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Dissipation of Sulfosulfuron in Different Soils under Laboratory Conditions

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Abstract

The safe and efficient use of sulfosulfuron would require knowledge of its dissipation behavior in different soil. To accomplish this task, a suitable and sophisticated method of estimation by HPLC was standardized. The dissipation behavior of sulfousulfuron at 25 and 50 g/ha was studied in three different soils under laboratory conditions. Compared to zero day incubation, the degradation in different soils applied at 25 g/ha at 30 days was 33 to 40%, whereas it was 44-53% at 50 g/ha. The degradation of sulfosulfuron was of first order kinetics. After four months of application, sulfosulfuron applied at 25g/ha was found to degrade to the extent of 87.5, 91.9 and 89.6% in loamy sand, sandy loam and clay loam soils, respectively.

Key words : Sulfosulfuron, Different soils, Incubation, HPLC.

Sulfosulfuron at 25g/ha applied as post-emergence provides satisfactory control of P minor with 50 to 70% additional control of major broadleaf weeds in wheat. However, the safe and efficient use of herbicides including sulfosulfuron would require knowledge of their dissipation behavior in the soil. Saha and Kulshrestha (1) studied that the degradation of sulfosulfuron is influenced by abiotic factors. Saha et al. (2) standardized the method of estimation of residues of sulfosulfuron by HPLC. Saha et al. (3) also studied the residual persistence of sulfosulfuron and reported its half life equal to 5.3 days. Previous methods based on GC have relied on detection of breakdown products due to the thermal instability of sulfonylurea. However, the detection of sulfonylurea by derivatization were shown to be suitable as the basis for a method for chlorsulfuron and metsulfuron-methyl in soil and water but inferences were a problem with plant matrices. Hence, to get rid of the problem of thermal instability, there is a need to develop a suitable and sophisticated method of estimation by liquid chromatography. From the cited literature, it is clear that so far, no systematic studies have been undertaken on persistence of sulfosulfuron in Indian soils. Therefore, the present studies were undertaken to assess the dissipation behavior of sulfosulfuron in different soils by high performance liquid chromatography during 2009-2010.

Methods

Three types of soil were collected from different locations i. e. Balasamand, Research Farm, CCS HAU, Hisar and RRS, Uchani, Karnal, which were loamy sand, sandy loam and clay loam in texture, respectively. (Table 1). All the three soils were low in available N, medium in P_2O_5 and K_2O with slightly alkaline in reaction. These soils were grounded, homogenized, sieved through 2 mm sieve and then treated with sulfosulfuron at 25 and 50g/ha. After proper mixing of treated soils, prepared a set of five polyethylene bags weighing 200 g soil each and marked to represent 0, 30, 60, 90 and 120 days incubations at 25 g/ha. Similarly prepared another set of 5 polyethylene bags for 50 g/ha for each type of soil representing different incubations. To each bag added measured quantity of water of water to keep the soil at field capacity (for loamy sand—22ml/100g, sandy loam-17ml/100g and clay loam-20 ml/100g) at room temperature. Now defreeze the zero-day samples (samples taken just after application of the herbicide) of all three treated soils for HPLC analysis. The rest of the soil sample were maintained at field capacity for the respective incubation period by weighing and adding measured quantity of water as per requirement. The soil samples after completion of each incubation period were kept in deep freezer before subjecting to further extrac-

Table 1. Physico-chemical properties of different soils.

Locations	Soil texture	Soil pH (1.2)	EC (ds/m)	Available N (kg/ha)	P_2O_5 (kg/ha)	K_2O (kg/ha)
Balasamand	Loamy sand	8.2	0.34	112.7	17	265
Research farm CCS HAU, Hisar	Sandy loam	8.4	0.22	123.9	14	240
R R S, Uchani, Karnal	Clay loam	8.0	0.17	149.3	10	284

tion, processing and HPLC analysis.

Procedure for Estimation of Residues

Apparatus used were as follows : Water's HPLC, equipped with PDA (photodiode detector array) detector, mechanical shaker, rotary vacuum evaporator, manifold evaporator, required glass wares. Reagents were Acetonitrile (GR), 1-M ammonium carbonate aqueous solution, anhydrous sodium sulfate (Na_2SO_4), 5% aqueous saline solution, dichloro methane (GR), silica gel (60x 120 mesh), activated charcoal, acetonitrile (HPLC grade), Water (HPLC grade).

Extraction of sulfosulfuron residues from soil. Twenty gram of air dried, grounded, homogenized and sieved through 2 mm sieve representative of above three different soils samples were taken in different erlenmeyer flasks. The extraction mixture solution 200 ml was prepared by mixing acetonitrile and 1-M ammionium carbonate aqueous solution in the 9 : 1 (vol/vol) ratio. The extraction process for the desired residue from each soil sample was carried out in three lots. In first lot, added 70 ml of this extraction mixture solution into every sample flask and kept on mechanical shaker for 30 minutes. Filtered the above extract through cotton pad roofed with anhydrous sodium sulfate to remove the moisture from desired extract. Again repeated the same processing by adding second lot of (70 ml) extraction mixture in each of the same sample flask and kept on mechanical shaker for 30 min and repeated the same steps of sample processing as in first case. Similarly, repeat the same processing steps by adding third lot of the extraction mixture solution. The extraction mixture solutions were prepared fresh for each processing sample. Combined the filtered extracts and reduced its volume up to 20 ml by rotary flash evaporator.

Clean Up. Then the concentrated extracts of three soils diluted with (four to five times of its volume) 5% aqueous saline solution i.e. 100 ml added to each sample and made the total volume about 120 ml. Partitioned these extracts by separating funnel with 50 ml of dichloromethane (3) and collected the organic layers after passing through anhydrous sodium sulfate to remove any remaining moisture. The volume of organic layers reduced up to 15 ml by rotary flash evaporator. To remove the remaining co-extracts, column chromatographic technique was followed for complete clean up of the sample. The chromatographic column was packed with the homogeneous mixture of silica gel (5g) and activated Charcoal (0.5g) between the two layers of anhydrous sodium sulfate of two inches each. Pre-wet the column with 40 ml of dichloromethane and placed this concentrated extract at the top of the column and elute with 150 ml of dichloromethane. The eluent was concentrated by rotary flash evaporator and dried with manifold evaporator. Finally, the desired volume of the dried and cleaned sample was prepared in selected and desired mobile phase i. e. acetonitrile : water in the 9 : 1 ratio and injected the 20μl sample into HPLC column for analysis.

Standardization of Method of Estimation of Sulfosulfuron from Three Soils

Calibration Curve. The stock standard solution of sulfosulfuron was prepared at concentration 100 μg /ml in acetonitrile (HPLC grade) and from this stock solution prepared standard solutions of different concentrations i.e. 10, 5, 2.5, 1 and 0.5 μg/ml. All these standard solutions of different concentrations run on HPLC and got the chromatograms at same retention time (R_t). R_t is the base of identification of desired chemical. From this data, the calibration curve was prepared for the integration of unknown samples.

Recovery Experiment. For the authenticity of the method of estimation, recovery experiment is manda-

Table 2. Residues of sulfosulfuron in different soils at various incubation periods. * Percent loss of herbicide at different incubations compared to 0 day incubation.

Soil types	Sulfosulfuron applied (g/ha)	Residues of sulfosulfuron (µg/g) at different incubation periods (days)				
		0	30	60	90	120
Balasamand	25	0.0120	0.0072	0.0036	0.0029	0.0015
			(40.0)*	(70.1)	(76.1)	(87.5)
(loamy sand)	50	0.0245	0.0124	0.0062	0.0045	0.0026
			(48.4)	(74.7)	(81.6)	(89.4)
Research farm CCS HAU,	25	0.0111	0.0070	0.0027	0.0015	0.0009
Hisar (sandy loam)			(36.9)	(75.70	(86.5)	(91.9)
	50	0.0225	0.0125	0.0050	0.0030	0.0015
			(44.4)	(77.7)	(86.7)	(93.3)
R R S Uchani,	25	0.0115	0.0077	0.0032	0.0022	0.0012
Karnal			(33.0)	(72.2)	(80.8)	(89.6)
(clay loam)	50	0.0235	0.0111	0.0049	0.0037	0.0023
			(52.7)	(79.1)	(84.3)	(90.2)

tory. Fifty gram of grounded and homogenized sieved three different soils were taken in different flasks and fortified each soil sample by adding standard solution of known concentration i. e. fortification level was 1 µg/g. These samples were mixed well to form slurry and kept for evaporation up to dryness of the acetonitrile. Each of the fortified soil sample was processed, extracted and cleaned by following the aforesaid procedure for HPLC analysis. The recovery obtained was 93—98% in loamy sand, 88—90% in sandy loam and 92—94% in clay loam at fortification level of µg/g.

HPLC Analysis

The selected/desired mobile phase was prepared by mixing the acetonitrile : water (HPLC grade) in the ratio of 9 : 1 and degassed to remove the dissolved air in it prior to run on HPLC. The above processed and cleaned soil samples were dissolved in mobile phase and filtered through 0.45 µm membrane filter prior to injection into HPLC column. The fortified and experimental soil samples were run on HPLC under mentioned conditions and identified the residual chromatogram at same R_t,

Water's HPLC Operating Parameters

HPLC column : XTerra RP18 5µm 4.6 × 250mm
Detector : Photodiode Array (PDA 996)
Mobile phase : acetonitrile : Water (90 : 10)
Flow rate : 0.8 ml/min
Volume injected in column : 20µl
Adsorption maxima (λ_{max}) : 212nm.
Detection limit (sensitivity) : 0.05 µg/g
Retention time (R_t) of sulfosulfuron : 3.7 minutes

Results and Discussion

Compared to zero day incubation, the degradation in different soils applied at 25g/ha at 30 days was 33 to 40%, whereas it was 44-53% at 50g/ha (Table 2). The trend of dissipation at 60 days was almost identical to that of obtained at 30 days, and it was 70—76% at 25 g/ha and 75—79% at 50g/ha. This data clearly indicates that the degradation of sulfosulfuron is of first order kinetics as the magnitude of dissipation was governed by the amount of herbicide available in the soil at that point of time. The degradation was slowed down after 60 days in all soils and it was only 5—10 % up to 90 days and similar thereafter. At 120 days, the residues of sulfosulfuron applied at 25 and 50 g/ha varied from 0.0009 to 0.0015 µg/g and 0.0020 to 0.0026 µg/g, respectively. After four months of application, sulfosulfuron applied at 25g/ha was found to degrade to the extent of 87.5, 91.9 and 89.6% in loamy sand (Balasamand), sandy loam (CCSHAU, Farm) and clay loam (Uchani, Karnal), respectively. The corresponding figures at 50g/ha were 89.4, 91.1

and 90.2%. Based on present investigation, it was realized that the trend of sulfosulfuron dissipation in different soils was almost identical. This could be possible obviously because the experiment was carried out under controlled conditions where soil moisture and temperature were kept constant. However, dissipation behavior of sulfosulfuron in different soils could be different under field conditions as might be influenced by various levels of moisture, microbes, temperature.

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