Indian J. Weed Sci. **42** (1 & 2) : 1-8 (2010)

Residues of Sulfosulfuron, Mesosulfuron+Iodosulfuron and Pinoxaden in Soil, Wheat and Successive Crops

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ABSTRACT

The experiment was conducted at the Research Farm, Department of Agronomy, PAU, Ludhiana, during the **rabi** seasons of 2006-07 and 2007-08 to study the effect of different planting patterns and straw management techniques on residues of different herbicides in soil at different intervals and grain and straw at harvest and on growth and development of few test crops through bioassay studies. The experiment was laid out in strip plot design with planting patterns of wheat in main plots and weed control treatments in sub-plots. Herbicidal treatments were applied as post-emergence at their respective doses. Residues of sulfosulfuron, mesosulfuron+iodosulfuron and pinoxaden were detected at 1 day after spray in soil depth 0-15 cm only but residues of herbicides were not detected in soil at other observational periods i. e. 30 and 60 DAS and at harvest as well as in grain and straw at harvest by analytic method. Nine **kharif** season crops viz., maize, bajra, sorghum, cotton, summer moong, bhindi, *dhaincha*, bottle gourd and muskmelon were sown after harvesting of wheat in four replications for bioassay studies. Bioassay studies indicated no residual toxicity of pinoxaden on any test crop. However, application of sulfosulfuron and mesosulfuron+iodosulfuron showed residual toxicity on maize, bajra, sorghum and bottle gourd, whereas no residual toxicity of these two herbicides was observed in cotton, summer moong, bhindi, *dhaincha* and muskmelon.

Key words : Bioassay, wheat, sulfosulfuron, mesosulfuron+iodosulfuron pinoxaden, HPLC

INTRODUCTION

Wheat is one of the premier and widely cultivated cereal crops of the world. In India, it is the second most important source of staple food next to rice and covers an area of 26.54 m ha with a production of 74.9 mt and an average yield of 26.71 q/ha (Anonymous, 2006). In Punjab, wheat was grown on an area of 3.49 m ha with a total production of 15.7 mt and average yield was 45.07 q/ha during 2007-08 (Anonymous, 2008).

Wheat fields in north India are badly infested with wide range of grassy and non-grassy weeds, in general, and *Phalaris minor* in particular. Due to morphological similarity, this weed escapes manual weeding and hence its control through selective herbicides is very important, otherwise drastic reduction in yield is expected. Many herbicides have been recommended on wheat crop. Generally farmers use sulfosulfuron and mesosulfuron + iodosulfuron to control *P. minor* and broad leaf weeds in wheat, which belong to sulfonylurea group of herbicides and it is considered to be very long residual group of herbicide. Continuous use of single herbicide may lead to build-up of residues in the soil which may harm the succeeding crops. Walker *et al.* (1989) reported that sulfonylurea group of herbicides is highly persistent in soil and may cause residual effect on the succeeding crops like sugarbeet, red beet, lucern, etc. Ideally, an herbicide should remain biologically active long enough to provide satisfactory weed control at least upto critical period of crop-weed competition and after that period, it must degrade into non-toxic compounds both in soil and plant biomass.

Keeping this in view, the residue of herbicides was estimated by analytic methods and through bioassay.

MATERIALS AND METHODS

A field experiment was carried out during **rabi** seasons of 2006-07 and 2007-08 at Research Farm, Department of Agronomy, Punjab Agricultural University, Ludhiana. The experiment consisting of five planting patterns viz., Zero till sowing with Happy Seeder (combine harvested), Zero till sowing in standing stubbles

(loose straw removed), Zero till sowing (complete burning of rice straw), Bed sowing (rice straw removed) and Conventional tillage (partial burning of rice straw) in main plots and weed control treatments viz., sulfosulfuron 25 g/ha, mesosulfuron+iodosulfuron 14.4 g/ha, pinoxaden 50 g/ha and control (unweeded) in sub-plots was laid out in strip plot design with four replications. The sowing of wheat variety PBW 502 was done on 31 October, 2006 and 9 November, 2007 with tractor drawn zero till/Happy Seeder/ordinary drills as per treatment using seed rate of 100 kg/ha. Herbicidal treatments (sulfosulfuron, mesosulfuron + iodosulfuron and pinoxaden) were applied as post emergence (after first irrigation) 35 days after sowing at their respective doses as per treatments. Spraying was done with the help of Knapsack sprayer fitted with flat fan nozzle using 250 liters of water/ha.

The other agronomic practices were carried out regularly. After the application of herbicide, the soil samples were collected from the experimental plots at 1, 30 and 60 days after spray and at harvest from each plot from 0-15 and 15-30 cm depth with post-hole auger. The soil samples were ground, sieved and subjected to analysis. Samples of wheat grain and straw were taken at harvest.

Method of Extraction

1. Sulfosulfuron

Fifty gram of ground sample of soil, grain or straw was weighed into a stoppered conical flask and extracted with 100 ml of distilled water : acetonitrile mixture (1:1) over an end-over-end mechanical shaker for 30 min. The sample was filtered into 500 ml round bottom flask and rinsed with 50 ml of the same solvent. Then the volume was reduced to 50 ml in a rotary evaporator.

Partitioning : The sample was transferred to a clear 250 ml separatory funnel and was extracted with 50 ml portions of methylene chloride each time. The combined extract was collected and concentrated to dryness by rotary evaporation.

Cleanup : A glass column packed with 2 g florisil was washed twice with 5 ml portions of 2% methanol in methylene chloride followed by 5 ml of isooctane twice. After the conditioning of the column, the sample was redissolved in 2 ml of methylene chloride and diluted with 8 ml isooctane. The entire sample was then transferred into the column; sulfosulfuron was eluted with 40 ml of 2% methanol in methylene chloride at a rate of 1 ml/min. The collected elute was evaporated to dryness by rotary evaporator.

HPLC : The sample was redissolved in 3 ml of 10% acetonitrile in water, filtered and analyzed by HPLC using method of sulfosulfuron developed by Ramesh and Thulasiramaraja (2003).

2. Mesosulfuron+Iodosulfuron

The representative sample (50 g) of each type of soil and grain and 10 g in case of straw was accurately weighed into a 500 ml Erlenmeyer flask. Then extracted with 50 ml acetonitrile : water (70 : 30) over an end-over-end shaker for about 30 min. The extract was filtered through Whatman No. 41 filter paper and later on concentrated to a smaller volume using vacuum rotary evaporator. The sample was dissolved in mobile phase and then analysed by HPLC by Bayer Crop Science Limited.

3. Pinoxaden

The representative sample (50 g) of each type of soil and grain and 10 g in case of straw was accurately weighed into a 500 ml Erlenmeyer flask. Then extracted with 100 ml of 1 M HCl/acetonitrile solution and reflux for 2 h on a heating mantle with condenser. The extract was cooled to room temperature.

Filtration : Three per cent ammonia solution was added till the pH of the solution was adjusted to pH 3-4. The solid matter was allowed to precipitate.

Solid phase extraction : The samples were transferred on to a solid phase extraction cartridge washed with 2 ml alcohol under vacuum. The samples were drawn along with 1 ml of water rinsing under vacuum at a rate of 2 ml/min. About 2 ml of hexane was added to a solid phase extraction cartridge and drawn through vacuum at a rate of 2 ml/min to the top frit. The column eluates were discarded.

Screw capped glass vials were placed to the

SPE cartridge and 3 ml of dichloromethane : ethyl acetate : formic acid (80 : 20 : 0.5) mixture was added and drawn under the vacuum at a rate of 2 ml/min collecting the column eluates. High vacuum for approximately 5-10 sec to remove any remaining solvent was applied from the cartridges. The residue in this fraction was eluated.

One ml of HCl was added to the eluate fractions and mixed thoroughly by capping the vials and shaking vigorously. The samples were evaporated under a stream of dry air (< 45°C) and the final volume of the sample was adjusted to 2 ml using mobile phase by using HPLC method developed by Syngenta India Ltd.

Chromatographic separation parameters for sulfosulfuron, mesosulfuron+iodosulfuron and pinoxaden were analysed by HPLC.

HPLC chromatograph parameters

Instrument	Waters High Performance Liquid Chromatograph Model Water 2487 Dual
Detector	UV-VIS detector
Column	Phenomnex C_{18} (25 cm length × 4.6 mm i. d.)
Detector sensitivity	0.01 A. U. F. S.
Volume injected	10 µl using fixed loop Rheodyne injector

HPLC chromatograph parameters	Sulfosulfuron	Mesosulfuron+ iodosulfuron	Pinoxaden
Flow rate (ml/min)	1.50	1.00	1.00
Mobile phase (Acetonitrile : water)	70 : 30	70 : 30	60 : 40+1 ml formic acid/l
Wavelength (nm)	220	220	254

Method of Calculation

During the process of routine sample analysis, wherever necessary, the concentration of sample solution was adjusted and injected. To assure the integrity of the samples, analytical standards were injected after each seven sample injections. The residues were determined by using the below given formula :

$$\text{Residue concentration } (\mu g/g) = \frac{A_1}{A_2} \times \frac{V}{W} \times C$$

Where,

A_1=Peak area of sample (µV-sec)
A_2=Peak area of standard (µV-sec)
V=Volume of sample extracts (ml)
W=Weight of the sample (g)
C=Concentration of herbicide (ppm)

Through Bioassay Studies

Nine **kharif** season crops viz., maize (*Zea mays* L.), bajra (*Pennisetum typhoides* L.), sorghum (*Sorghum bicolor* L.), cotton (*Gossypium hirsutum*), summer moong (*Vigna radiata* L.), bhindi (*Abelmoschus esculentus* L.), *dhaincha* (*Sesbania aculeata*), bottle gourd (*Lagenaria siceraria*) and muskmelon (*Cucumis melo*) were sown on 17 April, 2007 and 2008 after harvesting wheat in four replications. Five plants of each test crops were selected randomly in each plot and their height was recorded at the time of termination of bioassay studies i. e. after 40 days after sowing. Ten plants/crop were selected randomly and the fresh weight of above ground portion of these plants was taken 40 days after sowing during both the years and expressed in g/plant.

RESULTS AND DISCUSSION

The details of sample analysis and the residues of the herbicides under all the planting patterns i. e. zero till sowing with Happy Seeder, sowing in standing stubbles, zero till sowing, bed sowing and conventional tillage, respectively and at different soil depths (0-15 and 15-30 cm) are presented in Tables 1 to 3. Sulfosulfuron, mesosulfuron+iodosulfuron and pinoxaden were found to exist in the 0-15 cm soil depth at 1 day after the spray. At this time, concentration reduced to less than 0.02, 0.001 and 0.01 ppm in case

Table 1. Residue of sulfosulfuron, mesosulfuron+ iodosulfuron and pinoxaden (ppm) in soil (0-15 and 15-30 cm depth) at different intervals as influenced by planting patterns of wheat during 2006-07 and 2007-08

Herbicide	Planting pattern	2006-07								2007-08							
		Days after spray								Days after spray							
		1		30		60		At harvest		1		30		60		At harvest	
		Soil depth (cm)								Soil depth (cm)							
		0-15	15-30	0-15	15-30	0-15	15-30	0-15	15-30	0-15	15-30	0-15	15-30	0-15	15-30	0-15	15-30
Sulfosulfuron	Zero till sowing with Happy Seeder	0.03	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.03	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Zero till sowing within standing stubbles	0.02	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.03	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Zero till sowing after burning	0.03	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.05	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Bed sowing	0.03	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.04	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Conventional tillage after partial burning	0.04	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.03	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mesosulfuron	Zero till sowing with Happy Seeder	0.002	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.008	BDL	BDL	BDL	BDL	BDL	BDL	BDL
+iodosulfuron	Zero till sowing with in standing stubbles	0.005	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.008	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Zero till sowing after burning	0.005	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.008	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Bed sowing	0.004	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.006	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Conventional tillage after partial burning	0.002	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.008	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Pinoxaden	Zero till sowing with Happy Seeder	0.05	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.06	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Zero till sowing with in standing stubbles	0.05	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.08	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Zero till sowing after burning	0.07	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.06	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Bed sowing	0.05	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.06	BDL	BDL	BDL	BDL	BDL	BDL	BDL
	Conventional tillage after partial burning	0.06	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.06	BDL	BDL	BDL	BDL	BDL	BDL	BDL

BDL : Below detectable limit.
Minimum detectable limit for pinoxaden < 0.05 ppm.

Table 2. Residue of herbicides (ppm) in wheat grain and straw in different planting patterns and soil depths in wheat during 2006-07 and 2007-08

Planting pattern	At harvest					
	Sulfosulfuron		Mesosulfuron+iodosulfuron		Pinoxaden	
	Grain	Straw	Grain	Straw	Grain	Straw
Zero till sowing with Happy Seeder	BDL	BDL	BDL	BDL	BDL	BDL
Zero till sowing within standing stubbles	BDL	BDL	BDL	BDL	BDL	BDL
Zero till sowing after burning	BDL	BDL	BDL	BDL	BDL	BDL
Bed sowing	BDL	BDL	BDL	BDL	BDL	BDL
Conventional tillage after partial burning	BDL	BDL	BDL	BDL	BDL	BDL

BDL : Below detectable limit.

Minimum detectable limit for sulfosulfuron < 0.02 ppm, mesosulfuron+iodosulfuron < 0.001 ppm and pinoxaden < 0.05 ppm.

of sulfosulfuron, mesosulfuron+iodosulfuron and pinoxaden, respectively, which were the minimum detectable limit. Sulfosulfuron was reported to exist in the 0-15 cm soil depth upto five days after the spray. At subsequent samplings (30 and 60 days after spray and at harvest), the residue of the herbicides under all the main plot treatments and at the soil depths (0-15 and 15-30 cm) was found to be below 0.02, 0.001 and 0.05 ppm in case of sulfosulfuron, mesosulfuron + iodosulfuron and pinoxaden, respectively, which were the minimum detectable limit. Upon analysis of samples, no residues of sulfosulfuron, mesosulfuron+ iodosulfuron and pinoxaden were detected in soil 30 and 60 days after spray and at harvest (Tables 1 to 3) and in grain and straw (Table 4) as in none of the samples the peak was obtained at the respective retention time. There was lot of time gap between the time of herbicide application and sampling and lot of rainfall was received during this period and that might have helped in herbicide degradation and leaching down from surface layer. Secondly, the temperature was also high during the months of March and April that also helped in degradation of the herbicides. Saha *et al*. (2003) reported that sulfosulfuron was thermally unstable. Ramesh and Thulasiramaraja (2003) reported that wheat grain and soil samples collected at harvest showed no detectable residues of sulfosulfuron when analysed at the detection limit. Kaur (2005) and Singh (2007) reported that residues of sulfosulfuron and mesosulfuron+iodosulfuron were below the detection limit when samples were taken at the time of harvest.

Bioassay Studies

After the harvest of experimental wheat, nine succeeding crops viz., maize, bajra, sorghum, cotton, summer moong, bhindi, dhaincha, bottle gourd and muskmelon were sown in the each herbicide treated as well as untreated (control) plots immediately after wheat harvest in all the planting pattern plots after seed bed prepration. The trial was terminated 40 days after sowing after recording final plant height and fresh weight of 10 randomly selected plants and the results are discussed crop wise as under :

The differences in plant height and fresh weight/ plant recorded 40 days after sowing due to different planting patterns were found to be non-significant (Tables 3 and 4) in case of all the succeeding crops.

Among the weed control treatments, the application of sulfosulfuron, mesosulfuron+iodosulfuron to wheat resulted in significant reductions in plant height of succeeding maize, bajra, sorghum and bottle gourd crops indicating thereby residual effects of these herbicides (Table 3). Kaur (2005) reported that mesosulfuron+iodosulfuron caused slight residual toxicity to the succeeding maize crop. Singh *et al.* (2003) noticed residual toxicity of mesosulfuron+iodosulfuron at 15 and 30 g/ha applied to wheat on the growth of succeeding maize crop. Plant height of succeeding bajra crop (Kaur *et al.*, 2007), sorghum crop (Brar *et al.*, 2007) and bottle gourd (Singh and Walia, 2005) was significantly more in unsprayed plots as compared to the crop sown in the plots where sulfosulfuron and

Table 3. Effect of planting patterns and weed control treatments applied to wheat on plant height of bioassay crops

Treatment	Maize		Bajra		Sorghum		Cotton		Summer moong		Bhindi		*Dhaincha*		Bottle gourd		Muskmelon	
	I	II	I	II	I	II	I	II	I	II	I	II	I	II	I	II	I	II
Planting pattern																		
Zero till sowing with Happy Seeder	37.4	37.7	46.7	49.3	36.4	39.6	22.7	26.9	32.4	37.3	15.8	18.0	50.2	54.9	84.8	95.4	64.3	70.4
Zero till sowing in standing stubbles	37.3	37.3	46.0	47.4	35.6	39.0	21.8	26.8	32.0	37.2	15.0	17.5	50.4	53.7	84.3	94.6	64.7	70.2
Zero till sowing after burning	34.9	35.8	43.4	46.4	32.8	35.9	21.2	25.7	29.4	35.1	12.2	15.4	49.0	52.6	80.9	92.4	61.8	66.7
Bed sowing	35.8	36.2	45.5	47.0	35.4	37.4	22.9	27.7	31.9	36.8	14.7	17.0	51.9	54.6	82.4	94.1	63.6	68.4
Conventional tillage after partial burning	34.5	35.5	42.1	45.2	32.8	35.0	20.7	25.0	29.2	34.4	12.9	15.2	48.7	52.0	80.1	91.0	61.0	66.3
LSD (P=0.05)	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Weed control treatments																		
Sulfosulfuron	32.3	31.6	37.1	40.4	26.4	30.1	22.1	26.0	30.1	35.8	12.4	14.4	50.2	54.1	82.4	91.2	62.1	67.3
Mesosulfuron+iodosulfuron	30.0	29.8	35.2	37.0	23.2	27.2	21.6	25.5	29.0	34.4	10.4	14.0	50.3	53.9	79.2	88.4	60.2	66.0
Pinoxaden	39.8	41.5	44.6	48.2	36.4	40.2	22.9	26.7	31.4	36.2	14.3	16.4	51.2	54.8	94.2	100.1	63.4	69.0
Control (unweeded)	41.9	43.2	48.2	51.4	39.2	42.1	24.4	27.4	33.1	37.1	15.4	18.3	53.4	56.0	98.4	104.5	66.9	71.7
LSD (P=0.05)	2.2	2.4	5.5	4.8	3.9	3.4	NS	NS	NS	NS	NS	NS	NS	NS	5.0	5.4	NS	NS

Interactions : All interactions are non-significant. I–2006-07, II–2007-08.

Table 4. Effect of planting patterns and weed control treatments applied to wheat on fresh weight of bioassay crops

Treatment	Maize		Bajra		Sorghum		Cotton		Summer moong		Bhindi		*Dhaincha*		Bottle gourd		Muskmelon	
	I	II	I	II	I	II	I	II	I	II	I	II	I	II	I	II	I	II
Planting pattern																		
Zero till sowing with Happy Seeder	23.4	32.1	18.4	20.4	19.4	21.4	10.9	13.9	14.2	18.4	15.8	18.0	19.7	23.7	148.2	164.5	90.2	70.7
Zero till sowing in standing stubbles	23.0	33.3	17.8	20.1	17.8	21.1	10.4	12.6	13.8	18.1	15.0	17.5	18.8	22.8	146.4	162.4	90.0	70.4
Zero till sowing after burning	20.9	30.7	15.6	16.4	15.4	18.4	9.0	11.9	11.2	16.2	12.2	15.4	18.3	21.1	143.9	160.5	87.3	66.7
Bed sowing	21.8	31.6	17.0	19.9	17.1	20.5	10.1	12.1	13.1	17.6	14.7	17.0	19.3	22.6	145.2	162.3	90.4	68.4
Conventional tillage after partial burning	20.5	30.2	15.4	15.2	14.1	18.0	9.5	11.2	11.0	15.2	12.9	15.2	17.7	20.0	143.4	160.4	87.1	66.4
LSD (P=0.05)	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Weed control treatments																		
Sulfosulfuron	19.3	25.3	15.3	17.4	14.5	18.8	10.5	12.3	12.9	16.2	12.4	14.4	18.0	22.8	144.3	165.3	91.2	107.3
Mesosulfuron+iodosulfuron	17.0	23.4	12.6	16.0	11.0	15.5	10.1	11.3	12.0	14.4	10.4	14.0	17.5	20.1	137.2	158.0	89.0	103.4
Pinoxaden	24.8	37.8	23.1	25.4	21.1	25.4	11.7	12.4	14.4	18.1	14.3	16.4	18.8	23.7	168.4	192.1	92.3	109.4
Control (unweeded)	26.9	39.9	24.4	26.3	22.6	28.2	13.2	14.2	15.4	19.5	15.4	18.3	19.9	26.7	171.1	195.4	95.3	112.0
LSD (P=0.05)	2.1	2.2	2.7	2.3	4.0	4.0	NS	NS	NS	NS	NS	NS	NS	NS	3.8	4.1	NS	NS

Interactions : All interactions are non-significant. I–2006-07, II–2007-08.

mesosulfuron+iodosulfuron were applied to wheat. Plant height of bhindi (*okra*), cotton, summer moong, bhindi and *dhaincha* recorded 40 days after sowing was found to be non-significant when raised after the application of sulfosulfuron and mesosulfuron+iodosulfuron to wheat (Preet, 2006).

Lower fresh weight of maize was observed when it was grown after the application of sulfosulfuron and mesosulfuron+iodosulfuron which was significantly low when sown in control (unsprayed plots) as well as after the application of pinoxaden 50 g/ha (Table 4). Yadav *et al.* (2004) also reported residual toxicity by sulfosulfuron at 25 and 50 g/ha applied to wheat on the succeeding maize crop. The fresh weight of bajra was significantly higher in the plot sown after the application of pinoxaden compared to sulfosulfuron and mesosulfuron+iodosulfuron. The fresh weight of maize/ plant growing in no residue condition (unsprayed plots) and sprayed with pinoxaden was significantly higher than the crop succeeding wheat treated with sulfosulfuron and mesosulfuron+ iodosulfuron indicating residual effect of these herbicides. Brar *et al.* (2007) noticed residual toxicity of sulfosulfuron applied to wheat on the growth of succeeding sorghum crop. Similarly, no residual toxicity of sulfosulfuron, mesosulfuron+ iodosulfuron and pinoxaden on cotton was reported by Kaur *et al.* (2007). The fresh weight of bottle gourd was significantly higher in the plot sown after the application of pinoxaden than sown after the application of sulfosulfuron and mesosulfuron+iodosulfuron. Singh and Walia (2005) also reported that bottle gourd was quite sensitive crop towards higher dose of sulfonylurea herbicide. The differences due to variable weed control treatments were found to be non-significant with respect to fresh weight of *dhaincha* (Table 4).

Interaction effects for plant height and fresh weight per plant due to planting patterns and weed control treatments were found to be non-significant.

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