

Dissipation of Sulfosulfuron in Soil and Wheat Plant under Predominant Cropping Conditions and in a Simulated Model Ecosystem

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Environmental fate and dissipation of the sulfonylurea herbicide sulfosulfuron was investigated in soil (inceptisol) and wheat plant under predominant cropping conditions. Studies were conducted in natural field conditions and in a simulated model ecosystem. Thirty days after the wheat seeds had been sown, sulfosulfuron [*N*-[[[4,6-dimethoxy-2-pyrimidinyl]amino]carbonyl]-2-(ethylsulfonyl)imidazo[1,2-*a*]pyridine-3-sulfonamide] 75% w/w WG formulation was applied once in the field. The dosages were 25 and 50 g of active ingredient (ai)/ha. Studies were conducted in significantly separated individual plots to avoid contamination. In a predetermined interval, soil samples were collected and analyzed for the residues of sulfosulfuron. At harvest, wheat grain, straw, and soil samples were analyzed for the residues. Similar experiments were conducted in a model ecosystem. Apart from this, after harvest, the succeeding crops coriander (*Coriandrum sativum*) and edible amaranth (*Amaranthus mangostanus* L.) were raised in the model ecosystem and studied for the residues. No residues were detected in wheat grain, straw, and soil samples collected at harvest from both experiments or in the succeeding crops coriander and edible amaranth in the model ecosystem when tested at the minimum detection level of 0.001 μg/g. The dissipation of sulfosulfuron was found to have first-order kinetics in soil and plant in both studies. The dissipation data of sulfosulfuron in the model ecosystem were compared with those from the natural field conditions.

KEYWORDS: Sulfosulfuron; dissipation in soil and plant

INTRODUCTION

Since 1982, 20 sulfonylurea herbicides have been commercialized for use under a wide variety of agronomic conditions in numerous crops (1). These new herbicides collectively span a wide range of soil residual properties that are assigned to meet specific agricultural needs. The discovery of the sulfonylurea herbicide is one of the most dramatic breakthroughs in herbicide research. The herbicidal properties of sulfonylurea analogues were first reported in 1966 for the compounds prepared as derivatives of propazine (2). Excessive mobility and persistence of herbicides in soils may cause groundwater contamination and phytotoxic effects for sensitive crops grown in the following season. Transport and degradation processes are dependent on the properties of the herbicide and edaphic conditions. The properties of the herbicide usually cannot be altered, but the modification of soil properties may lead both to optimizing herbicide behavior and increasing its mobility and persistence.

The fate of a pesticide from its point of application on soil is governed by various interactive processes such as adsorption, transformation, and transportation in addition to the influence of factors such as herbicide application rate, crop type,

agricultural practices, and climatic conditions (3–5). Attempts to simulate field conditions in the laboratory can lead to artifacts and the erroneous quantification of dissipation behavior. In general, the dissipation behavior of a herbicide differs under different environmental conditions. There are many simulation models used to predict and evaluate the environmental fate of herbicides (6, 7). These models in general are used to describe a very well-defined environmental condition. Thus, a model ecosystem is designed to assess the data on distribution and degradation of herbicides near the field scenario.

Sulfosulfuron, a sulfonylurea herbicide chemically known as *N*-[[[4,6-dimethoxy-2-pyrimidinyl]amino]carbonyl]-2-(ethylsulfonyl)imidazo[1,2-*a*]pyridine-3-sulfonamide, is a postemergence herbicide extensively used for the control of grasses and broad-leaved weeds in cereals (wheat). Not much work has been done to study the environmental behavior of the compound (8–19).

The objectives of the present investigations were (1) to investigate the dissipation behavior of sulfosulfuron, a postemergence herbicide under predominant cropping conditions in two different environments (one is under natural field conditions and the other is in a model ecosystem); (2) to study the influence of herbicide on succeeding crop products; and (3) to study the possible accumulation of residues in succeeding crops (vegetables).

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MATERIALS AND METHODS

Field Trial Details. *Natural Field Conditions.* Field experiments were conducted at Ootacamund, Tamil Nadu, India. The field was prepared by irrigation with water to a level of 5 cm above the surface and ploughed once with a mould board plough. The field was leveled using a leveling board. Nine different trial plots each with a dimension of 5 × 4 m (20 m²) were prepared. A 1 m distance was maintained between the plots. All four sides of the plots were protected by soil boundaries (bunds) raised to a level of 30 cm height and 30 cm width. Wheat seeds were sown as per the local agricultural practice with a spacing of 15 cm × 22 cm. Thirty days after sowing, two different dosages were sprayed in the field: one the recommended dose [25 g of active ingredient (ai)/ha of sulfosulfuron 75% w/w WG formulation, T_1] and the other double the recommended dose (50 g of ai/ha of sulfosulfuron 75% w/w WG formulation, T_2). Three different plots (triplicates) were sprayed for each dose. A further three different triplicate plots were sprayed with water (T_0) without any herbicide and maintained as control.

Model Ecosystem. The model ecosystem (20) also contains nine plots as in the case of the field study. The plots were constructed with bricks and cement. The internal dimensions were 2.0 m × 1.0 m × 0.30 m. The distance between adjacent plots was 0.5 m. All of the plots in the model ecosystem were protected with a clear plastic roof on the top at a height of 10 ft to prevent the effect of rain. The soil used in the field was inceptisol with the following properties: sand, 43.4%; silt, 13.2%; and clay, 46%; pH 8.1, CEC = 27.1 mequiv/100 g; and organic carbon content = 0.61%. The same soil was used in the model ecosystem. Wheat seeds of Malavika variety (local) were sown in the field. Thirty days after the wheat seeds had been sown, sulfosulfuron was sprayed in the model ecosystem as in the case of field study. To avoid any deviations with respect to the pH of the water, all of the experiments were conducted using water collected from a single source. The ecosystem was managed similarly to field studies.

Sampling under Natural Field Conditions. Soil samples were collected on different occasions: 0 day (3 h after application) and 1, 3, 5, 7, 10, 15, 20, 25, 30, and 40 days after the herbicide application. About 1 kg of soil sample was collected randomly from each plot using a soil auger up to a depth of 15 cm from the surface. After pebbles and other unwanted materials had been removed, the soil sample was mixed thoroughly and 250 g was subsampled for the analysis of sulfosulfuron. Plant samples were also collected on different occasions: 0, 5, 10, 15, 20, and 25 days after herbicide application. At harvest from each plot was subsampled 200 g of wheat grain, straw, and soil for residue analysis.

Sampling in the Model Ecosystem. Soil samples were collected at 0, 1, 3, 5, 7, 10, 15, 20, 25, 30, and 40 days after herbicide application. Plant samples were collected at 0, 5, 10, 15, 20, and 25 days after herbicide spraying. At harvest from each plot was subsampled 200 g of wheat grain, straw, and soil for residue analysis. After the wheat crop had been harvested, coriander (*Coriandrum sativum*) and edible amaranth (*Amaranthus mangostanus* L.) were planted in the same field and the vegetable leaves analyzed 7 weeks later.

Method of Sampling. *Soil.* A composite surface soil sample was drawn from the experimental field for initial analysis. At each sampling occasion, soil samples were collected randomly from nine different spots of each plot. Likewise, soil samples were collected from all of the triplicate plots at two different doses. Samples from the control plots were collected before samples were collected from the herbicide-treated plots. The sample size was ~1 kg from each plot. Pebbles and stones were removed, the soil was mixed thoroughly, and 250 g was subsampled and analyzed.

Wheat Plants. Ten grams of representative plant leaf samples were collected from each plot. The plant samples were cut into small pieces and used for analysis.

Leafy Vegetables. To assess the residual effect of sulfosulfuron, if any, on the following crop, the products of the succeeding crops coriander and edible amaranth were collected and analyzed. The above-ground portions of leaf samples (10 g) were collected from all nine plots.

Sample Storage. All of the samples were stored at -10 °C, processed, and analyzed within 7 days.

Wheat Grain, Straw, and Soil. Sixty-five days after herbicide spraying, the entire produce from each plot was harvested, wheat grain and straw were separated, and 200 g was subsampled for the analysis of residues of sulfosulfuron. At this time point soil samples were also collected and analyzed.

Analytical Methodology: Sulfosulfuron. *Soil.* A 10 g soil sample was weighed into an Erlenmeyer flask and extracted with 100 mL of a distilled water/acetonitrile mixture (1:1 v/v) using an end-over-end mechanical shaker for 30 min. The sample was filtered into a 500 mL round-bottom flask and rinsed with 50 mL of the same solvent; the volume was reduced to 50 mL in a rotary evaporator at 40 °C.

(a) *Partition.* The sample was transferred to a clean 250 mL separatory funnel and extracted with two 100 mL portions of methylene chloride each time. The combined extract was collected and concentrated to near dryness in a vacuum rotary evaporator at 40 °C.

(b) *Cleanup.* A glass column was packed with 2 g of Florisil and washed with two 5 mL portions of 2% methanol in methylene chloride followed by two 5 mL portions of isooctane. After the column had been conditioned, the sample was redissolved in 2 mL of methylene chloride and diluted with 8 mL of 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 eluent was evaporated to dryness by rotary evaporator. The sample was redissolved in 3 mL of 10% acetonitrile in water, filtered, and analyzed by HPLC.

Wheat Plant and Vegetables. Wheat plant and vegetable samples were mixed thoroughly and cut into small pieces using a mechanical slicer and homogenized using a high-speed blender for 2 min. Ten grams of the homogenized sample was extracted with 100 mL of a distilled water/acetonitrile mixture (1:1) for 30 min and processed as described earlier.

Instrumentation. Sulfosulfuron was separated and quantified by using a Shimadzu high-performance liquid chromatograph consisting of an LC-10 ATvp pump and an SPD-10 AVvp UV-vis detector. A CBM-101 communication module supported by CLASS-LC10 software was used. A Phenomenex C₁₈ column, 25 cm length × 4.6 mm i.d., with a mobile phase of acetonitrile/water (70:30) at a flow rate of 1.50 mL/min was used. The λ_{max} was set at 220 nm for detection purposes. The approximate retention time for sulfosulfuron is 5.3 min. At the limit of detection, 0.001 $\mu\text{g/g}$, the signal-to-noise ratio of 3:1 is maintained. A sulfosulfuron reference analytical standard of 99% purity supplied by Monsanto Enterprises Ltd., Mumbai, India, was used in the study. All other chemicals used in the study were analytical grade reagents.

Data Analysis. Rate of Degradation. Rates of degradation (21) for sulfosulfuron in soil and wheat plant were calculated and found follow first-order kinetics. The degradation rate constant was calculated by linear regression from the transformed first-order rate equation, $\ln C_t = \ln C_0 - Kt$, where C_t is the sulfosulfuron concentration as a function of time in days (t), C_0 is the highest sulfosulfuron concentration, and K is the degradation rate constant. The time of dissipation of 50% (DT_{50}) of the highest concentration was calculated from the equation $DT_{50} = 0.693/K$.

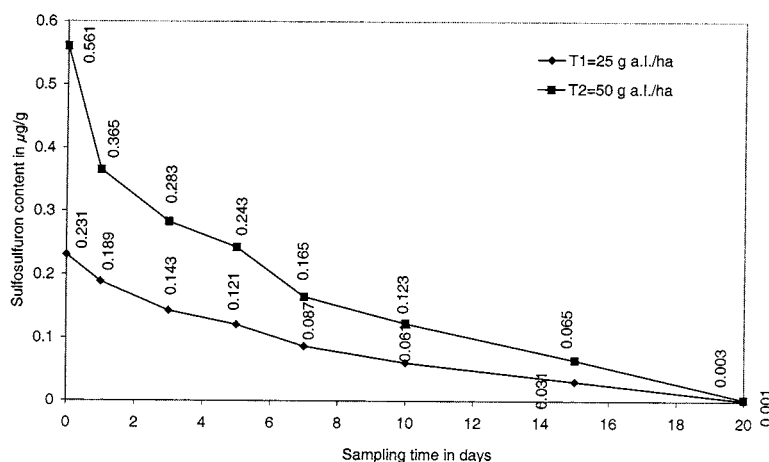
RESULTS AND DISCUSSION

Recovery and Detection Limits. Different known concentrations of sulfosulfuron (1.0, 0.2, 0.1, 0.02, 0.01, and 0.001 $\mu\text{g/g}$) were prepared in acetonitrile by diluting the stock solution. Twenty microliters of standard solution was injected and the peak area measured. Validation of the method was performed in terms of recovery studies before analysis of unknown sample. The recovery study was conducted for different substrates. Ten grams of control soil sample was taken. Using a pipet, 1 mL of a standard solution of sulfosulfuron of known concentration was added uniformly on the surface of the matrix and mixed well before extraction. The extraction was performed as described under Materials and Methods. Recovery study was conducted

Table 1. Recovery and Limits of Detection of Sulfosulfuron from Various Samples^a

	wheat grain	wheat plant	coriander	edible amaranth	soil	water
recovery (%)	77 ± 1.5	76 ± 2.6	72 ± 4.6	75 ± 6.4	78 ± 6.4	76 ± 1.0
RSD (%)	1.6–3.4	2.0–2.8	4.1–4.7	6.0–7.1	6.1–6.5	1.0–1.5

^a Fortification level, 1.0–0.001 $\mu\text{g/g}$; limit of quantitation, 0.001 $\mu\text{g/g}$; limit of detection, 0.001 $\mu\text{g/g}$; no. of analyses, six replications.

**Figure 1.** Dissipation of sulfosulfuron in soil under natural field conditions.**Table 2.** DT₅₀ of Sulfosulfuron under Natural Field Conditions and in a Model Ecosystem

dosage	DT ₅₀ (days)				
	natural field conditions		model ecosystem		
	soil	plant	soil	plant	water
T ₁ = 25 g of ai/ha	3.00	2.94	3.50	3.92	3.15
T ₂ = 50 g of ai/ha	3.17	3.07	3.72	4.02	3.28

for wheat plant, water, and the subsequent crops coriander and edible amaranth.

The recovery and detection limits of sulfosulfuron are shown in **Table 1**. The recovery ranges from 72 to 78% in different substrates, the limit of quantification is 0.001 $\mu\text{g/g}$, and the signal-to-noise ratio is 3:1. No substrate interferences were observed at this quantification level as evidenced by the control sample analysis.

Dissipation of Sulfosulfuron in Soil and Wheat Plant under Natural Field Conditions. *Soil.* Under the natural field conditions the initial concentrations of sulfosulfuron were 0.231 and 0.561 $\mu\text{g/g}$ for treatments 25 and 50 g of ai/ha treatments, respectively. By the first day the concentrations had dissipated to 0.189 $\mu\text{g/g}$ (T₁) and 0.365 $\mu\text{g/g}$ (T₂), and by seventh day the sulfosulfuron contents were 0.087 $\mu\text{g/g}$ (T₁) and 0.165 $\mu\text{g/g}$ (T₂), respectively. There was a steady decrease in the content of sulfosulfuron, so that by the 20th day the concentration levels were down to 0.001 $\mu\text{g/g}$ (T₁) and 0.003 $\mu\text{g/g}$ (T₂). Thereafter the sulfosulfuron content dissipated to below the detectable level (**Figure 1**). The DT₅₀ values calculated were 3.00 and 3.17 days (**Table 2**).

Wheat Plant. The initial concentrations of sulfosulfuron were found to be 0.120 and 0.279 $\mu\text{g/g}$ for treatments at 25 and 50 g of ai/ha, respectively. By the fifth day the sulfosulfuron contents had decreased to 0.110 $\mu\text{g/g}$ (T₁) and 0.195 $\mu\text{g/g}$ (T₂). There was a gradual decrease in the sulfosulfuron content, and by the 20th day sulfosulfuron had dissipated to 0.001 and 0.003 $\mu\text{g/g}$; thereafter, the content fell to below the detectable level (**Figure 2**). The calculated DT₅₀ values were 2.94 and 3.07 days (**Table 2**).

Table 3. Dissipation Data of Sulfosulfuron in Soil under Natural Field Conditions and in a Model Ecosystem

sampling time (days)	natural field conditions		model ecosystem	
	T ₁ ^a	T ₂ ^b	T ₁	T ₂
0	0.231	0.561	0.371	0.621
1	0.189	0.365	0.222	0.512
3	0.143	0.283	0.123	0.392
5	0.121	0.243	0.091	0.211
7	0.087	0.165	0.062	0.181
10	0.061	0.123	0.041	0.121
15	0.031	0.065	0.021	0.062
20	0.001	0.003	0.012	0.031
25	ND ^c	ND	0.001	0.003

^a T₁ = 25 g of ai/ha of sulfosulfuron 75% w/w WG formulation. ^b T₂ = 50 g of ai/ha of sulfosulfuron 75% w/w WG formulation. ^c Not detectable.

Dissipation of Sulfosulfuron in Wheat Model Ecosystem. Under the model ecosystem the initial sulfosulfuron concentrations in soil were 0.371 and 0.621 $\mu\text{g/g}$ for treatments at 25 and 50 g of ai/ha, respectively. During the later occasions there was a gradual decrease, and by the 15th day the sulfosulfuron had dissipated to 0.021 $\mu\text{g/g}$ (T₁) and 0.062 $\mu\text{g/g}$ (T₂). Concentrations were at the minimum level by the 25th day, and thereafter the residues fell to below the detectable level (**Figure 3**). The calculated DT₅₀ values were 3.50 and 3.72 days (**Table 2**).

In wheat plant the initial concentrations of sulfosulfuron calculated were 0.181 and 0.361 $\mu\text{g/g}$ for treatments at 25 and 50 g of ai/ha, respectively. By the fifth day the sulfosulfuron content had dissipated to 0.072 $\mu\text{g/g}$ (T₁) and 0.141 $\mu\text{g/g}$ (T₂). There was gradual decrease in the sulfosulfuron content, and by the 25th day sulfosulfuron had dissipated to 0.001 and 0.003 $\mu\text{g/g}$; thereafter, the content fell to below the detectable level (**Figure 4**). The calculated DT₅₀ values were 3.92 and 4.02 days (**Table 2**). The dissipation data are presented in **Tables 3** and **4**.

Residues of Sulfosulfuron in Wheat and Subsequently Harvested Vegetables. Wheat grain, straw, and soil samples collected at harvest from both of the studies showed no

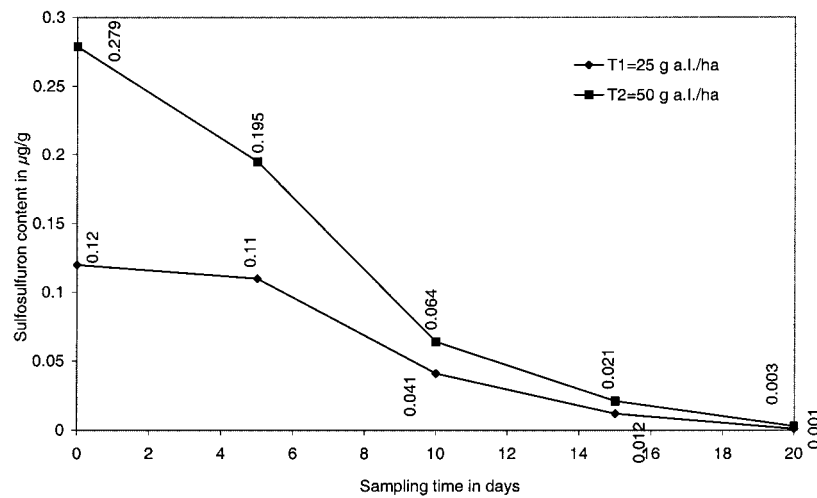


Figure 2. Dissipation of sulfosulfuron under natural field conditions in wheat plant.

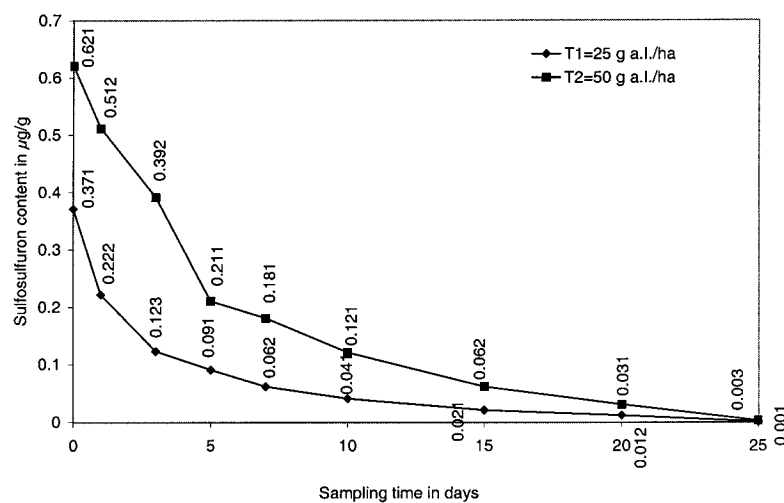


Figure 3. Dissipation of sulfosulfuron in a model ecosystem in soil.

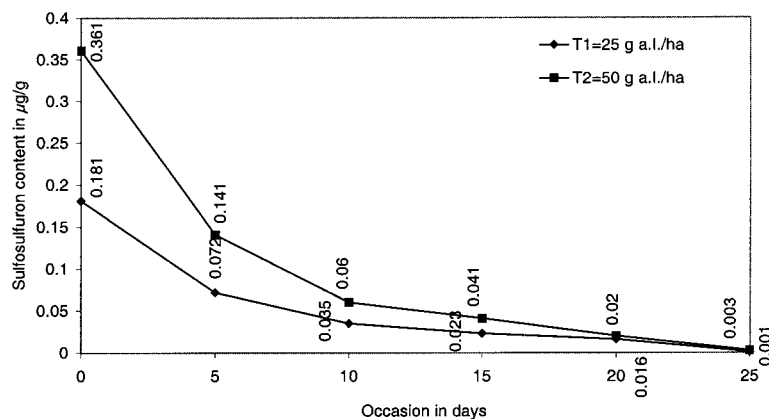


Figure 4. Dissipation of sulfosulfuron in a model ecosystem in wheat plant.

detectable residues of sulfosulfuron when analyzed at the detection limit of $0.001 \mu\text{g/g}$. An attempt was made to understand the uptake of sulfosulfuron residue from soil, if any, by vegetables planted subsequently. After the wheat crop was harvested, coriander and edible amaranth were planted in the same plots. Vegetable samples were analyzed for residues 7 weeks later. In none of the edible plant leaf samples were detectable concentrations of sulfosulfuron observed, indicating that sulfosulfuron in soil was not taken up by vegetables grown under the experimental conditions described in this study.

Influence of Environmental Properties. In the present experiment the influences of soil properties and the pH of the water on the degradation pattern are likely to be identical because the soil and water used for both of the experiments are similar and from the same source. During the experimental period occasional rainfall (2–6 mm) was observed in the natural field condition, and the maximum day temperatures ranged from 22 to 33 °C, whereas the inside maximum atmospheric temperature of the ecosystem was in the range of 19–30 °C. The dissipation data obtained in the study clearly show that

Table 4. Dissipation Data of Sulfosulfuron in Wheat Plant under Natural Field Conditions and in a Model Ecosystem

sampling time (days)	natural field conditions		model ecosystem	
	T_1^a	T_2^b	T_1	T_2
0	0.120	0.279	0.181	0.361
5	0.110	0.195	0.072	0.141
10	0.041	0.064	0.035	0.060
15	0.012	0.021	0.023	0.041
20	0.001	0.003	0.016	0.020
25	ND ^c	ND	0.001	0.003

^a $T_1 = 25$ g of ai/ha of sulfosulfuron 75% w/w WG formulation. ^b $T_2 = 50$ g of ai/ha of sulfosulfuron 75% w/w WG formulation. ^c Not detectable.

sulfosulfuron is rapidly degradable, and the rates of dissipation are similar in both the field and model ecosystem and follow first-order kinetics. The reason may be due to the similar properties of the pH of water, the soil used, and the similar temperature conditions. In neither the field nor the modern ecosystem were detectable levels of sulfosulfuron found 65 days after herbicide application in wheat grain, straw, and soil. Even though it has been reported that sulfosulfuron affects sensitive crops (22), the observation in the present study shows no uptake when succeeding crops of coriander and edible amaranth were analyzed. The data generated in the present studies suggest that the model ecosystem is highly useful and can be considered as an alternative to field study in a defined environment, and the data thus generated on the environmental fate of herbicides can be extrapolated to different field conditions.

Further major advantages of conducting the studies in a model ecosystem are that it requires limited space, is easily maintained, is environmentally safe, and costs very little when compared with field studies. The integrity of the crop can be maintained during the complete duration of the trial. Contamination of the soil can be minimized and, if required, remedy can be taken very easily.

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