

## Sulfosulfuron Persistence in Soil Under Different Cultivation Systems of Wheat (*Triticum aestivum*)



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Marziyeh YOUSEFI<sup>1</sup>, Behnam KAMKAR<sup>1,\*</sup>, Javid GHEREKHLOO<sup>1</sup> and Rohollah FAEZ<sup>2</sup>

<sup>1</sup>Department of Agronomy, Gorgan University of Agricultural Sciences and Natural Resources (GUASNR), Gorgan 49189-43464 (Iran)

<sup>2</sup>Cotton Research Institute of Iran, Gorgan 4916685915 (Iran)

(Received March 27, 2015; revised June 27, 2016)

### ABSTRACT

Many sulfonylurea herbicides have been used under a wide variety of agronomic conditions in numerous crops. An understanding of dissipation rate of herbicide is fundamental for predicting the fate of herbicide in soil. In order to study the sulfosulfuron persistence under different cultivation systems of wheat, a four replicated experiment was carried out in the Hashemabad Research Center of Gorgan, Iran in 2010 in a split plot design with two factors. Cultivation system as the main factor consisted of six levels, including conservation tillage by Combine, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk. Secondary factor included two levels of sulfosulfuron application (with and without sulfosulfuron). Soil samples were taken at 6 stages and soil microbial respiration and soil pH were measured as factors affecting sulfosulfuron persistence. Results showed the least time of sulfosulfuron persistence belonged to the cultivation system of no-tillage by Baldan grain drill with a half-life of 4.62 d. Then, conservation tillage by Combine and conventional tillage with a half-life of 6.30 d and conservation tillage by Delta Model with a half-life of 9.90 d were ordered. The most time of sulfosulfuron persistence (11.55 d) was related to conservation tillage by Chizelpacker. Ninety percent reduction of sulfosulfuron concentration occurred 15.34, 20.92, 32.88, and 36.38 d after sulfosulfuron application, respectively, for no-tillage system, conservation tillage by Combine and conventional tillage, conservation tillage by Delta Model and surface tillage, and conservation tillage by Chizelpacker. In all the cultivation systems, toxicity symptoms were not observed 40 d after spraying sulfosulfuron onto the tomato plants which were used as test plant. Effects of different cultivation systems on soil microbial respiration were also significant.

**Key Words:** bioassay, conservation tillage, conventional tillage, half-life, soil microbial respiration, surface tillage

**Citation:** Yousefi M, Kamkar B, Gherekhloo J, Faez R. 2016. Sulfosulfuron persistence in soil under different cultivation systems of wheat (*Triticum aestivum*). *Pedosphere*. 26(5): 666–675.

Weeds have been a problem for human ever since plants are domesticated and therefore weed management seems to be as old as agriculture itself (Singh and Kulshrestha, 2007). The conventional method of weed control (hoeing or hand weeding) is laborious, expensive, and insufficient. Moreover, weeding during critical growth stages is not possible due to increased cost of human labor and its scarce availability, thus necessitating the use of herbicides (Sondhia, 2010). Soil acts as a major sink for the bulk of the pesticides used in agriculture. Pesticides reach the soil through various routes like direct application to soil, spray drift, dislodging and run off. In soil, pesticide residues are subjected to various transformations and transportation processes (Srivastava *et al.*, 2006). Herbicide residue estimation in soil and edible plant parts is very essential to determine the duration of herbicide activity in soil and its effect on the crops and to analyze the quality of the food and feed (Sondhia, 2010).

Since 1982, more than 20 sulfonylurea herbicides have been commercialized for use under a wide variety of agronomic conditions in numerous crops (Ramesh and Mahesvari, 2003). Sulfonylureas, a novel group of highly selective herbicides, are used at low application rates for weed control in cereal crops. They have become very popular as replacements for old high-application-rate herbicides. Moreover, they exhibit low toxicity to mammals. However, due to their moderate to high mobility and increasing use, they are being detected in natural waters. Due to their high phytotoxicity, they can present environmental risks, especially for crops such as soybean and rice (Wu *et al.*, 2010), aquatic plants and microorganisms, and indirectly affect the whole trophic food web of aqueous biota, such as ponds (Perreau *et al.*, 2007). Sulfonylurea herbicides are weak acids and they exist primarily in the anionic form in agronomic soils. Consequently, sulfonylurea herbicides are generally weakly adsorbed by soil. Also,

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\*Corresponding author. E-mail: kamkar@gau.ac.ir.

their adsorption decreases when soil pH increases, as a result of the increased amount of anionic species in solution (Sondhia, 2008). Sulfonylureas are highly sensitive to chemical and biotic hydrolysis in water and soil, and have half-lives ranging from 1–8 weeks in water (Perreau *et al.*, 2007).

Sulfosulfuron [1-(4,6-dimethoxypyrimidin-2-yl)-3-(2-ethylsulfonylimidazo [1,2- $\alpha$ ]pyridin-3-yl)] is a sulfonylurea herbicide that is characterized as acetolactate synthase inhibitors. Recently this herbicide has been registered for control of broad-leaves and grass weeds in cereal crops in Iran. Its recommended application rate is 19.95 g active ingredient (a.i.) ha<sup>-1</sup> and it is usually applied with adjuvants to improve its efficacy (Hadizadeh, 2008). Maheswari and Ramesh (2006) expressed sulfosulfuron had a half-life of 3.97 d in Alfisol at 25 g a.i. ha<sup>-1</sup> and 4.54 d at 50 g a.i. ha<sup>-1</sup> whereas in Inceptisol the half-lives were 4.68 and 5.52 d, respectively. Saha *et al.* (2003) reported the half-life of sulfosulfuron in soil was 5.3 d. Singh and Kulshrestha (2007) determined sulfosulfuron residues in soil under wheat and explained that sulfosulfuron dissipated with a half-life of 5.4–6.3 d. Sulfosulfuron possesses high solubility in water, which implies a high potential for movement in soil (Sondhia and Singhai, 2008).

The persistence of an herbicide in soil is influenced by several factors such as sunlight photochemical degradation, microbial degradation, adsorption-desorption, physical/chemical bonding, ion exchange, rain/water flow, *etc* (Atmakuru *et al.*, 2007). Chemical hydrolysis and microbial breakdown are the most important pathways of sulfonylurea degradation in soil, whereas photolysis and volatilization are relatively minor processes (Sondhia, 2009). Soil pH, temperature, moisture and organic matter are the major factors that influence sulfonylurea chemical hydrolysis and microbial degradation. With increasing pH, sulfosulfuron becomes more soluble and hence able to leach from the relatively microbe-rich topsoil to deeper soil, where microbial breakdown is less likely to occur (Sondhia and Singhai, 2008). The effective performance at very low application levels can be phytotoxic to susceptible species, causing injury to certain rotational crops such as sugar beet, lentils, peas, mustard, sunflower, and other non-target plants.

A quantitative estimation of small amounts of residues, which can not be measured by instrumental methods, is of sufficient practical use for predictions of succeeding crops or potential side-effects. In this sense, the use of bioassays can complement the instrumental methods since they can detect residues less than 1  $\mu$ g a.i. ha<sup>-1</sup> and provide information regarding herbi-

cide bioavailability for plant and its possible phytotoxicity (Santin-Montanya, 2006). Studies related to the fate of pesticides in the environment are required for predicting the potentiality of groundwater contamination and their behaviour in the aquatic environment. Hydrolytic degradation and its kinetics in the normal pH and temperature ranges are of utmost importance to predict the persistence of a particular pesticide in the ecosystem. Sulfonylurea herbicides were hydrolyzed more quickly in acidic than in alkaline conditions (Saha and Kulshrestha, 2002). Wu *et al.* (2010) expressed that a high-performance liquid chromatography (HPLC) method with ultraviolet (UV) detection was feasible for the multi-residue determination of sulfonylurea herbicides in soil. There is very limited information available in literature on the fate of sulfosulfuron herbicide in the agro-ecosystem as well as on the analytical method for the determination of its residues from environmental samples (Saha *et al.*, 2003). This study was aimed to determine sulfosulfuron persistence under different cultivation systems of wheat by HPLC method.

## MATERIALS AND METHODS

### *Field experiment*

A field experiment was carried out in the Hashemabad Research Center of Gorgan, Iran in 2010 with spilt-plot design in four replications and two factors. Cultivation system as the main factor consisted of six levels, including conservation tillage by Combine, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk. Combine just moves the soil in a 5-cm depth without soil turning upside down. Baldan grain drill does not move and cultivate the soil as a no tillage equipment, while Chizelpacker and Delta Model are used as compound cultivators which mix the soil without turning it upside down and aerate the soil well. Conservation tillage has many advantages compared to conventional tillage: increasing water infiltration into the soil, increasing organic matter, cycling of nutrients in the soil, reducing soil erosion, improving soil biological fertility, saving fuel and time and reducing labour. Recently, the number of growers adopting conservation tillage is increasing in Golestan Province of Iran because of its benefits. Agriculture Ministry of Iran is going to extend these systems, especially in the areas faced with serious soil erosion. Golestan Province is one of higher risk regions in this respect and many machines are now available, es-

pecially for pioneer farmers. Secondary factor included two levels of sulfosulfuron application (with and without sulfosulfuron). Sulfosulfuron was applied at field recommended dose of 20 g a.i. ha<sup>-1</sup>. Wheat (N<sub>8019</sub> cultivar) was direct drilled in January 2010. The soil is a silty clay loam, including 38% clay, 44% silt, and 18% sand. Sulfosulfuron treatment was applied on March 30, 2010 at the end of tillering stage. Soil samples were collected from 0–15 cm depth at 5 h and 7, 15, 30, 65, and 80 d after herbicide application. Samples were taken from 4 points in each plot (25 m × 12 m) and then the combined sample was analyzed.

#### Chemical analysis

Soil samples were air-dried for 24 h, sieved through a 2-mm sieve and then collected in glass vials. Afterward, soil samples were stored at -26 °C until further analysis. To determine the amount of sulfosulfuron herbicide in the soil, preparation of standard solutions, extraction, separation and cleaning steps were performed according to Srivastava *et al.* (2006).

Ten milligram of sulfosulfuron (97% purity) was dissolved in acetonitrile (HPLC grade) in a volumetric flask (10 mL) to give a 1 000 µg mL<sup>-1</sup> stock solution A. One milliliter of stock solution A was transferred and diluted up to the mark in a 50-mL volumetric flask with acetonitrile to give a 20 µg mL<sup>-1</sup> of stock solution B. The stock solution B was diluted appropriately with acetonitrile to obtain standard solutions containing 0.01, 0.05, 0.1, 0.5, 1, 5, and 10 µg mL<sup>-1</sup> sulfosulfuron, respectively. Twenty milliliters of each solution were injected using an injector in HPLC (Merck-Hitachi, Japan-Germany). Each run was performed 3 times. Calibration curve was drawn according to the standard concentrations (Fig. 1).

Soil sample (50 g) from each plot was taken in 250-

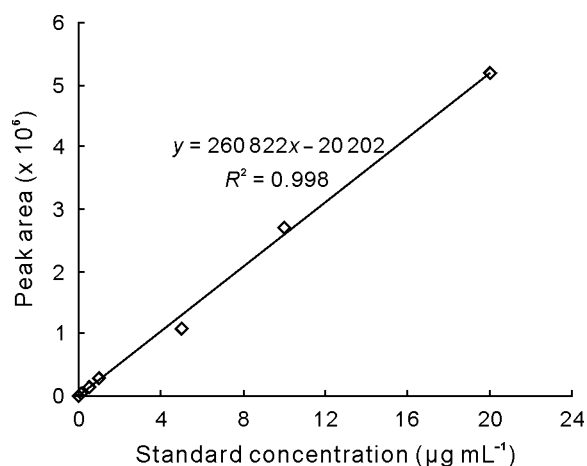


Fig. 1 Calibration curve of standard concentrations for sulfosulfuron herbicide.

mL Erlenmeyer flask and 50 mL mixture of acetonitrile and ammonium carbonate (9:1, volume:volume) was added. Then the Erlenmeyer flask was shaken for 30 min and the upper organic layer was separated and filtered through Buchner funnel and Whatman No. 1 filter paper. The soil residue was once again shaken after addition of 50 mL mixture of acetonitrile and ammonium carbonate (9:1, volume:volume) and the upper organic phase was separated and combined with the first fraction. The filtrate was concentrated on a rotary evaporator at 40 °C to reduce its volume to 20 mL. Then the extract was dissolved in 50 mL of 1 mol L<sup>-1</sup> NaCl. The solution was transferred to one separating funnel and partitioned with 50 mL of dichloromethane (HPLC grade) twice to separate organic phase from the aqueous. The combined dichloromethane extract was collected and passed through Na<sub>2</sub>SO<sub>4</sub> to remove traces of moisture. Filtrate was collected, pooled and dried at 40 °C on a rotary evaporator to near dryness. Finally, the residue was dissolved in 2 mL of HPLC grade acetonitrile and filtered through 0.45-µm filter (Millipore<sup>TM</sup>) before injection into the HPLC system for the determination of sulfosulfuron concentration.

The HPLC system was equipped with EZchrom software, photodiode array detector and RP-C18 column (Hitachi, Japan; 250 mm × 4 mm). Mobile phase consisted of a mixture of acetonitrile, water, and orthophosphoric acid (80:20:0.1, volume:volume:volume) at a flow rate of 1 mm min<sup>-1</sup> at 25 °C with 20 µL injection volume. The UV absorbances of sulfosulfuron in acetonitrile were recorded at different wavelengths starting from 212 nm on photodiode array detector in HPLC. Sulfosulfuron showed three suitable wavelengths, *i.e.*, 220, 232, and 234 nm. The peak at 234 nm showed sharp area of sulfosulfuron and was used as maximum absorbance wavelength for all further analysis. In this condition, sulfosulfuron retention time was 2.7 min. Analytical grade sulfosulfuron (97% purity) was supplied by Dr. Ernestofer Co., Germany. Acetonitrile and dichloromethane were HPLC grade and all chemicals were from Merck Co., USA.

The instrument detection limit (IDL, µg mL<sup>-1</sup>) for sulfosulfuron was estimated by 10 repetitive injections of a standard solution containing 1.0 µg mL<sup>-1</sup> of sulfosulfuron (Singh and Kulshrestha, 2007).

$$IDL = (SD \cdot St \cdot C) / A \quad (1)$$

where SD is the standard deviation; St is the Student's coefficient (2.262); C is the concentration of sulfosulfuron (µg mL<sup>-1</sup>); and A is the mean area of sulfosulfuron at that concentration. In this study, the IDL

for sulfosulfuron was calculated as  $0.27 \mu\text{g mL}^{-1}$ .

#### Recovery experiment

Recovery experiment of sulfosulfuron from soil was conducted to determine the efficacy of the analytical procedure undertaken during the experiment. Sulfosulfuron of 12.5 or 25.0  $\mu\text{g}$  (2.5 or 5 mL of  $5 \mu\text{g mL}^{-1}$  standard concentration) was added to 50 g of soil without herbicide in triplicate and were extracted by above-mentioned method. Then, it was injected into the HPLC system. The average recovery was 73.1%.

Estimated method detection limit (EMDL,  $\mu\text{g g}^{-1}$ ) of sulfosulfuron was estimated from the IDL as follows (Singh and Kulshrestha, 2007):

$$\text{EMDL} = (\text{IDL} \cdot 100 \cdot V) / (M \cdot \text{REC}) \quad (2)$$

where  $M$  is the mass of soil (g);  $V$  is the volume made for analysis (mL); and REC is the average recovery of sulfosulfuron by the described method (%). In the present study, EMDL was calculated as  $0.015 \mu\text{g g}^{-1}$ .

Degradation rate of sulfosulfuron in soil followed first-order kinetics. The degradation rate constant ( $K$ ) was calculated by linear regression from the transformed first-order rate equation,  $\ln C_s = \ln C_0 - Kt$ , where  $C_s$  is the sulfosulfuron concentration as a function of time ( $t$ , d); and  $C_0$  is the highest sulfosulfuron concentration (Maheswari and Ramesh, 2006; Singh and Kulshrestha, 2007).

The time of dissipation of 50% ( $\text{DT}_{50}$ ) and 90% ( $\text{DT}_{90}$ ) of the highest sulfosulfuron concentration were calculated as follows (Eqs. 3 and 4):

$$\text{DT}_{50} = 0.693/K \quad (3)$$

$$\text{DT}_{90} = 2.302/K \quad (4)$$

#### Soil microbial respiration

Soil microbial respiration was determined according to Stotzky (1965). In brief, 50 g of soil sample from each plot was placed in erlen, where a beaker was also placed and 10 mL NaOH solution ( $0.5 \text{ mol L}^{-1}$ ) was added in the beaker. Each erlen was covered with a piece of parafilm to avoid  $\text{CO}_2$  absorption from the atmosphere and then incubated at  $25\text{--}28^\circ\text{C}$  for 5–7 d. After the incubation, 1 mL of  $\text{BaCl}_2$  was added to the NaOH solution to precipitate carbonate as barium carbonate ( $\text{BaCO}_3$ ) and the remaining NaOH was titrated with HCl using phenolphthalein as an indicator. Total  $\text{CO}_2$  was calculated following Eq. 5:

$$\text{Total CO}_2 = (V_1 - V_2) \cdot C_{\text{acid}} \cdot E \quad (5)$$

where  $V_1$  is the volume of HCl to titrate the blank;  $V_2$  is the volume of HCl to titrate the soil sample;  $C_{\text{acid}}$  is the molar concentration of HCl; and  $E$  is the equivalent weight of  $\text{CO}_2$ , which is considered as 22.

#### Statistical analysis

Statistical analysis for degradation of sulfosulfuron was done using Sigmaplot 10 and Microsoft Office Excel 2003. Analyses of soil microbial respiration and pH data were performed using SAS software. Mean comparisons were carried out using the least significant difference test at  $P = 0.05$ .

## RESULTS AND DISCUSSION

#### Sulfosulfuron persistence in soil

The degradation rate of sulfosulfuron under different cultivation systems followed first-order kinetics. Results showed that the least time for sulfosulfuron persistence belonged to the cultivation system of no-tillage by Baldan grain drill with a half-life of 4.62 d, which was followed by conservation tillage by Combine and conventional tillage with a half-life of 6.3 d and conservation tillage by Delta Model with a half-life of 9.9 d. The most persistence (11.55 d) was related to conservation tillage by Chizelpacker (Table I).

Ninety percent reduction of sulfosulfuron concentration occurred after 15.34, 20.92, 32.88, and 36.38 d of sulfosulfuron application, respectively, for no-tillage system, conservation tillage by Combine and conventional tillage, conservation tillage by Delta Model and surface tillage, and conservation tillage by Chizelpacker. The soil samples collected from field had the initial sulfosulfuron concentrations in 5 h after herbicide application as 0.22, 0.15, 0.10, 0.13, 0.15, and  $0.14 \mu\text{g mL}^{-1}$ , respectively, for the conservation tillage by Combine, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk. The residues dissipated rapidly to 0.11, 0.04, 0.07, 0.07, 0.10, and  $0.05 \mu\text{g mL}^{-1}$  after 7 d of herbicide application. These values on day 15 were  $0.02 \mu\text{g mL}^{-1}$  in all tillage systems and thereafter a steady decrease was observed. After 80 d the residues were  $0.01 \mu\text{g mL}^{-1}$  in conservation tillage by Chizelpacker and  $0.02 \mu\text{g mL}^{-1}$  in the other systems (Fig. 2).

The gradient of dissipation curve indicated that sulfosulfuron decreased quickly in the first stages and thereafter a slow decrease was observed. The dissipation rate of sulfosulfuron was faster in the no-tillage system

TABLE I

Parameters estimated in the first-order kinetics equation ( $C_s = C_0 e^{-Kt}$ )<sup>a)</sup> expressing sulfosulfuron degradation in soil and half-life of sulfosulfuron in soil in 6 cultivation systems: conservation tillage by Combine, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk

Cultivation system	$C_0$ $\mu\text{g mL}^{-1}$	$K$	$R^{2b)}$	$P$ value	Half-life d
Conservation tillage by Combine	$0.22 \pm 0.01^c)$	$0.11 \pm 0.01$	0.93	< 0.000 1	6.3
No-tillage by Baldan grain drill	$0.14 \pm 0.01$	$0.15 \pm 0.03$	0.82	< 0.000 1	4.6
Conservation tillage by Chizelpacker	$0.10 \pm 0.007$	$0.06 \pm 0.01$	0.86	< 0.000 1	11.6
Conservation tillage by Delta Model	$0.13 \pm 0.01$	$0.07 \pm 0.01$	0.85	< 0.000 1	9.9
Surface tillage by heavy disk	$0.15 \pm 0.01$	$0.07 \pm 0.01$	0.82	< 0.000 1	9.9
Conventional tillage	$0.14 \pm 0.01$	$0.11 \pm 0.02$	0.84	< 0.000 1	6.3

<sup>a)</sup>  $C_s$  is the sulfosulfuron concentration as a function of time ( $t$ );  $C_0$  is the highest sulfosulfuron concentration;  $K$  is the degradation rate constant of sulfosulfuron in soil.

<sup>b)</sup> Coefficient of determination.

<sup>c)</sup> Means  $\pm$  standard errors ( $n = 10$ ).

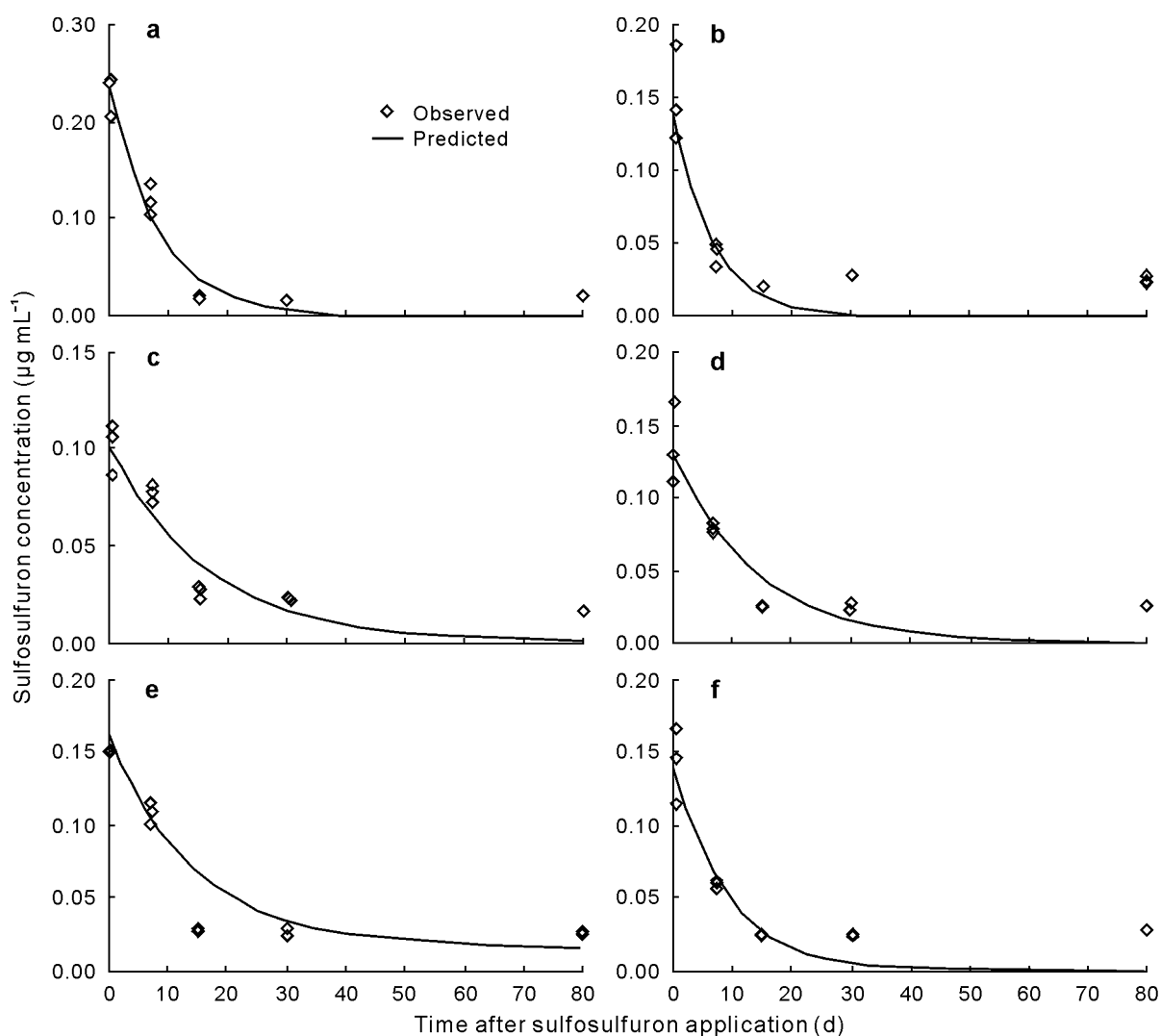


Fig. 2 Dissipation curves of sulfosulfuron in soil at 5 stages (5 h and 7, 15, 30, and 80 d after sulfosulfuron application) in 6 cultivation systems, including conservation tillage by Combine (a), no-tillage by Baldan grain drill (b), conservation tillage by Chizelpacker (c), conservation tillage by Delta Model (d), surface tillage by heavy disk (e), and conventional tillage by moldboard plow and twice disk (f).

than the other systems. The dissipation of sulfosulfuron residues between 5 h and 15 d after herbicide application was more than that at the other stages, which may be due to microbial degradation, chemical hydrolysis, and photodegradation.

#### Soil microbial respiration

Soil microbial respiration is an indicator of microorganisms activity. Increasing soil respiration in the first stages after sulfosulfuron application can result in microbial breakdown of sulfosulfuron and the expedition in herbicide decline. Results showed that the effect of different tillage systems on soil microbial respiration was significant 5 h and 7 d after sulfosulfuron application (Table II).

Soil microbial respiration 5 h after herbicide application was equal to 0.14, 0.19, 0.20, 0.19, 0.17, and 0.13 mg CO<sub>2</sub> g<sup>-1</sup> soil d<sup>-1</sup>, respectively, in conservation tillage by Combine, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk. These values in 7 d after herbicide application were 0.18, 0.26, 0.17, 0.24, 0.18, and 0.22 mg CO<sub>2</sub> g<sup>-1</sup> soil d<sup>-1</sup>. Thereafter a rapid decrease was observed and soil microbial respiration 15 d after herbicide application attained 0.02, 0.03, 0.04, 0.04, 0.05, and 0.03 mg CO<sub>2</sub> g<sup>-1</sup> soil d<sup>-1</sup>, respectively. The most soil microbial respiration occurred 5 h and 7 d after herbicide application (Fig. 3).

The most variation in soil microbial respiration was observed between 5 h and 15 d after herbicide application. This was consistent with the results observed for the dissipation of sulfosulfuron residues at these stages, suggesting induction of soil microbial population and soil microorganisms' use of sulfosulfuron for growth and nutrition in the first stages. The primary

population of certain groups of microorganism starts to decompose herbicides many few days after their applications; however, the secondary population, which produces induced enzymes, starts to decompose herbicides after a period of adaptation and the rate of soil microbial respiration increases after then (Milosevic and Govedarica, 2002). There are three different phases which can occur during herbicide transpiration into soil. In the first phase, a slight decrease in herbicide concentration occurs due to its low absorption in soil. In the second phase, there are little changes in herbicide concentration for a long period, which refers to a period of inactivity and also a period of adaptation of microbial decomposer population. In the third phase, logarithmic sharp decline in the amount of herbicide occurs, which coincides with the rapid growth of adapted microbial population (Fallah *et al.*, 2006).

According to Milosevic and Govedarica (2002), the number of this group of nitrogen-fixing bacteria decreased considerably in the period of 7–14 d after herbicide application. Simultaneously, the number of actinomycetes and fungi increased, indicating that these microorganisms use herbicides as sources of biogenous elements. Fungi are less sensitive to herbicide application in comparison with bacteria (Ghinea *et al.*, 1998). Comparison of soil respiration among cultivation systems showed that the most soil respiration was related to no-tillage that had the least herbicide persistence in soil (4.62 d). This observation was supported by the earlier findings of Aislabie and Llyod-Jones (1995), who reported that no-tillage systems led to the reduction of herbicide persistence in soil.

#### Other affecting factors

Soil pH also plays an important role in the degradation of sulfosulfuron herbicide. Analysis of variance showed that cultivation systems had no significant effect

TABLE II

Analysis of variance on soil microbial respiration at 6 stages (5 h and 7, 15, 30, 65, and 80 d after sulfosulfuron application in 6 cultivation systems, including conservation tillage by Combine, no-tillage, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage, and conventional tillage)

Source of variation	Degree of freedom	F value					
		5 h	7 d	15 d	30 d	65 d	80 d
Block	3	3.81*	9.64**	6.93**	2.96ns	1.67ns	0.40ns
Cultivation	5	3.77**	10.18**	2.86*	2.16ns	0.34ns	1.10ns
Cultivation × block	15	8.94**	8.06**	7.62**	1.05ns	0.83ns	1.76ns
Sulfosulfuron	1	57.18**	131.81**	0.32ns	3.88ns	0.40ns	0.10ns
Cultivation × sulfosulfuron	5	0.46ns <sup>a)</sup>	1.23ns	0.18ns	1.35ns	2.83*	4.16**
Coefficient of variation (%)		25.26	17.25	20.75	26.20	16.56	20.94

\*, \*\*Significant at  $P = 0.05$  and  $P = 0.01$  levels, respectively.

<sup>a)</sup>Not significant.

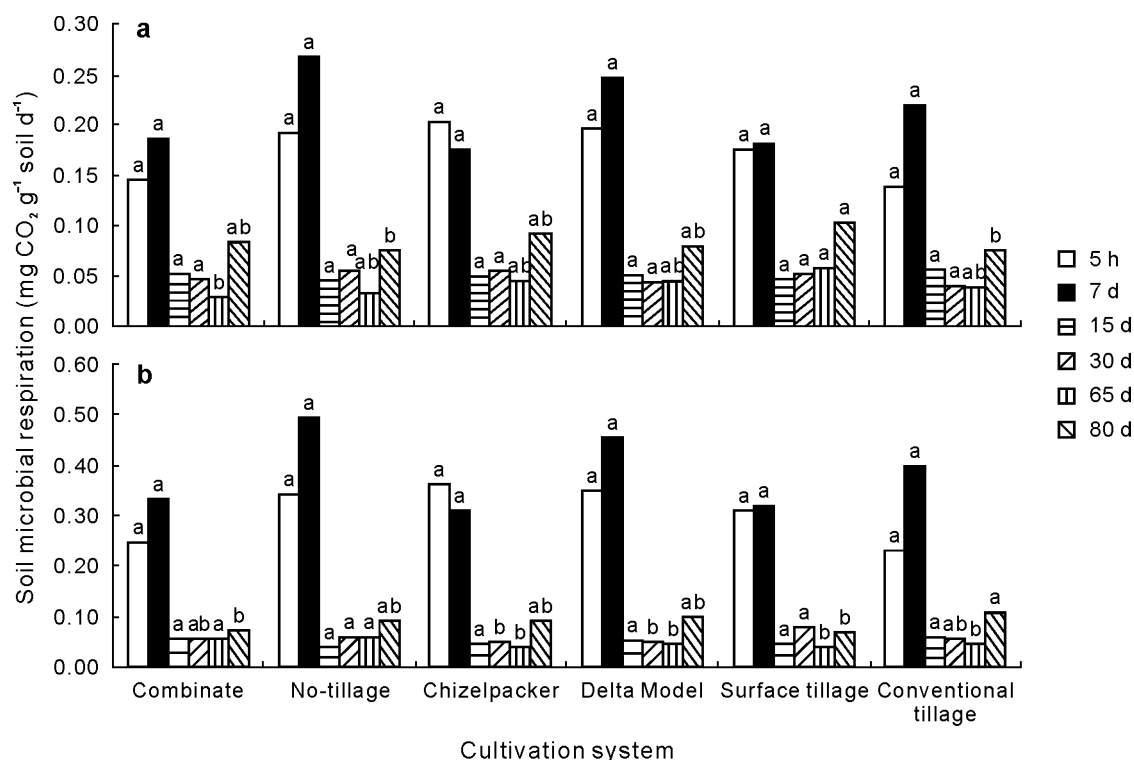


Fig. 3 Soil microbial respiration with herbicide (a) and without herbicide (b) at 6 stages (5 h and 7, 15, 30, 65, and 80 d after sulfo-sulfuron application) in 6 cultivation systems, including conservation tillage by Combineate, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk. Bars with the same letter(s) within a given stage are not significantly different among different cultivation systems at  $P = 0.05$ .

cts on pH in 0–15 cm soil layer 5 h and 15, 65, and 80 d after herbicide application, while the effect of cultivation systems on soil pH was significant 7 and 30 d after herbicide application (Table III).

The highest and lowest pHs 7 d after herbicide application were related to conservation tillage by Chizelpacker and no-tillage, respectively. The highest and lowest pHs 30 d after herbicide application were related to surface tillage and conservation tillage by Chizelpacker, respectively (Fig. 4).

Maheswari and Ramesh (2006) reported that soil pH played a vital role in degradation of herbicides,

because hydrolysis of sulfo-sulfuron in soil was mainly pH-dependent, and that the degradation rate increased with decreasing pH. Soil pH is an effective factor on soil microbial population and activity. Soil bacteria prefer alkaline and neutral pHs, while acidic pH is suitable for fungi and actinomycetes are abundant in alkaline pH. Nonetheless, most soil microorganisms are active at neutral pH (Lakzian *et al.*, 2004). In the present study, soil pH was slightly alkaline, which was suitable for optimum microbial activity, suggesting that the degradation of sulfo-sulfuron was mainly affected by soil microorganisms, but not by chemical hydrolysis. The de-

TABLE III

Analysis of variance on soil pH at 6 stages (5 h and 7, 15, 30, 65, and 80 d after sulfo-sulfuron application) in 6 cultivation systems, including conservation tillage by Combineate, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk

Source of variation	Degree of freedom	Mean of square					
		5 h	7 d	15 d	30 d	65 d	80 d
Block	3	0.001ns <sup>a)</sup>	0.0003ns	0.0009ns	0.0005ns	0.0004ns	0.001ns
Cultivation	5	0.002ns	0.01**	0.001ns	0.002**	0.001ns	0.001ns
Error	15	0.0009	0.0009	0.0006	0.0005	0.001	0.0006
Coefficient of variation (%)		0.38	0.38	0.33	0.31	0.55	0.32

\*\*Significant at  $P = 0.01$  level.

<sup>a)</sup>Not significant.

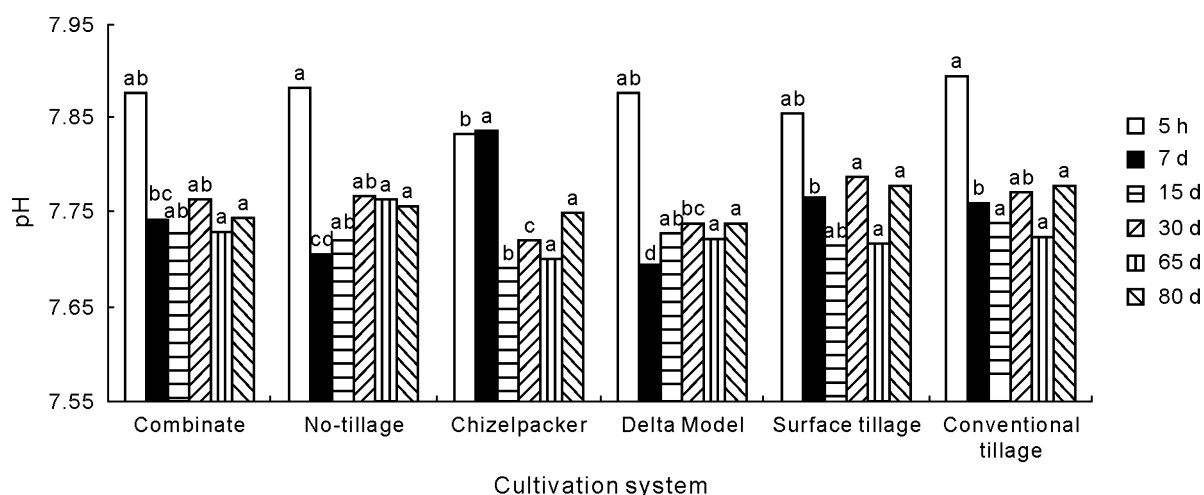


Fig. 4 Soil pH at 6 stages (5 h and 7, 15, 30, 65, and 80 d after sulfosulfuron application) in 6 cultivation systems, including conservation tillage by Combine, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk. Bars with the same letter(s) within a given stage are not significantly different among different cultivation systems at  $P = 0.05$ .

crease in the concentration of pesticide in soil is compensated by the increased microbial activity, thereby increasing the rate of degradation (Sondhia, 2008). Saha and Kulshrestha (2002) studied the effective abiotic factors on degradation of sulfosulfuron and found that sulfosulfuron degraded at a faster rate in acidic ( $\text{pH} = 4.0$ ) than in alkaline condition ( $\text{pH} = 9.2$ ) and the least rate occurred in neutral pH. These results were supported by Sondhia and Singhai (2008) and Sondhia (2009), who reported that the greater soil pH was, the less chemical hydrolysis occurred, and that the dissipation of sulfosulfuron in alkaline soil was mostly through microbial activity. Pusino *et al.* (2003) conducted one study on adsorption and desorption of a sulfonylurea herbicide (triasulfuron) in three soils and found that soil pH was the main factor influencing the adsorption with high level of adsorption measured in the soils with low pH and high organic carbon content. The pH of the experimental soil was slightly alkaline, allowing less adsorption of sulfosulfuron to the soil. Thus, in the present study, weak adsorption of sulfosulfuron on soil and leaching could also mainly cause unavailability of sulfosulfuron in the surface soil.

Environmental conditions such as temperature and moisture are effective factors on the dissipation rate of sulfosulfuron in soil and also on soil microbial activity. In this study, temperature average during the experimental period increased from  $14.5^\circ\text{C}$  5 h after herbicide application to  $29^\circ\text{C}$  80 d after herbicide application (Fig. 5).

Akbari (2009) studied the variation trend of soil microbial carbon biomass under wheat crop and expressed that  $16\text{--}17^\circ\text{C}$  was the best temperature con-

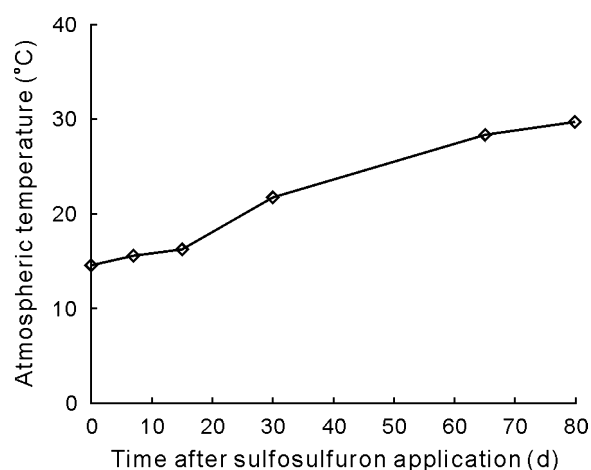


Fig. 5 Atmospheric temperature trend during the experimental period.

dition for soil microbial activity. Akbari (2009) also reported that the most microbial carbon biomass was observed at elongation stage, when temperature average increased from  $7.5$  to  $12^\circ\text{C}$ . In this study, temperature average between 5 h and 15 d after herbicide application was  $15\text{--}16^\circ\text{C}$ , which was optimal for soil microorganism activity, therefore increasing dissipation rate of sulfosulfuron.

Soil moisture contents for the 6 cultivation systems, including conservation tillage by Combine, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk, were  $257.5$ ,  $223.4$ ,  $262.3$ ,  $250.1$ ,  $249.8$ , and  $262.9\text{ g kg}^{-1}$ , respectively, 5 h after sulfosulfuron application and  $70.5$ ,  $61.0$ ,  $54.0$ ,  $54.2$ ,  $64.1$ ,  $68.4$ , and  $61.7\text{ g kg}^{-1}$  80 d after sulfosulfuron app-



lication (Fig. 6). It should be mentioned that occasional rainfall occurred during the experimental period.

In this study, soil moisture between 5 h and 15 d after sulfosulfuron application was the optimum moisture range for soil bacteria and fungi activity, so sulfosulfuron was degraded rapidly. These results were supported by the earlier findings of Akbari (2009), who reported that the best moisture condition for activity of soil microbial population was around 150–220 g kg<sup>-1</sup>. Moreover, soil moisture affects leaching of herbicide. Sondhia and Singhai (2008) found that the solubility of sulfosulfuron was high in water and that sulfosulfuron was able to leach from the topsoil to deeper profile and thus may not be available in the surface soil (0–15 cm).

According to Alonso-Prados *et al.* (2002), soil and climatic conditions could determine the behavior and dissipation of herbicide in soil. They reported that temperatures above or near 30 °C during the summer season could contribute to the degradation of herbicide and thus there was no injury on barley. Also, Sondhia and Singhai (2008) reported high precipitation and high temperature reduced the chances for carryover effect of herbicide, due to high losses through leaching, microbial degradation, hydrolysis, and surface runoff. Therefore, soil pH and climatic conditions during the intervening periods and duration between herbicide application and following crops are important in determining the potential for herbicide carryover.

#### Carryover effect

Carryover effect of sulfosulfuron was surveyed on tomato plants, which were used as index plant. The results of shoot length and dry weight showed that there

was no significant difference between the plants grown in the control and treated soil 40 d after sulfosulfuron application. In all cultivation systems, toxicity symptoms were not observed and sulfosulfuron had no effect on germination of tomato plants.

Analytical methods are very sensitive, but the methods lack extraction techniques. In this sense, the use of bioassays can complete these methods. Bioassays as suitable screening tests could be useful to exclude the occurrence of low levels of residues of phytotoxic compounds in soils, when the phytotoxic concentration for non-target organisms is lower than 0.05 mg a.i. kg<sup>-1</sup> soil (which equals to 75 g a.i. ha<sup>-1</sup>, incorporated in the upper 10 cm soil layer with the bulk density of 1.5 g cm<sup>-3</sup>) (Alonso-Prados *et al.*, 2002). Due to the high level of sulfosulfuron activity, very low level residues can persist in the soil to injure certain crops 1 to 3 years after application, depending upon soil and climate conditions (Senseman and Armbrust, 2007).

#### CONCLUSIONS

An understanding of dissipation rate of herbicide is fundamental for predicting the fate of herbicide in soil. The results of the current study indicated that the dissipation of sulfosulfuron was significantly related with tillage systems, microbial respiration and soil pH. The cultivation method could be an effective factor on herbicide persistence in soil, because cultivation systems affected soil microbial respiration and pH. Sulfosulfuron application induced soil microbial population, resulting in microbial degradation of herbicide and decrease of sulfosulfuron persistence in soil. The most soil respiration was related to no-tillage system that had

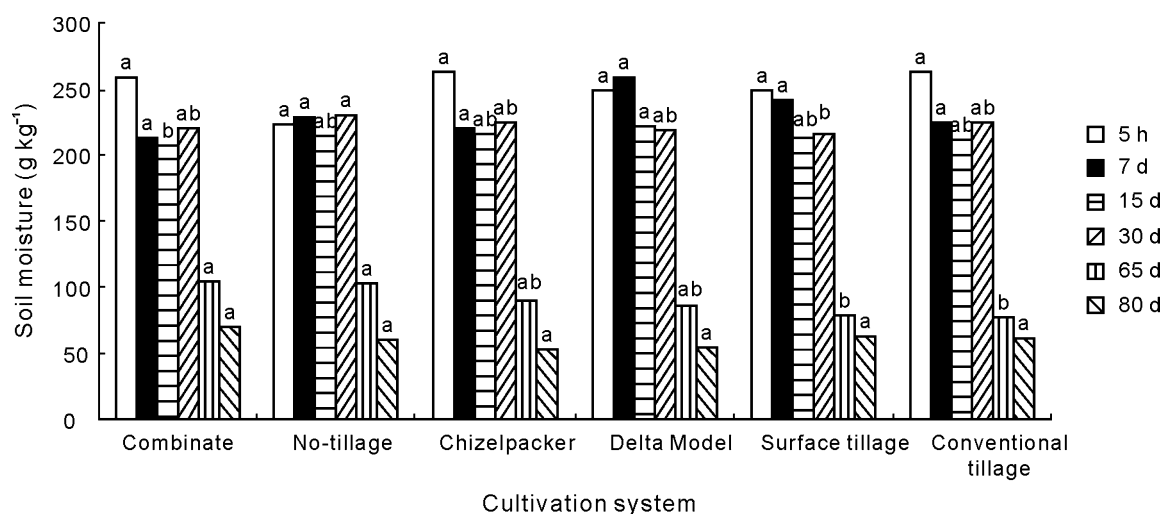


Fig. 6 Soil moisture at 6 stages (5 h and 7, 15, 30, 65, and 80 d after sulfosulfuron application) in 6 cultivation systems, including conservation tillage by Combinate, no-tillage by Baldan grain drill, conservation tillage by Chizelpacker, conservation tillage by Delta Model, surface tillage by heavy disk, and conventional tillage by moldboard plow and twice disk. Bars with the same letter(s) within a given stage are not significantly different among different cultivation systems at  $P = 0.05$ .

the least sulfosulfuron persistence in soil. Half-life of sulfosulfuron in soil in different cultivation systems ranged from 4 to 9 d. Because of interactions among soil physical, chemical and biological properties, climate condition, and management practices, it is not easy to study herbicide persistence in cultivation systems and further continual experiments are required.

Herbicide application is considered as the last option to control the weeds in integrated weed management protocol. On the other hand, food safety is a concern along with food security. Therefore, reducing residual effects of herbicides in agricultural products is one of challenges when it is needful to apply chemical inputs. In the regions classified as high risk zones in respect to weeds infestation, it is necessary to build the cultivation systems that can reduce residual effects of herbicis in order to produce more safe products.

Since climatic conditions can affect the persistence of herbicide, further experiments should be conducted in different geographical regions. Our findings revealed that a great amount of herbicide in soil could be degraded through chemical hydrolysis and soil microbial activity; however, phytotoxic potential of herbicide residual for subsequent crops should not be ignored. Our findings also revealed that soil respiration could be used as an indicator to investigate herbicide persistence in soil. Moreover, it is advised to use test plants, as an indirect and low cost method, for measurement of the amount of herbicide in soil.

#### ACKNOWLEDGEMENT

This study was supported by the research and technology vice presidency, Gorgan University of Agricultural Sciences and Natural Resources, Iran.

#### REFERENCES

- Aislabie J, Lloyd-Jones G. 1995. A review of bacterial-degradation of pesticides. *J. Soil Res.* **33**: 925–942.
- Akbari F. 2010. Investigation of soil microbial biomass C dynamics using different crops residues and its relationship with wheat yield (N8019 variety) (in Persian). M. S. Thesis, Gorgan University of Agricultural Sciences and Natural Resources, Gorgan.
- Alonso-Prados J L, Hernández-Sevillano E, Llanos S, Villarroya M, García-Baudín J M. 2002. Effects of sulfosulfuron soil residues on barley (*Hordeum vulgare*), sunflower (*Helianthus annuus*) and common vetch (*Vicia sativa*). *Crop Prot.* **21**: 1061–1066.
- Atmakuru R, Elumalai T P, Sivanandam S. 2007. Identification of residues of sulfosulfuron and its metabolites in subsoil-dissipation kinetics and factors influencing the stability and degradation of residues from topsoil to subsoil under predominant cropping conditions. *Environ Monit Assess.* **130**: 519–528.
- Fallah A, Besharati H, Khosravi H. 2006. Soil Microbiology (in Persian). Aeej Publication, Tehran.
- Ghinea L, Lancu M, Turcu M, Gheorghe S. 1998. The impact of sulfonyl-urea and non-selective herbicides on biological activity of sandy soils. *Romanian Agric Res.* **9**: 55–60.
- Hadizadeh M H. 2008. Investigation of the effects of organic matter amendments and sulfosulfuron application rates on the herbicide persistence and biological traits of soil in wheat (in Persian). Ph. D Thesis. Ferdowsi University, Mashhad.
- Lakziyan A, Sheibani S, Bahadoriyan M, Shaddel L. 2004. Soil Microbiology (in Persian). Sokhangostar Publication, Mashhad.
- Maheswari S T, Ramesh A. 2007. Adsorption and degradation of sulfosulfuron in soils. *Environ Monit Assess.* **127**: 97–103.
- Milošević N A, Govedarica M M. 2002. Effect of herbicides on microbiological properties of soil. *Proc Acad Nat Sci Philadelphia.* **102**: 5–21.
- Perreau F, Bados P, Kerhoas L, Nelieu S, Einhorn J. 2007. Trace analysis of sulfonylurea herbicides and their metabolites in water using a combination of off-line or on-line solid-phase extraction and liquid chromatography-tandem mass spectrometry. *Anal Bioanal Chem.* **388**: 1265–1273.
- Pusino A, Fiori M G, Braschi I, Gessa C. 2003. Adsorption and desorption of triasulfuron by soil. *J Agric Food Chem.* **51**: 5350–5354.
- Ramesh A, Maheswari A R. 2003. Dissipation of sulfosulfuron in soil and wheat plant under predominant cropping conditions and in a simulated model ecosystem. *J Agric Food Chem.* **51**: 3396–3400.
- Saha S, Kulshrestha G. 2002. Degradation of sulfosulfuron, a sulfonylurea herbicide, as influenced by abiotic factors. *J Agric Food Chem.* **50**: 4572–4575.
- Saha S, Singh S B, Kulshrestha G. 2003. High performance liquid chromatographic method for residue determination of sulfosulfuron. *J Environ Sci Health.* **38**: 337–347.
- Santin-Montanya I, Alonso-Prados J L, Villarroya M, Garcia-Baudin J M. 2006. Bioassay for determining sensitivity to sulfosulfuron on seven plant species. *J Environ Sci Health.* **41**: 781–793.
- Senseman S A, Armbrust K (eds.). 2007. Herbicide Handbook. Weed Science Society of America, Lawrence.
- Singh S B, Kulshrestha G. 2007. Determination of sulfosulfuron residues in soil under wheat crop by a novel and cost-effective method and evaluation of its carryover effect. *J Environ Sci Health.* **42**: 27–31.
- Sondhia S. 2008. Determination of imazosulfuron persistence in rice crop and soil. *Environ Monit Assess.* **137**: 205–211.
- Sondhia S. 2009. Persistence of metsulfuron-methyl in paddy field and detection of its residues in crop produce. *Bull Environ Contam Toxicol.* **83**: 799–802.
- Sondhia S. 2010. Persistence and bioaccumulation of oxyfluorfen residues in onion. *Environ Monit Assess.* **162**: 163–168.
- Sondhia S, Singhai B. 2008. Persistence of sulfosulfuron under wheat cropping system. *Bull Environ Contam Toxicol.* **80**: 423–427.
- Srivastava A, Agarwal V, Srivastava P C, Guru S K, Singh G. 2006. Leaching of sulfosulfuron from two texturally different soils under saturated moisture regime. *J Environ Agric Food Chem.* **45**: 287–290.
- Stotzky G. 1965. Microbial respiration. In Black C A (ed.) Methods of Soil Analysis. Part 2. American Society of Agronomy, Madison. pp. 1550–1572.
- Wu Q, Chen X, Xu Y, Han L. 2010. Dissipation and residues of nicosulfuron in corn and soil under field conditions. *Bull Environ Contam Toxicol.* **85**: 79–82.