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Determination of Pesticide Residue in Soil, Water and Grain from IPM and Non-IPM Field Trials of Rice

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Abstract Soil, water and rice grain samples from field trials conducted under the IPM and non-IPM modules in Kaithal (Haryana) region were analyzed for pendimethalin, atrazine, lindane and chlorpyriphos, and in Dehradun (Uttarakhand) region, samples were analyzed for carbendazim only. The pesticide residues were found below the detectable limit in the soil and water samples of the Kaithal region. From Dehradun region the residues of carbendazim in rice grains were detected at 0.001 mg/kg level, and in soil they were in the range of 0.03–0.001 mg/kg. The insecticides applied in IPM as well as non-IPM trials in both regions were observed to be below the prescribed maximum residue level.

Keywords Pesticides · Residues · Basmati rice · IPM · Non-IPM

Synthetic pesticides are important component of our IPM module. The rice crop is the second highest pesticide consuming crop in India. Basmati rice fetches higher price in domestic and export markets due to which farmers do not hesitate to apply high inputs including pesticides for managing the pests. Moreover pesticide application to rice may affect the quality of environmental resources such as ground and surface water besides affecting soil health. Farmers have mostly been relying on chemical control methods for managing insect pests and diseases of paddy.

NCIPM has proposed an IPM schedule for rice in the northern states of India. The module is being validated at different farmer's field. For the module to be successful, it is imperative for the estimation of pesticide residues in the IPM trials and need to be compared with the pesticide practices followed by the local farmers, which is denoted as NIPM. It is mandatory for IPM as well non-IPM grown crops.

Rice is attacked mainly with stem borer and leaf folder insect pests, blast and blight diseases besides few weeds like Echinocloa (Garg et al. 2004). Pendimethalin and atrazine are used to control *Echinocloa*, the major weed and other minor weeds of paddy cropas as is also documented in http://agmarknet.nic.in/paddy-mannual.htm, Chlorpyriphos and lindane, insecticides are used for managing stem borers, leaf folder and leaf and plant hoppers on paddy in Haryana region. The main aim of the present investigations was to compare the pesticide load in IPM and non-IPM crops of rice fields.

Materials and Methods

In Kaithal region of Haryana, rice, variety 'Taraori Basmati', is grown organically without any application of chemical pesticides in IPM trials while farmers spray pesticides viz, pendimethalin, atrazine, lindane and chlorpyriphos in non-IPM trials. Carbendazim is being applied as one of chemical component of IPM programs in IPM trials in rice crop (variety Dehradun Basmati) at Dehradun.

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The field trials were conducted during the year 2005–2006 for both the regions.

Five samples each of soil, water and grain of paddy, collected from IPM and non-IPM fields of Basmati rice fields, were analyzed for insecticides viz pendimethalin, atrazine, lindane and chlorpyriphos; and carbendazim widely used on paddy cultivation in the regions of Haryana and Uttranchal, respectively. The samples were collected in triplicate including control from Tilwari village in Dehradun and Kaithal region in Haryana. The samples of soil and irrigation water from Kaithal region were analyzed for the detection of pendimethalin, atrazine, lindane and chlorpyriphos pesticides as per the history of previous applications, while the samples of soil, water and rice grain from Dehradun region were analyzed for carbendazim. For recovery studies of pendimethalin, atrazine, lindane, chlorpyriphos and carbendazim, the control samples (with no chemical treatment) of paddy grain, soil and water were spiked with all these pesticides at two fortification levels i.e. 0.1 and 1.0 μg/g.

The soil samples were extracted in a Soxhlet extractor for 6 h using a mixed solvent of acetone:hexane (1:1) as per methodology by Mukherjee and Gopal (2005). The samples were further cleaned up over a glass column (30 cm × 1.5 cm id) packed with anhydrous neutral alumina (5 g) between two layers of anhydrous sodium sulfate (2 g). The column was pre-washed with hexane and the samples were eluted with 100 mL hexane:acetone (1:1) solvent system. The solvent was concentrated and made up in hexane (5 mL) and was analyzed using GLC–ECD.

The rice grain sample (50 g) in triplicate was also subjected to Soxhlet extraction with hexane:acetone (1:1, 350 mL) for 6 h. The extract was concentrated and subjected to liquid–liquid partitioning with acetonitrile saturated with hexane (3 × 50 mL) as described by Gupta and Gajbhiye (2004). The combined hexane layer was discarded and the aqueous acetonitrile layer containing residues was diluted with 2% sodium chloride solution (100 mL) and exchanged into Dichloromethane (DCM, 3 × 50 mL). The DCM layer was evaporated completely and made up in hexane (10 mL) before analysis by GLC. For HPLC analysis the DCM extract was evaporated completely and the sample was made up in acetonitrile.

The pH of water samples was found to be 8.2, it was neutralized to pH 7 using 0.1N HCl. Five hundred milliliters of water sample was transferred to separatory funnel and partitioned with DCM (50 mL × 3) (Mukherjee and Gopal 2001). The DCM was evaporated completely and transferred to hexane analytical grade solvent (10 mL) for analysis of atrazine, chlorpyriphos, lindane and pendimethalin using GLC–ECD.

The GLC parameters were: the column (megabore, 12 m × 0.52 μm × 1 μm) temperature was programmed from 180 to 240°C. The injector and detector temperatures were 250 and 300°C, respectively. Detection limit was 0.1 μg/L for lindane, chlorpyriphos and atrazine; and for pendimethalin it was 0.5 μg/L. The retention times were 2.82, 3.52, 4.42 and 6.12 min. for lindane, atrazine, chlorpyriphos and pendimethalin, respectively.

HPLC parameters were: HPLC with UV–VIS detector. Column RP-18 (30 cm × 4 mm, 5 μm) was set at λ_{max} 250 nm, mobile phase was acetonitrile–water (70:30) @ 0.5 mL/min flow rate. The retention time was 4.32 min for carbendazim.

Results and Discussion

The recoveries of different pesticides from paddy grain, soil and water is presented in Table 1. The recovery of lindane in paddy grain, soil and water was more than 80% at 1.0 μg/g while 70–85% at 0.1 μg/g fortification level for all commodities. Similar result was observed for pendimethalin and chlorpyriphos. For atrazine it was in the range of 75–80% recovery for all commodities at both the fortification levels. For carbendazim the recovery was in the range of 65–80% for all paddy commodities at both the fortification levels.

Table 1 Recovery of pesticides from different commodities

S. No.	Pesticide	Fortification level (μg/g)	Percent recovery of pesticides			
			Paddy grain	Paddy straw	Paddy soil	Paddy water
1	Lindane	1.0	82–85	87	87	88
2		0.1	72–76	82	76	84
3	Pendimethalin	1.0	79–83	79	86	83
4		0.1	74–81	72	78	81
5	Chlorpyriphos	1.0	88–91	88	82	88
6		0.1	77–81	75	80	81
7	Atrazine	1.0	78–84	80	79	83
8		0.1	73–78	73	75	79
9	Carbendazim	1.0	75–79	74	69	79
10		0.1	72–74	69	64	72

Table 2 Residues of pesticides in soil and water in Kaithal region

Commodity	Pesticide	Residues (mg/kg; μg/L)							
		IPM				Non-IPM (farmers practices)			
		R1	R2	R3	Mean	R1	R2	R3	Mean
Soil (mg/kg)	Lindane	0.001 (0.1)*	0.001	0.001	0.001	0.003	0.006	0.005	0.001
	Atrazine	0.01 (0.01)*	0.02	0.01	0.015	0.002	0.003	0.002	
	Chlorpyriphos	0.003 (0.05)*	0.001	0.001	0.001	0.001	0.004	0.003	
	Pendimethalin	0.03 (0.05)*	0.03	0.05	0.01	0.03	0.05	0.02	
Water (μg/L)	Lindane	ND	ND	ND	ND	ND	ND	ND	ND
	Atrazine	ND	ND	ND	ND	ND	ND	ND	ND
	Chlorpyriphos	ND	ND	ND	ND	ND	ND	ND	ND
	Pendimethalin	ND	ND	ND	ND	ND	ND	ND	ND

ND, Not detected; R_1, R_2, R_3 three replicates from IPM and non-IPM fields in triplicate; * MRL of pesticides

Table 3 Residues of Carbendazim in Soil and water in Dehradun region

Commodity	Pesticide residues (μg/g; μg/L)							
	IPM				Non-IPM (farmers practices)			
	R1	R2	R3	Mean	R1	R2	R3	Mean
Soil (μg/g)	0.03 (5 ppm)*	0.001	0.001	0.03–0.001	ND	ND	ND	ND
Water (μg/L)	ND	ND	ND	ND	ND	ND	ND	ND
Rice grain (μg/g)	0.002	0.0009	0.001	0.001	ND	ND	ND	ND

ND, Not detected; R_1, R_2, R_3 three replicates from IPM and non-IPM fields in triplicate; * MRL of pesticides

The residues of the atrazine, lindane, chlorpyriphos and pendimethalin pesticides in soil and water samples of IPM trials (organic farming) were below the detectable limit in Kaithal area (Table 2), while that of non-IPM trials showed traces of these pesticides. Pendimethalin was detected in trace amounts in two samples.

In Tilwari village of Dehradun region, farmers were not spraying any pesticides in rice crop; instead carbendazim was being applied as chemical component in IPM trials for increasing the productivity of rice crop, which was comparatively low in earlier cases. The non-IPM samples of rice grain and soil did not contain the residues of carbendazim in Dehradun region (Table 3). In 3, out of 15, IPM rice grain samples, residues of carbendazim were detected at 0.01 μg/g level. In the soil samples, the residues of carbendazim were detected in five samples in the range of 0.03–0.001 μg/g for IPM trials. This may be due to leaching and persistence behavior of carbendazim applied as seed dressing for paddy crop in Dehradun region. The pesticides were below the detectable limit in Dehradun region (Table 4).

Table 4 Limit of detection of various pesticides in paddy crop

S. No.	Pesticide	Limit of detection in	
		Rice grain/soil (μg/g)	Water (μg/L)
1.	Lindane	0.001	0.001
2.	Atrazine	0.01	0.01
3.	Chlorpyriphos	0.001	0.001
4.	Pendimethalin	0.01	0.01
5	Carbendazim	0.01	0.01

The insecticides applied in IPM as well as non-IPM trials of both the regions were found to be below maximum residue level (MRL). The yield of Taraori Basmati rice in Kaithal region for IPM (organic farming) and non-IPM (farmers practice) field trials were 32.9 and 31.2 q/ha, respectively, while for Dehradun Basmati rice in IPM and non-IPM filed trials, it was observed to be 21 and 18 q/ha, respectively.

The results show that harvest produce from IPM trials were safe to consume as the residues of insecticides were either below their MRL or not detected. Similar kind of work has been carried out for okra and brinjal vegetable crops (Arora and Singh 2004; Arora 2008).

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