Bull. Environ. Contam. Toxicol. (1995) 55:276–282

Environmental Contamination and Toxicology

Residues of Fluazifop-*p*-butyl Following Application to Soybean

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Received: 18 May 1994/Accepted: 5 January 1995

Fluazifop-p-butyl [(R) Butyl (2-(4-(5-(trifluoromethyl) -2-pyridyloxy) phenoxy) propionate)], the R-stereoisomer of fluazifop butyl (racemic mixture of R and S enantiomers) is the active herbicide. The chemical was introduced by ICI under a code No. of PP005 and the trade name Fusilade.

This potent selective herbicide is applied post-emergent in several pulses, oilseed crops and vegetables (potato, onion etc.) to control grasses (Worthing, 1989; Wigfield and Lanouette, 1993). Fluazifop-p-butyl applied under field conditions degraded rapidly to fluazifop (Raut and Kulshrestha, 1992), which is bound in soil or conjugated in plants. The method used to determine the residues of fluazifop-p and its butyl ester in soil and crop involved acid or base hydrolysis of the butyl ester to the acid, multiple extraction steps, methylation of the acid to the methyl ester and Florisil cleanup followed by gas chromatographic determination (Clegg, 1987).

A detailed study was conducted on the persistence of fluazifop-p-butyl in/on soybean foliage and soil. Terminal residues were also estimated in soybean straw, grain, oil, cake and soil.

MATERIALS AND METHODS

Fluazifop-p-butyl (analytical grade, 85.6% purity) was provided by ICI India Ltd. The solvents were distilled prior use.

Soybean (Var. PK-327) raised in the experimental fields of Indian Agricultural Research Institute (IARI), New Delhi during Kharif 1990 was sprayed with 0.125 Kg a.i./ha (half dose), 0.250 kg a.i./ha (normal dose) and 0.500 kg a.i./ha (double dose) fluazifop-p-butyl (@ 500

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L/ha water), separately as post-emergent treatment (30 days after sowing the crop). Each treatment was replicated thrice in randomised block design.

A similar experiment was conducted with soybean in the field of Jawaharlal Nehru Krishi Vishvavidyalay (JNKVV) in Kharif 1989 and 1990 with the same three doses of fusilade II in 250 L/ha water.

Foliage and pod samples were drawn 1, 2, 3, 7, 14, 30 and 60 days after spray from the farm of IARI, New Delhi. Grain and straw samples were collected at the time of harvest. Grain samples were also collected at the harvest from the experimental fields of JNKVV, Jabalpur. Soil samples were collected randomly from 0-7.5 and 7.5-15 cm depths at 0, 1, 2, 3, 7, 14, 30, 60 and 90 days after application (DAA) from experimental fields of IARI, New Delhi.

A representative sample (20g) was taken for analysis of fluazifop-p-butyl residues. Soybean foliage were extracted with methanol (3X50 ml) in a waring blender. The contents were mixed with an equal amount of 0.2 N NaOH and were maintained at 60 ^{o}C on water bath for 1 h. The contents were filtered through Buchner funnel and extracts combined. The cleanup was done by partitioning with hexane (2X50 ml) followed by acidification with 6 N HCl (pH 2.5) and extraction with methylene chloride (3X50 ml). The combined extracts were concentrated on rotary evaporator to about 5 ml. Soybean straw and grain were extracted and cleaned up as mentioned for soybean foliage. Soybean oil was obtained from grain samples by extracting with hexane in soxhlet apparatus. Oil and cake were extracted with 0.2 N NaOH-methanol (1:1) mixture as described for grain.

The soil samples were extracted with 0.2 N NaOH-methanol (1:1) mixture (3X50 ml) by shaking on mechanical shaker for half an hour and maintaining at 60 ^{o}C on water bath for 1 h. The cleanup was done by partitioning with hexane as described above.

Fluazifop-p-acid in the sample extracts was derivatized to methyl ester by reacting overnight with diazomethane followed by column cleanup on Florisil with 5% diethyl ether in methylene chloride solvent system.

Residues were determined by gas liquid chromatography (GC) using Hewlett Packard (Model 5890A) gas chromatograph equipped with 2 M X 2 mm i.d. glass column packed with 3% OV-101 on 100-200 mesh chromosorb

^{63}W and ^{63}Ni electron capture detector using following Parameters, Temperature (^{o}C): Injection port 300, column 250, detector 300 and high purity nitrogen carrier gas (60 ml/min). The efficiency of extraction, cleanup and estimation procedure were checked by recovery experiments. The recovery values ranged from 85 to 92 percent.

The meteorological data for the period of experiment has been presented in Table 1. The rate of

Table 1 . Meteorological data from July-October 1990

month	Fort-night	Av.temperature (^{o}C)		Humidity (% mean)	Total rainfall (mm)
		Maximum	Minimum		
July	I	33.3	26.4	80.5	79.8
	II	34.8	25.9	74.6	56.0
August	I	34.5	25.5	82.0	78.8
	II	33.9	25.8	73.7	211.8
September	I	30.7	25.0	77.1	162.2
	II	32.9	23.8	76.1	74.8
October	I	33.1	19.7	72.2	0.0
	II	30.6	11.7	61.6	0.0

dissipation of fluazifop-p-butyl residues was worked out by determining RL_{50} values.

RESULTS AND DISCUSSION

The residue data of flauzifop-p-butyl from double dose on soybean foliage is presented in Table 2. The initial deposit of 2.37 ppm herbicide on soybean foliage declined to 1.98 ppm after 1 day of application and further degraded to 1.0 ppm after 7 days and 0.65 ppm after 14 days, thus representing a loss of 16.45, 57.80 and 72.57%, respectively. Residues following one and two months of application were 0.07 and 0.01 ppm, respectively and represented dissipation of 97.05 and 99.58%, respectively. The calculated half-life of fluazifop-p-butyl in soybean foliage was 7.9 days.

Table 2. Dissipation of fluazifop-p-butyl from double dose in/on soybean foliage

Days	Residues ± S.D.* (ppm)	Dissipation (%)
0	2.37 ± 0.23	-
1	1.98 ± 0.21	16.45
2	1.73 ± 0.12	27.00
3	1.27 ± 0.08	46.41
7	1.00 ± 0.05	57.80
14	0.65 ± 0.02	72.57
30	0.07 ± 0.01	97.05
60	0.01 ± 0.001	99.58
90	BDL	100.00

Regression equation : Y = -0.310 - 0.0381 X ;
r = 0.960, Half-life : 7.9 days

* Average of three replicates

Maximum Residue Limit (MRL) of fluazifop-p-butyl on soybean has not been assigned by FAO.

The residue data of fluazifop-p-butyl in 0-7.5 and 7.5-15.0 cm soil depth has been presented in Table 3. The initial deposit of 0.21 ppm from half dose of fluazifop-p-butyl in soil dissipated to 0.18 ppm after 1 day of application and further degraded to 0.07 ppm at 14th day and 0.01 ppm at 60th day. The percent dissipation during these days was 14.09, 81.22 and 99.07, respectively (Table 4). With normal dose, the initial deposit was 0.60 ppm which declined to 0.52, 0.22 and 0.06 ppm on 1st, 14th and 60th day of application. The initial deposit with double dose was 1.40 ppm which disappeared to 1.13, 0.47 and 0.11 ppm after 1, 14 and 60 days of application.The percent dissipation of the herbicide on these days from the highest dose was 19.13, 66.5 and 92.2, respectively. The statistically calculated half life values for the half, normal and double dose were 7.2, 6.2 and 7.2 days, respectively (Table 5) during initial stage (0-

Table 3. Residues of fluazifop-p-butyl in surface and sub-surface soil

Days	Depth (cm)	Residues* (ppm)		
		0.125 kg/ha	0.250 kg/ha	0.500 kg/ha
0	0-7.5	0.213	0.604	1.399
1	0-7.5	0.183	0.523	1.130
2	0-7.5	0.154	0.447	1.076
3	0-7.5	0.075	0.305	0.881
	7.5-15	0.043	0.071	0.129
7	0-7.5	0.060	0.183	0.523
	7.5-15	0.031	0.102	0.122
14	0-7.5	0.054	0.131	0.362
	7.5-15	0.014	0.090	0.106
30	0-7.5	0.030	0.079	0.104
	7.5-15	0.010	0.033	0.059
60	0-7.5	0.009	0.044	0.088
	7.5-15	0.002	0.017	0.021
90	0-7.5	ND	0.010	0.031
	7.5-15	ND	0.002	0.005

* Average of three replicates

14 days) and 17.7, 21.3 and 24.6 days, respectively during subsequent period (14-90 days). The rates of dissipation pattern of fluazifop-p-butyl in soybean field soil were found to follow first order kinetics at all the three rates of treatment during initial period as also evident from the similar half life values.

Fluazifop-p-butyl from double dose was found to translocate in minute quantities in soybean grain. Herbicide from all the three doses leached down in small amounts to 15 cm zone as could be seen from data. The residue data obtained for soybean grains, oil, cake, straw and soil at IARI, New Delhi are presented in Table 6.While residues from two lower doses were below detectable level (BDL),the residues from double dose in grain, oil, cake and straw were 0.005 ppm, BDL, 0.001 ppm and BDL, respectively.

Table 4. Perercent dissipation of fluazifop-p-butyl in IARI soil

Day	Dissipation of fluazifop-p-butyl (%)		
	0.125 kg/ha	0.250 kg/ha	0.500 kg/ha
0	-	-	-
1	14.09	13.42	19.23
2	27.70	26.00	23.09
3	44.66	37.75	27.81
7	57.28	52.82	53.90
14	81.22	63.41	66.55
30	95.31	81.46	88.35
60	99.07	89.91	92.21
90	ND	98.02	97.43

Table 5. Half -life, correlation coefficient and regression equation of fluazifop-p-butyl in soil

Rate (kg a.i./ha)	Regression Equation	Correlation Coefficient	Half Life
0-14 days			
0.125	Y = -0.78-0.042 X	0.840	7.2 days
0.250	Y = -0.28-0.048 X	0.950	6.2 days
0.500	Y = -0.10-0.042 X	0.970	7.2 days
14-90 days			
0.125	Y = -1.02-0.017 X	0.990	17.7 days
0.250	Y = -1.02-0.017 X	0.980	21.3 days
0.500	Y = -0.40-0.012 X	0.940	24.6 days

Residues were below detectable level in grain, oil and cake in soybean grain samples obtained from JNKVV, Jabalpur in 1989 and 1990.

The residues in soil at the time of harvest due to spray of fluazifop-p-butyl to soybean crop were found to be below detectable limit from half dose, 0.012 ppm from normal dose and 0.036 ppm from double dose in 0-15 cm depth.

Table 6. Terminal residues of fluazifop-p-butyl in soybean grain, oil, cake, straw and soil at IARI, New Delhi

Substrate	Average Residues (ppm)*		
	0.500 kg/ha	0.250 kg/ha	0.125 kg/ha
Grain	0.005	BDL	BDL
Oil	BDL	BDL	BDL
Cake	0.001	BDL	BDL
Straw	BDL	BDL	BDL
Soil	0.036	0.012	BDL

* Average of three replicates, BDL Below detectable level

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