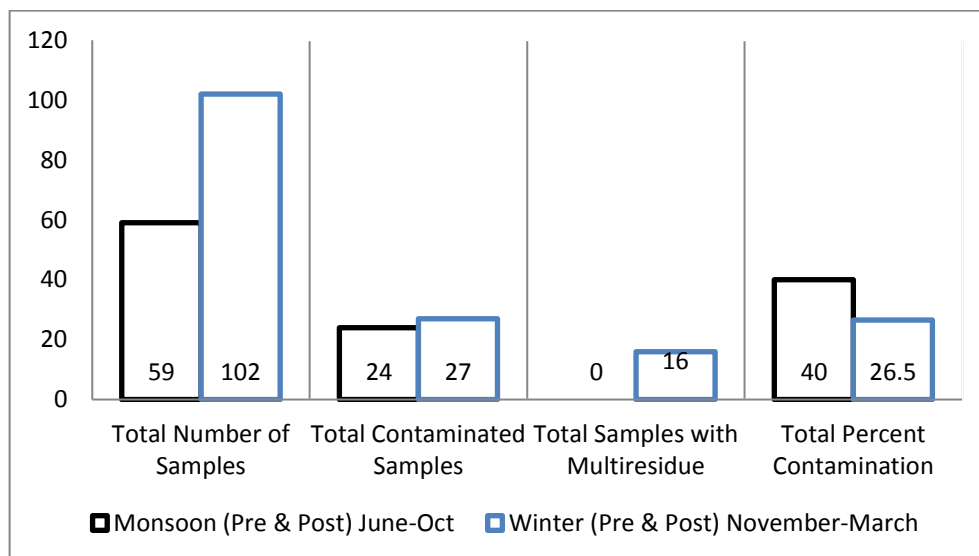


Fig. 2: Seasonal Contamination of Pesticides

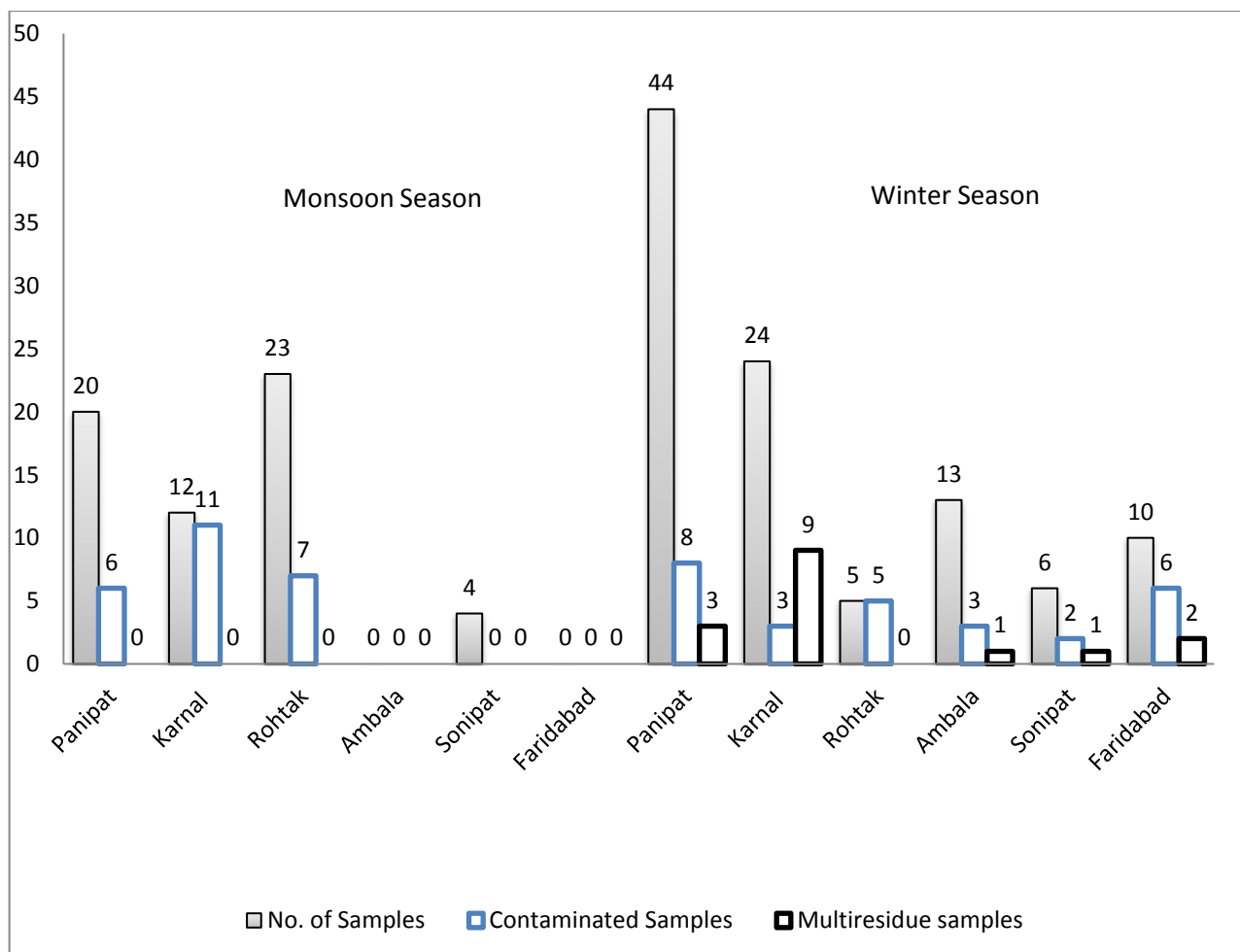


Fig. 3: Seasonal Contamination of pesticides in soil samples from some districts of Haryana

Atrazine, DDT, malathion, chlorpyrifos, quinalfos, bifenthrin, cypermethrin, pendimethlin, β -endosulphan, pretilachlor, butachlor, deltamethrin, λ -cyhalothrin were detected in the analyzed soil samples of Panipat, Karnal, Rohtak, Ambala, Sonipat and Faridabad (Table 2). Chlorpyrifos was detected in maximum number of 30 samples followed by cypermethrin which was detected in 12 samples, third was malathion in 10 samples and DDT in 9 samples. Pendimethlin and butachlor were detected in 8 soil samples each whereas λ -cyhalothrin was detected in 6 samples and pretilachlor in 3 samples. Only one sample out of the total analyzed 161 samples shows the presence of quinalfos, bifenthrin, β -endosulphan and deltamethrin. β -endosulphan, butachlor, deltamethrin and λ -cyhalothrin were detected in Karnal samples and bifenthrin was detected in Sonipat soil sample and atrazine in Panipat field soil.

Chlorpyrifos was detected in samples from each location. Malathion was detected in samples from all the study locations except in Ambala. Similar study was also conducted earlier in which DDT isomers and endosulphan isomers were recorded in agricultural soil of Hissar, Aligarh, Kasimedu, Ennore, Cochin, Visakapatnam and Agra of India^{16,18} whereas similar trends were observed in surface soil from Central Europe, Beijing, Shenzhen Shanghai of China, Mexico, Kihhihi Uganda and Hyderabad Pakistan and Korba India⁹. Similar trend in the studies of top soil of 11 European countries was reported for 76 pesticide residues with maximum of total pesticide concentration of 2.87 mg/kg in 6 cropping systems having more than one pesticide residue. 17% of the fields were reported with no pesticides and 25% of the fields were reported with single pesticide, while more than one pesticide residue was found in 58% of the fields²⁰.

Yadav et al²⁷ have reviewed the literature on organochlorine pesticides in soil and reported values for 4 cities in Nepal. A

range of values is reported for data from China, India, Mexico, Uganda, Kenya, Pakistan, Central Europe and Nepal for DDT, hexachlorocyclohexane and endosulfans. The largest value reported is 2.179 mg/kg and 2179 ng/g for DDT and metabolites of DDT²⁷.

Table 3 shows the range of pesticides detected in soil samples in the study area. Among the detected pesticides, chlorpyrifos, cypermethrin, malathion, DDT isomers, butachlor and Lambda λ -cyhalothrin were detected in maximum number of soil samples. All samples which showed the presence of atrazine, bifenthrin, β endosulfan, deltamethrin, λ cyhalothrin and cypermethrin were above residue limit (<http://emalab.com/EPA8081W.html>) and were in the range of 0-1.13; 0-0.07; 0-1.73; 0.292-0.674; 0.094-0.678 and 0.132-12.4 mg/kg respectively. DDT isomers, malathion, chlorpyrifos, pendimethlin were detected in the range of 0.001-1.57; 0.01-2.22; 0.008-1.515 and 0.01-4.62 mg/kg respectively.

The residue level of both DDT and endosulfhan isomers ranged from 0.001-1.57 and 0.001-1.57 mg/kg respectively which is similar to reported values in literature (0.0009 to 0.0103 and 0.029 to 0.095 mg/kg respectively) from studies conducted in other parts of the country^{7,14} and the world^{5,22}.

All the soil samples showed the presence of pesticides with atrazine, bifenthrin, cypermethrin, β endosulphan, Deltamethrin and - λ cyhalothrin which were above EPA residue limit of 0.05 mg/Kg for atrazine, cypermethrin 0.02 mg/Kg for bifenthrin, 0.04 mg/Kg for β Endosulfan and 0.01mg/Kg deltamethrin and - λ - cyhalothrin (Table 3). Malathion (6 sample), chlorpyrifos (24 samples), DDT (6 samples) and pendimethlin (7 samples) were above EPA residue limits of 0.05 mg/Kg for DDT and malathion, 0.03 mg/Kg for chlorpyrifos and 0.02 mg/Kg for pendimethlin.

Table 2
Name and number of pesticides detected in some districts of Haryana.

Pesticides	Panipat	Karnal	Rohtak	Ambala	Sonipat	Faridabad	Total Pesticides Detected
Atrazine	1	-	-	-	-	-	1
DDT	3	-	-	-	-	6	9
Malathion	2	1	1		1	5	10
Chlorpyrifos	10	5	4	2	1	8	30
Quinalfos	1	-	-	-	-	-	1
Bifenthrin	-	-	-	-	1	-	1
Cypermethrin	-	9	2	1	-	-	12
Pendimethlin	-	7	-	1	-	-	8
Beta-Endosulphan	-	1	-	-	-	-	1
Pretilarchlor	2	1	-	-	-	-	3
Butachlor	-	8	-	-	-	-	8
Deltamethrin	-	1	-	-	-	-	1
Lambda-cyhalothrin	-	6	-	-	-	-	6

Table 3

Pesticides detected in contaminated samples, percent contamination and concentration range and regulatory limit

Pesticides	Total No. of Contaminated Samples	Range of Conc. mg/kg	Residue Limit EPA-8081	Sample Above Residue Limit	Percent Contaminated Samples
Atrazine	1	0-1.13	0.05	1	1.96
DDT	9	0.001-1.57	0.05	6	17.64
Malathion	10	0.01-2.22	0.05	8	19.60
Chlorpyrifos	30	0.008-1.515	0.03	24	58.74
Quinolfos	1	0-0.004	-	-	1.96
Bifenthrin	1	0-0.07	0.02	1	1.96
Cypermethrin	12	0.132-12.41	0.05	12	23.5
Pendimethalin	8	0.01-4.62	0.02	7	15.68
Beta- Endosulphan	1	0-1.73	0.04	1	1.96
Pretilachlor	3	0.125-0.99	-	-	-
Butachlor	8	0.005-0.124	-	-	-
Deltamethrin	1	0.292-0.674	0.01	2	1.96
Lambda-cyhalothrin	6	0.094-0.678	0.01	6	11.76

EPA- Environment Protection Agency

Conclusion

The study reveals the chances of exposure of agricultural soil due to non-target use of pesticides of the area. The soil gets exposed to pesticide contamination from irrigation water wind, rainfall etc. From the study, it may be concluded that almost 32% of the soil of the area detected the presence of pesticide residues. Seasonal variation of pesticides in soil samples in the study conducted in regions during winter and monsoon season reveals that 40% of monsoon and 26.50% of winter season soil samples showed the presence of pesticides. 58.74% of the soil samples showed the presence of chlorpyrifos and 23.5% of sample show presence of cypermethrin. About 10% of the samples showed the presence of more than one pesticide. 22%, 39%, 43% of samples detected with pesticides were from Panipat, Karnal and Rohtak respectively.

Similar studies were also carried out in India and abroad, the levels of pesticides detected were comparable. Mostly DDT and endosulfan were of concern in all locations. Other pesticides like atrazine, DDT, malathion, chlorpyrifos, quinalfos, bifenthrin, cypermethrin, pendimethlin, β - Endosulphan, pretilachlor, butachlor, deltamethrin, Lambda λ -cyhalothrin were also detected. Panipat, Rohtak, Ambala and Faridabad soils mainly showed the presence of chlorpyrifos whereas in Karnal soil samples, cypermethrin was commonly detected.

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