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Residues of Pendimethalin after Weed Control in Cabbage Crop (*Brassica oleracea* var L. *Capitata*)

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Cabbage (*Brassica oleracea L. Capitata*) crop is one of the most important winter vegetables grown in India. It is attacked by number of pests (Babu *et al.*, 2001) including weeds. While the losses caused by insect pests and diseases of cabbage crop are visible, there are various studies reported about them and they can be effectively controlled. The weed control has however not been studied that extensively. Major weeds of this crop are *Coronopus didymus* and *Melilotus indica* and minor weeds are *Partheniun hysterophorus, Rumex dentatus* etc. in and around Delhi. Experiments were carried out to record main weeds in this crop and quantitate the losses caused by these in realizing the potential yield of cabbage, besides recording the rate of dissipation of its residues in soil, under cabbage. Soil and cabbage samples were extracted with acetone and three methods, known for residue analysis of pendimethalin [N-(1-ethylpropyl)-2,6-dinitro-3,4-xylidine] were evaluated to choose the best method by recovery experiments. The method giving highest recovery was used for residue analysis of samples.

The purpose of this study was to determine whether the herbicide and its metabolite translocated in the edible portion and for how long it remained in soil.

MATERIALS AND METHODS

Cabbage (var. Golden acre) was raised in the fields of the Indian Agricultural Research Institute, New Delhi in the month of November 2001. A randomized block design (RBD) with 3 replications and 3 treatments, including an untreated control, was used. The plot size was 10 m^2 (5 X 2 m^2) with plant to plant distance 40 cm and row to row distance also 40 cm. The number of ridges/plot was 5 and plants/ridge were 12.

Each replicate was further sub divided into two-sub replicate. One sub replicate was given one hoeing while the other was left without hoeing. Stomp 35 EC, a pendimethalin formulation was applied @ 1000 g a.i./ha (recommended dose) and 2000 g a.i./ha as T_1 and T_2 respectively, as a pre emergent herbicide to all sub replicates of T_1 and T_2. The population of various weeds was counted in each replicate of treated as well as untreated (control) plots before harvesting the crop. Fresh weight of individual weeds from each plot was taken. The weeds were then

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dried under sun for one day and then kept in an incubator at 70^{0}C for 2 days in brown paper bags. The dry weight of the weeds was also taken after drying.

For residue analysis, representative samples (0-15 cm depth) of soil were collected from 10 places from each replicate of each treatment, using an auger tube. The samples were collected on 0 (1 hr after application), 5, 10, 15 and 30 days after treatments. The soil samples were dried under shade by spreading them on paper overnight. The soil samples were sieved through a 2 mm mesh sieve and a sub sample of 20 g was taken by quartering method, in a 250 ml conical flask. Three drops of ammonia solution was added to each flask and was thoroughly mixed and then left till there was no smell of ammonia.

Three methodologies namely column extraction, Soxhlet and shaking methods were tried for residue analysis of pendimethalin in soil. The column extraction method (single step) gave over 98% recovery (Table 1). The soil sample was packed in a glass column along with 0.5 g each of Florisil and charcoal after mixing them properly. It was eluted with 100 ml hexane:acetone (1:1, v/v) solvent system. The eluent were rotary evaporated and then made up to 10 ml with freshly distilled hexane, and finally subjected to analysis using a Hewlett Packard 5890 GLC equipped with Ni-63 electron capture detector (ECD). The GLC parameters were - Column (megabore, 10 m length, 0.53 mm id with film thickness 2.65 μm), column temperature-190^{0}C (7 minutes, isothermal), injector temperature-250^{0}C, and detector temperature- 280^{0}C.

RESULTS AND DISCUSSION

The populations of major and minor weeds (recorded using a quartet, 1 m^2) are presented in Table 2. Among the weeds, *Coronopus didymus* was found to be the major weed with highest density 55.5 weeds/m^2 and bio-mass 135.2 g/m^2 followed by *Phalaris minor* in bio-mass (51.3 g/m^2) and in weed count, it was followed by *Melilotus indica* with 40 weeds/m^2 in control plot (T_3).

Population density of *C. didymus* was reduced to 10 weeds/m^2 with normal dose of application of pendimethalin and to 0.5 weeds/m^2 with double dose treatment in T_1 and T_2, respectively. *M. indica* and *C. sativa* density reduced to zero in T_2 treatment. Similar trend was observed when fresh biomass of weeds was recorded in treated and control plots. The fresh as well as dry weights of weeds were reduced, as their number was reduced. Less weed density means less biomass in all the treated plots.

The data in Table 2 about weed population and in Table 3 about yield clearly indicates that the herbicide is highly effective for weed control in cabbage. However the results displayed in Table 3 show that double the recommended dose is injurious to the crop as reported by Khatib *et al.* (1995) and should not be used. The weed population in sub replicate with hoeing was relatively less as compared to the one without hoeing, even in treated plots. Maximum yield was realized in treatment T_d where both pendimethalin application as well as manual weeding was done (Table 3).

The yield of cabbage from the plots treated with pendimethalin was higher as compared to the untreated plot (T_a). The yield data were statistically analyzed and it was observed that treatments within same replication were significantly different for two sub replicates.

Table1. Recovery of pendimethalin from soil for selecting best method.

Method used	Amount of pendimethalin added (μg)	Amount recovered (μg)				Recovery %
		R_1	R_2	R_3	Mean (± SD)	
Column extraction	10.0	9.7	9.9	9.6	9.8 (0.2)	98.0
	20.0	19.94	19.21	19.89	19.68 (0.4)	98.4
Shaking method	10.0	5.9	5.9	6.2	6.0 (0.2)	60.0
	20.0	12.0	12.65	11.98	12.21 (0.4)	61.01
Soxhlet extraction	10.0	6.3	6.5	6.6	6.4 (0.2)	64.0
	20.0	13.4	13.8	13.57	13.59 (0.2)	67.95

Table 2. Biomass and density of weeds under different treatments.

Weeds	Fresh bio mass (g/m^2)			Density (No./m^2)		
	T_1	T_2	T_3 Control	T_1	T_2	T_3 Control
Coronopus didymus	30.3 (7.2)*	1.1 (0.19)	135.2 (21.0)	10.0	0.5	55.5
Melilotus indica	4.6 (1.1)	Nil	6.1 (0.9)	1.5	Nil	40.0
Phalaris minor	44.1 (10.4)	6.8 (1.2)	51.3 (8.0)	2.0	1.0	7.5
Canabis sativa	0.2 (0.04)	Nil	9.4 (1.5)	0.05	Nil	3.2
Rumex dentatus	2.1 (0.5)	0.6 (0.1)	21.6 (3.4)	0.9	0.3	7.2
Anagallis arvensis	Nil	Nil	1.2 (0.2)	Nil	Nil	1.5
Total	81.3	8.6	224.9	14.45	1.7	114.9

*Figures in parenthesis indicate dry weight

The statistical analysis of yield data in Table 3 shows that CD calculated at 5% level is 3.624 Q/ha and at 1% level is 5.155 Q/ha. The CD at both 1% and 5% levels is quite lower than the difference of T_a, T_b; T_c, T_d; and T_d, T_e which indicates that treatments T_a, T_b; T_c, T_d; and T_d, T_e are significantly different from each other. In other words, treatments with one hoeing showed higher yield as compared to treatments without hoeing. As the treatment difference values are higher than CD even at 1% level, therefore the results are excellent as this model gives 99% accuracy. Treatment T_d (with highest yield) is significant from rest of the treatments at both the levels (1 and 5%). But treatment T_b and T_c are at par with each other. It indicates that application of recommended dose of pendimethalin without hoeing gives statistically similar yield as the treatment without herbicide but with one hoeing. This implies that pre-emergence application of pendimethalin can save labor cost required for hoeing. Similarly T_e and T_f are almost at par. In these two treatments, pendimethalin was applied only once at double its recommended dose. This double dose was, no doubt, able to control almost all the weeds but some phytotoxicity was observed to cabbage resulting in reduction in yield (fresh weight) of cabbage, in these two plots. Here yield is lower as compared to plots receiving recommended dose of pendimethalin. The higher dose suppressed the crop and so the size of cabbage heads formed were smaller resulting in lower yield. The average yield in treatment T_f is slightly higher than T_e because of one hoeing involved in T_f, which could overcome the effect of toxicity caused by applying double dose of pendimethalin.

Table 3. Yield data of cabbage crop.

Treatments	Replicates			Av.yield/ plot (kg)	Yield (Q*/ha)	
	R_1	R_2	R_3			
T_a (No treatment)	30.0	35.0	34.0	33.0	330.0	T_a–T_b=109.3
T_b (No treat + one hoeing)	46.4	43.1	42.3	43.93	439.30	T_b–T_c=5.60
T_c (1 kg a.i./ha)	43.8	40.6	45.7	43.37	433.70	T_c–T_d=54.6
T_d (1 kg a.i./ha + hoeing)	49.2	48.6	48.7	48.83	488.30	T_d–T_e=101.3
T_e (2 kg a.i./ha)	37.1	38.4	40.6	38.7	387.0	T_e–T_f= 20.3
T_f (2 kg a.i./ha + hoeing)	40.9	39.9	41.4	40.73	407.30	

$CD_{5\%}$= 3.624 *Q=100 kg

$CD_{1\%}$=5.155

The treatments T_1 (split into T_a and T_b) with highest number of weeds and therefore highest total biomass resulted in reduction of cabbage yield. Treatment T_1 is therefore giving least yield of cabbage crop. But treatment T_2 (split into T_e and T_f) showing least number of weeds and bio-mass also recorded less yield. The reason being the phytotoxicity caused by pendimethalin to the cabbage crop. Applying double the recommended dose of the herbicide causes more expense and yield of the produce is also lowered.

Residue analysis of soil showed that the average amount of herbicide recorded on 0-day (1 hr after application) was 9.03 mg/kg which dissipated to 2.30 mg/kg in 30 days, when applied at the recommended rate (Table 4). The herbicide was found to be persistent in soil (Kulshrestha *et al.*, 2000).

The residue data was subjected to analysis using Hoskins method (Hoskin, 1961) and half life was calculated using graphical method. Regression equations for normal (T_1) and double dose (T_2) were found to be Y = 2.9735 - 0.0202 X (R^2 =0.9968) and Y = 3.2109 - 0.0180 X (R^2 = 0.9936), respectively. Half life of the

Table 4. Persistence of pendimethalin residues in/on soil under cabbage cultivation.

Days	Residues (ppm)							
	T_1 (Treatment)				T_2 (Treatment)			
	R_1	R_2	R_3	Mean	R_1	R_2	R_3	Mean
0	8.95	9.21	8.94	9.033	14.89	15.9	15.86	15.55
5	7.25	7.07	8.99	7.77	13.72	14.05	13.98	13.916
10	5.75	6.09	5.91	5.916	10.53	10.18	10.94	10.55
15	4.42	4.65	5.18	4.75	9.01	8.98	8.89	8.96
30	2.34	2.11	2.47	2.306	4.39	4.56	4.91	4.62

herbicide for two rates of application was found to be 15 and 17 days, respectively. Analysis of log of residue data using Microstat also gave the same half-life. One metabolite of pendimethalin [N-(1-ethylpropyl)-3,4-dimethyl-2-nitrobenzene-1,6-diamine] was also synthesized in the laboratory as per Singh and Kulshrestha (1991), as it was reported to be formed in soil. Residues of pendimethalin and its metabolite were not detected up to the 10th day after transplanting, ensuring that the herbicide and its metabolite did not translocate in edible portion of cabbage. Use of pre-emergent application of herbicide along with one manual weeding/hoeing could be recommended for cabbage cultivation.

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