

Residual fate and persistence behaviour of a mixed herbicide formulation (pyroxsulam 4.5% OD + sulfosulfuron 75% WDG) in wheat plant and field soil

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ABSTRACT

To find out the persistence behavior of a mixed herbicide formulation Pyroxsulam 4.5% oil dispersion (OD) and Sulfosulfuron 75% water dispersible granules (WDG), a supervised field study was conducted in B.C.K.V. experimental farm, Mohanpur, West Bengal during 2012-2013 and 2013-2014. The mixed herbicide formulation was applied @ 18 g and 36 g a.i. ha⁻¹ along with untreated control and was analyzed by LC-MS/MS in MRM mode. The method includes extraction with acetonitrile for Pyroxsulam and ethyl acetate for Sulfosulfuron separately followed by clean up using dispersive solid phase extraction (d-SPE) with PSA, GCB (only for plant) and activated MgSO₄. The ethyl acetate extract was evaporated under a nitrogen evaporator and reconstituted with acetonitrile. The average recovery percentage of Pyroxsulam and Sulfosulfuron was 87.67% and 85.07% for wheat plant, 88.87% and 87.40% for wheat straw, 85.67% and 90.80% for wheat grain and 86.27% and 86.00% for field soil respectively. Following 1st order kinetics, the half-life of Pyroxsulam and Sulfosulfuron was found in the range of 1.51-1.67 days and 1.68-2.95 on wheat plant and 1.95-2.84 days and 2.89-3.58 on field soil irrespective of dose and season. No residue of Pyroxsulam and Sulfosulfuron was detected in harvest samples of wheat straw, grain and field soil.

Keywords: Half-life, pyroxsulam, residue, sulfosulfuron, wheat

Wheat (*Triticum aestivum* L.) is an important cereal crop being used for human food and livestock feed. With a total production of around 93.382 Million Tonns from an area of about 30 million hectares in 2012 (FAOSTAT 2012), India is one of the principal wheat producing (about 14.14% share in total world Wheat Production) and consuming countries in the world. Among the food crops, wheat is one of the most abundant sources of carbohydrates and protein for human being and animals, and its increased production is essential for food security (Devi *et al.*, 2011). Weed infestation is the basic and major component of low yield in wheat production. To combat weed problem, different herbicides are being used. Pyroxsulam (N-(5,7-dimethoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide) and Sulfosulfuron (N-[(4,6-dimethoxy-2-pyrimidinyl)amino]-carbonyl]-2-(ethylsulfonyl)imidazo[1,2-a]pyridine-3-sulfonamide) are triazolopyrimidine sulfonamide and pyrimidinylsulfonylurea herbicide respectively, that provides broad spectrum post emergence annual grass and broadleaf weed control in cereals by inhibiting the action of acetolactate synthetase (ALS) enzyme.

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Pyroxsulam is a new sulfonamide herbicide that controls key annual grasses including *Alopecurus* spp., *Aperaspica venti*, *Lolium* spp., and others (Singh *et al.*, 2010). It also controls of key broadleaves in cereals including *Amaranthus* spp., *Brassica* spp., *Galeopsis tetrahit*, *Myosotis arvensis* and *Viola tricolor* (Pyroxsulam technical bulletin, 2007). Pyroxsulam is providing growers with a high performance, low dose herbicide with a desirable environmental profile (Jabusch *et al.*, 2008). It inhibits the plant enzyme acetolactatesynthase (ALS), which is essential for the synthesis of branched-chain amino acids valine, leucine, and isoleucine (Gonzalez *et al.*, 2008). Inhibition of amino acid production subsequently inhibits cell division and causes death in susceptible plants.

Sulfosulfuron, a sulfonylurea group of herbicide, is used for selective control of complex weed flora in wheat. Sulfosulfuron provides very good control of *Phalaris minor*, *Avena ludoviciana* and partial control of broad leaved weeds (Mishra *et al.*, 2005; Walia *et al.*, 2010; Chhokar *et al.*, 2011 and Singh *et al.*, 2013). It can be applied pre-emergence or early post-emergence and efficacy can be improved by the addition of adjuvants (Olson *et al.*, 2000). It controls many troublesome grass weeds, including downy brome, Japanese brome, cheat, and ripgut brome

(Blackshaw *et al.*, 1998 and Kelly *et al.*, 2003). Sulfosulfuron can also be used for control of volunteer barley (*Hordeum vulgare* L.) in winter wheat. This combination of pyroxsulam and sulfosulfuron can be effective for broad spectrum post emergence annual grass as well as broadleaf weed control with good crop safety. Due to their high potency, they are typically applied at low rates. This results in lower residue levels in environmental samples as compared to other pesticides.

From survey of literature it reveals that, the analytical method of residues of pyroxsulam has not been reported to date, although numerous of analytical methods for triazolopyrimidine herbicides including pyroxsulam residues have been published (Hernandez *et al.*, 2005; Rodriguez *et al.*, 2007 and Liu *et al.*, 2011). Sondhia *et al.* (2008) was developed a method for the residue analysis of sulfosulfuron in wheat by HPLC using Photo diode array detector. In order to strike a better balance between benefits of pesticides in agriculture and risks to human being, environment and food we viewed it as essential to investigate the residual fate and persistence behaviour of a mixed herbicide formulation composed of pyroxsulam 4.5% OD + sulfosulfuron 75% WDG applied to wheat field.

MATERIALS AND METHODS

A supervised field study was conducted in experimental farm of Bidhan Chandra Krishi Viswavidyalaya (B.C.K.V), Mohanpur, West Bengal, in two consecutive years 2012-2013 and 2013-2014 on wheat field (variety: PBW 343). The mixed herbicide formulation of pyroxsulam 4.5% oil dispersion and sulfosulfuron 75% water dispersible granules was applied with the help of Knapsack sprayer @ 18 g a.i. ha⁻¹ (T₁) and @ 36 g a.i. ha⁻¹ (T₂) in Randomized Block Designed (RBD) plots along with untreated control (T₃). The area of each plot was 20 m² (12 rows/plot). Three replications were used for each treatment.

For study of persistence behaviour, samples of wheat plant and field soil were collected randomly from 5-7 places of each plot at 0 (2 hrs after spraying), 1, 3, 7, 10, 15 and 30 days after application of the mixture formulation of herbicide in both the years. At harvest, wheat straw (0.5 kg), grain (0.5 kg), soil (1.0 kg) samples were also collected. Soil samples were collected from a depth of 0-15 cm, air dried and grains were grinded to facilitate extraction of the pesticides using organic solvent. A valid representative (50 g) of plant (50 g each for green foliage, grain and straw) and

soil samples were prepared separately and taken for quantification of herbicides residue analysis separately. The plant samples were homogenized using Polytron homogenizer (Model: Polytron, PT-MR-3100 Kinemetica AG, Lucerne, Switzerland) and extracted immediately after collection as far as practicable.

Extraction and d-spe cleaned up procedure of pyroxsulam residue

In order to quantify pyroxsulam residue, the representative samples (5g of wheat plant, 2g of straw, 5g of grain and 10g of soil) were taken separately in 50 ml fluorinated ethylene propylene (FEP) centrifuge tube (Nalgene, Rochester, NY) separately and 10 ml water was added to it and acidified with 0.1 ml acetic acid. Then acetonitrile (10 ml) was added and subjected to vortex for 1 min. After adding 5g of activated MgSO₄ and 1g sodium chloride, the sample was again vortex for 2 min. Then the sample was centrifuged using high-speed, refrigerated centrifuge, Model Avanti J-301 (Beckman Coulter, Fullerton, CA) for 5 min at 10,000 rpm and 4 ml supernatant extract was collected in 10 ml centrifuge tube. For cleaned up using dispersive solid phase extraction (d-SPE) approaches, 50 mg Primary Secondary Amine (PSA; Varian, Harbor City, CA; 40 mm particle size), 30 mg Florisil (60–100 mesh; Acros, Geel, Belgium) and 50 mg graphitized carbon black (GCB; United Chemical Technology, Bellefonte, PA: only for plant matrices) were added to it and vortex for 1 min. The extract was again centrifuged for 5 min at 8000 rpm and 2 ml supernatant cleaned extract was taken from it and evaporated to dryness in Nitrogen-Evaporator at 30°C. The residue was then reconstituted in 1 ml of mobile phase (1% acetic acid in Methanol and 5 mM ammonium acetate). The sample was then filtered through 0.2 µm membrane filter (0.2µm ultipor N66 nylon 6,6 membrane filter, Pall Corporation) and analyzed by liquid chromatography-tandem mass spectrometry (LC-MS/MS) to quantify pyroxsulam content present on wheat plant, straw, grain and soil.

Extraction and d-spe cleaned up procedure of sulfosulfuron residue

To quantify Sulfosulfuron residue, the representative samples (5g of wheat plant, 2g of straw, 5g of grain and 10g of soil) were taken separately in a 50 ml FEP centrifuge tube and 10 ml of water was added to it. Then 10 ml of ethyl acetate was added to it and subjected to vortex for 1 min. After adding 5g of activated MgSO₄, the sample was again vortex for 2 min. The sample was then centrifuged for 5 min at

10,000 rpm and 4 ml supernatant extract was transferred to a 10 ml centrifuge tube. For cleaned up using d-SPE methodology, 50 mg Primary Secondary Amine (PSA), 30 mg Florisil and 50 mg graphitized carbon black (GCB: only for plant matrices) were added to it and vortex for 1 min. The extract was again centrifuged for 5 min at 8000 rpm and 2 ml supernatant cleaned extract was taken and evaporated to dryness in Nitrogen-Evaporator at 30°C. The residue was then reconstituted in 1 ml of mobile phase (1% acetic acid in Methanol and 5 mm ammonium acetate). The sample was then filtered through 0.2 µm membrane filter (0.2µm ultipor N66 nylon 6, 6 membrane filter , Pall Corporation) and analyzed by liquid chromatography-tandem mass spectrometry (LC-MS/MS) to quantify Sulfosulfuron content present on wheat plant, straw, grain and soil.

Liquid chromatography–tandem mass spectrometry (lc–ms/ms) analysis

The residue analysis of both herbicides was performed by liquid chromatography-tandem mass spectrometry. The HPLC separation was performed on a Alliance 2695 separation module liquid chromatograph (Waters, Milford, MA, USA) equipped with a quaternary solvent delivery system by 20µl via autosampler on a reversed phase Symmetry C18 (5µm; 2.1 × 100 mm) column (Waters, USA) at the flow rate of 0.3 ml min⁻¹. The mobile phase was composed of (A) 0.1% acetic acid containing methanol and 5 mm ammonium acetate as a buffer and (B) 0.1 % acetic acid containing Milli-Q water and 5 mm ammonium acetate. The mobile phase gradients of HPLC for pyroxsulam and Sulfosulfuron are as follows: 100 % solvent A was used initially; after 0.90 min, its concentration was changed from 0 to 100% solvent B in 3 min, gradient changed to 100 % A at 8 min. Finally, the programs switched to initial condition in 10 min and kept for at least 2 min before starting a new analysis. The LC-MS or MS run time was 10 min per sample. The injection volume was 20 µl and the column temperature was 20°C ± 0.8°C.

A Micromass(Manchester,U.K.) QuattroMicro triple-quadruple spectrometer equipped with an electrospray source was used for detection and quantification. The optimized MS instrument parameters of pyroxsulam and sulfosulfuron obtained by the tuning were as follows: capillary voltage 3.0 kV and 1.0kV; cone voltage, 35 V and 15 V; extractor, 3 V and 2 V; source temperature, 120°C; desolvation temperature, 350 °C; desolvation gas flow, 650 l hr⁻¹ nitrogen; cone gas flow, 25 l hr⁻¹; argon collision gas

(argon) pressure to 3.5 e-3 psi for MS/MS respectively. The collision energy for each monitored transition was optimized in MRM mode. The transition monitored for pyroxsulam were m/z 434.85 > 123.89 at 47 eV, 434.85 > 165.81 at 33 eV and 434.85 > 195.04 at 27 eV. The MRM mode of the degradation patterns m/z 434.85 > 195.04 was used for quantification and 434.85 > 123.89 and 434.85 > 165.81 were used for confirmation respectively. The transition monitored for Sulfosulfuron were m/z 470.80 > 211.06 at 13 eV, 470.80 > 218.06 at 31 eV and 470.80 > 261.00 at 15 eV. The MRM mode of the degradation patterns m/z 470.80 > 218.06 was used for quantification and 470.80 > 211.06 and 470.80 > 261.00 were used for confirmation respectively. In the MRM transitions the dwell and inter scan times were 0.15 and 0.1 second, respectively. Pyroxsulam and sulfosulfuron were detected using electrospray ionization in the positive ion mode.

A linearity check study was carried out with the help of matrix match calibration standard (Fig. 1, 3), to know the interference of substrate. In this study a calibration curves were prepared by taking the area corresponding to different concentrations of matrix match calibration standard of pyroxsulam and sulfosulfuron (Fig. 2, 4). Recovery studies were carried out in order to establish the validity of the analytical method and to know the efficiency of extraction and clean up steps employed for the present study, by fortifying the field soil and wheat plant samples with different levels of the analytical standard solution.

RESULTS AND DISCUSSION

Linearity, detection limits and recovery

Good linearity was observed in the studied range with $R^2 \geq 0.99$, limit of detection (LOD) and limit of quantification (LOQ) considered when signal to noise ratio of 3:1 and 10:1, respectively. LOD and LOQ were determined as 0.005 and 0.02 mg kg⁻¹ for pyroxsulam and sulfosulfuron respectively. To determine the reliability of the above analytical methods, recovery studies were carried out by fortifying all the samples control at 0.25, 0.05 and 0.02 mg/kg. The average recovery percentage of pyroxsulam and sulfosulfuron was 87.67% and 85.07% for wheat plant, 88.87% and 87.40 % for wheat straw, 85.67% and 90.80% for wheat grain and 86.27% and 86.00% for field soil respectively. As the recovery percentage is satisfactory for all the substrates, these methods can be adopted for residue and persistence study of pyroxsulam and

sulfosulfuron in wheat plant and soil samples. Three replicates for each concentration were analyzed.

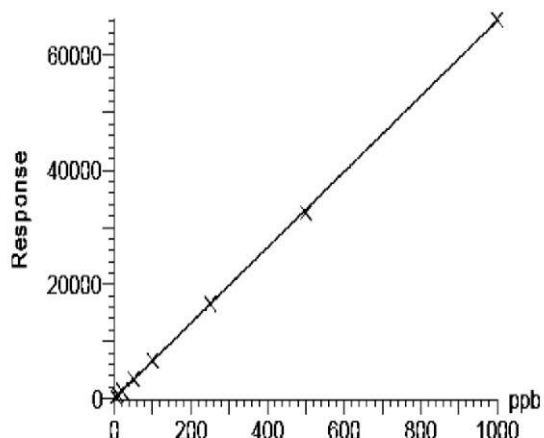


Fig 1: Calibration curve of Pyroxsulam $R^2 = 0.999$

Pyroxsulam

pyro-std-8

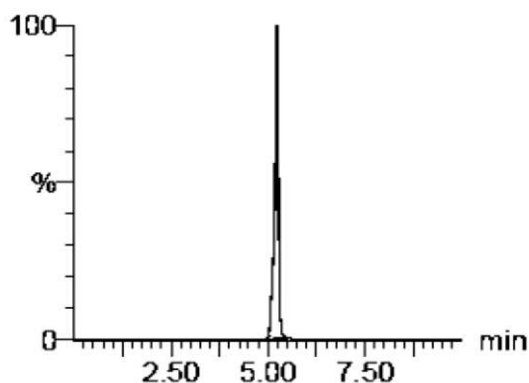


Fig 2: Analytical std. of Pyroxsulam (1mg kg^{-1})

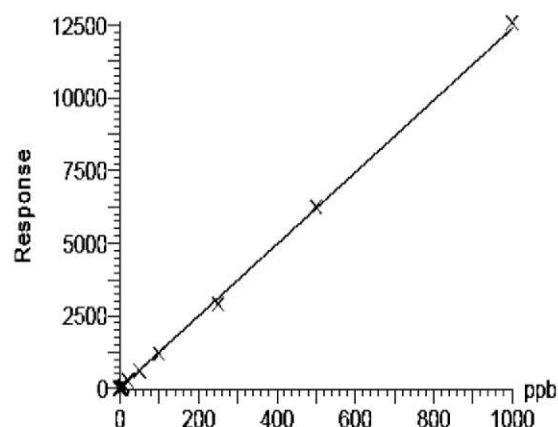


Fig 3: Calibration curve of Sulfosulfuron $R^2=0.997$

Sulfosulfuron

Sulfo-Std-8

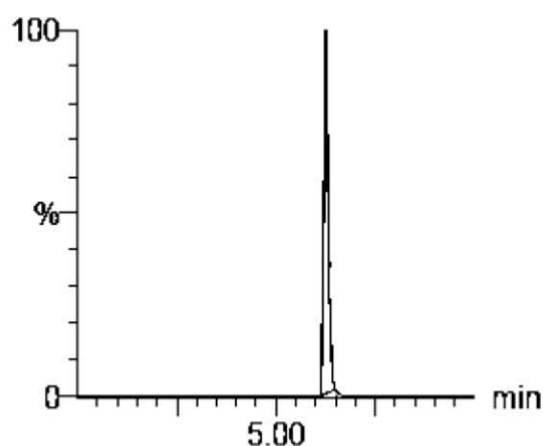


Fig 4: Analytical std. of Sulfosulfuron (1mg kg^{-1})

Table 1: Residue of pyroxsulam in wheat plant and soil sample at different time intervals after application of mixed formulations containing pyroxsulam 4.5% OD and sulfosulfuron 75% WDG @ 18 and 36 g a.i. ha⁻¹ respectively (year-I).

Day after Application	Pyroxsulam 4.5% OD +Sulfosulfuron 75 % WDG @ 18 g a.i. ha ⁻¹				Pyroxsulam 4.5% OD +Sulfosulfuron 75 % WDG @ 36 g a.i. ha ⁻¹			
	Wheat plant		Soil		Wheat plant		Soil	
	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)
0	0.099 (6.52)	-	0.127 (8.66)	-	0.155 (8.38)	-	0.173 (4.65)	-
1	0.058 (9.60)	41.41	0.073 (11.04)	42.52	0.091 (5.51)	41.29	0.107 (4.69)	38.15
3	0.024 (6.28)	75.76	0.042 (15.62)	66.93	0.044 (12.65)	71.26	0.062 (8.06)	64.16
7	BDL	-	BDL	-	BDL	-	0.025 (10.20)	85.55
10	BDL	-	BDL	-	BDL	-	BDL	-
Regression equation	y = -0.199x + 1.980		y = -0.154x + 2.065		y = -0.180x + 2.172		y = -0.119x + 2.211	
Half-life (T _{1/2})	1.51 Days		1.95 Days		1.67 Days		2.53 Days	

BDL : below determination limit (d'' LOQ: 0.02 mg kg⁻¹) * Average RSD : Relative Standard Deviation

Table 2: Residue of sulfosulfuron in wheat plant and soil sample at different time intervals after application of mixed formulations containing pyroxsulam 4.5% OD and sulfosulfuron 75% WDG @ 18 and 36 g a.i.ha⁻¹ respectively (year-I).

Day after application	Pyroxsulam 4.5% OD +Sulfosulfuron 75 % WDG @ 18 g a.i.ha ⁻¹				Pyroxsulam 4.5% OD +Sulfosulfuron 75 % WDG @ 36 g a.i.ha ⁻¹			
	Wheat plant		Soil		Wheat plant		Soil	
	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)
0	0.118 (5.50)	-	0.249 (5.03)	-	0.187(5.37)	-	0.399 (4.30)	-
1	0.078 (10.40)	33.90	0.176 (5.39)	29.32	0.129 (4.26)	31.02	0.353 (4.96)	11.53
3	0.042 (7.30)	64.41	0.119 (4.30)	52.21	0.086 (5.94)	54.01	0.211 (5.95)	47.12
7	BDL	-	0.045 (4.66)	81.93	0.034 (10.60)	81.82	0.115 (7.42)	71.18
10	BDL	-	BDL	-	BDL	-	0.048 (9.46)	87.97
Regression equation	y = -0.148x + 2.059		y = -0.104x + 2.378		y = -0.102x + 2.244		y = -0.089x + 2.619	
Half-life (T _{1/2})	2.03 Days		2.89 Days		2.95 Days		3.38 Days	

BDL : below determination limit (d'' LOQ: 0.02 mg kg⁻¹) * Average RSD : Relative Standard Deviation

Table 3: Residue of pyroxsulam in wheat plant and soil sample at different time intervals after application of mixed formulations containing pyroxsulam 4.5% OD and sulfosulfuron 75% WDG @ 18 and 36 g a.i.ha⁻¹ respectively (year-II).

Day after application	Pyroxsulam 4.5% OD +Sulfosulfuron 75 % WDG @ 18 g a.i.ha ⁻¹				Pyroxsulam 4.5% OD +Sulfosulfuron 75 % WDG @ 36 g a.i.ha ⁻¹			
	Wheat plant		Soil		Wheat plant		Soil	
	*Mean mg/kg(RSD)	Dissipation (%)	*Mean mg/kg(RSD)	Dissipation (%)	*Mean mg/kg(RSD)	Dissipation (%)	*Mean mg/kg(RSD)	Dissipation (%)
0	0.082 (4.30)	-	0.108 (7.17)	-	0.117 (5.34)	-	0.143 (5.97)	-
1	0.047 (10.64)	42.68	0.057 (6.68)	47.22	0.067 (7.09)	42.74	0.114 (8.45)	20.28
3	0.021 (4.76)	74.39	0.032 (9.65)	70.37	0.032 (7.95)	72.65	0.058 (7.48)	59.44
7	BDL	-	BDL	-	BDL	-	0.026 (11.54)	81.82
10	BDL	-	BDL	-	BDL	-	BDL	-
Regression equation	y = -0.193x + 1.891		y = -0.126x + 1.878		y = -0.185x + 2.044		y = -0.106x + 2.140	
Half-life (T _{1/2})	1.56 Days		2.39 Days		1.63 Days		2.84 Days	

BDL : below determination limit (d'' LOQ: 0.02 mg kg⁻¹) * Average RSD : Relative Standard Deviation

The results of the persistence behavior of a mixed herbicide formulation containing pyroxsulam and sulfosulfuron in wheat plant and field soil have been summarized in tables-1, 2 (year-I) and table-3, 4 (year-II). At the harvest time (140 days after application), residue of pyroxsulam and sulfosulfuron was below the detectable limit (d'' LOQ: 0.02 ppm) in all samples (wheat straw, grain and field soil).

It was found that the residues of pyroxsulam and sulfosulfuron gradually decreased with time following 1st order kinetics. The initial deposits (2 h after spraying) of pyroxsulam in field soil were found 0.127 mg kg⁻¹ (T₁) and 0.173 mg kg⁻¹ (T₂) in the 1st year and 0.108 mg kg⁻¹ (T₁) and 0.143 mg kg⁻¹ (T₂) in the 2nd year,

respectively. More than 59-72 % of the initial deposit was degraded within 3 days irrespective of dose and year of experiment. The initial deposits (2 h after spraying) of pyroxsulam in wheat plant were 0.099 mg kg⁻¹ (T₁) and 0.155 mg kg⁻¹ (T₂) in the 1st year and 0.082 mg kg⁻¹ (T₁) and 0.177 mg kg⁻¹ (T₂) in the 2nd year, respectively. More than 71 % of the initial deposit was degraded within 3 days. In case of Sulfosulfuron, the initial deposits in field soil 0.249 mg kg⁻¹ (T₁) and 0.399 mg kg⁻¹ (T₂) in the 1st year and 0.285 mg kg⁻¹ (T₁) and 0.461 mg kg⁻¹ (T₂) in the 2nd year and in wheat plant it was 0.118 mg kg⁻¹ (T₁) and 0.187 mg kg⁻¹ (T₂) in the 1st year and 0.108 mg kg⁻¹ (T₁) and 0.176 mg kg⁻¹ (T₂) in the 2nd year, respectively. More than 47% in field soil

and 58 % in wheat plant of the initial residues were degraded within 3 days irrespective of dose and year. The persistence behaviour of sulfosulfuron of the present study is in well agreement with the earlier studies conducted in wheat under Indian agro-climatic

(Maheswari *et al.* 2007 & Brar *et al.* 2006) and laboratory (Brar *et al.* 2006, Brar *et al.* 2007 and Arora *et al.*, 2009).

The persistence of any chemical is generally expressed in terms of half-life ($T_{1/2}$) or DT_{50} i.e. time for

Table 4: Residue of sulfosulfuron in wheat plant and soil sample at different time intervals after application of mixed formulations containing pyroxsulam 4.5% OD and sulfosulfuron 75% WDG @ 18 and 36 g a.i.ha⁻¹ respectively (year-II).

Day after application	Pyroxsulam 4.5% OD +Sulfosulfuron 75 % WDG @ 18 g a.i.ha ⁻¹				Pyroxsulam 4.5% OD +Sulfosulfuron 75 % WDG @ 36 g a.i.ha ⁻¹			
	Wheat plant		Soil		Wheat plant		Soil	
	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)	*Mean mgkg ⁻¹ (RSD)	Dissipation (%)
0	0.108(8.83)	-	0.285 (4.39)	-	0.176 (7.45)	-	0.461 (4.46)	-
1	0.069 (9.50)	36.11	0.203 (4.30)	28.77	0.123 (5.75)	30.11	0.338 (4.28)	26.68
3	0.033 (14.18)	69.44	0.149 (4.72)	47.72	0.073 (7.63)	58.52	0.231 (4.23)	49.89
7	BDL	-	0.056 (8.93)	80.35	0.024 (10.63)	86.36	0.141 (4.72)	69.41
10	BDL	-	BDL	-	BDL	-	0.052 (11.31)	88.72
Regression equation	y = -0.168x + 2.021		y = -0.097x + 2.437		y = -0.123x + 2.232		y = -0.084x + 2.653	
Half-life ($T_{1/2}$)	1.68 Days		3.10 Days		2.45 Days		3.58 Days	

BDL : Below determination limit (d" LOQ: 0.02 mg kg⁻¹) * Average RSD : Relative Standard Deviation

disappearance of pesticide to 50 per cent of its initial concentration. $T_{1/2}$ values are often obtained by fitting first-order kinetics to observed degradation patterns as $C_t = C_0 \times e^{-kt}$ where C_t is chemical concentration (mg kg⁻¹) at time t (h), C_0 is initial concentration (mg kg⁻¹), and k is the first order rate constant (h⁻¹) independent of C_t and C_0 . The $T_{1/2}$ of pyroxsulam and sulfosulfuron were calculated using Hoskins formula (Hoskins, 1961). Following 1st order kinetics, the half-life of pyroxsulam and sulfosulfuron was found in the range of 1.51-1.67 days and 1.68-2.95 on wheat plant and 1.95-2.84 days and 2.89-3.58 on field soil irrespective of dose and year. There is no available MRLs value for pyroxsulam and sulfosulfuron in wheat or other commodity in USA and EU. Hence, pre-harvest intervals (PHI) do not apply for the herbicides. The recommended acceptable daily intake (ADI) pyroxsulam is 1.0 mg kg⁻¹ body weight per day based on its no observed adverse effect level (NOAEL). Based on these observations, it may be concluded that residual pyroxsulam and sulfosulfuron concentrations at the time of harvest was found below detection limit (d" limit of quantification) when applied at the recommended dose and quite safe from health point of view of the consumer. This work would be helpful to establish MRLs of pyroxsulam and sulfosulfuron in wheat to provide guidance on the safe and proper use of these herbicides.

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