



Study on Movement and Accumulation of Trifluralin in Medium-Textured Soils

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ARTICLE INFO

Research Article

Received 12 February 2017

Accepted 15 April 2017

Keywords:

Trifluralin
Lysimeter
Irrigation
Soil
Accumulation

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ABSTRACT

The aim of the study was to evaluate the movement and accumulation of 2,6 – dinitro – NN – dipropyl – 4 - trifluoromethylanil (trifluralin) in soil under irrigated conditions. Despite its hazardous effects this herbicide is widely used in the country. The herbicide researched, is known to be one of the most resistant and least mobile pesticides used in the country. The investigations were carried out, using drainage type lysimeters with application of two different doses of trifluralin and three irrigation water levels. Irrigations were applied during three stages used as indicators for irrigation scheduling of the sunflower crop. Disturbed and undisturbed soil samples were taken from the original field in the beginning and from the tanks after completing of the study. Trifluralin analyses were completed using gas chromatography technique. The results of the study determined that the amounts of the herbicide and its degradation product (2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole) in the ranges of 3.04 – 0.1 and 4.128- 0.344 ppm, respectively were accumulated in the lysimeter soils during the 5-year research period. The highest amounts of trifluralin and its degradation product were measured in the 0-30 cm soil layer, of the treatment with higher applied herbicide amount and deficit irrigation.

DOI: <https://doi.org/10.24925/turjaf.v5i7.780-785.1189>

Introduction

To prevent 15-30% of various disease and harmful effects in a variety of cultured plants, the use of cheap pesticides appears to be easiest way compared to other agricultural defense methods. As a result since the 1950s increasing doses have been used globally. Among the different groups of pesticides, herbicides are more likely to pollute the soils (Temur et al., 2012).

Globally, in our country and in the Thrace region, herbicides with greater efficiency compared to other pesticides take first place in the list of pesticides used. In Turkey 37% of used herbicides are used for the grain group plants with the most commonly used reported to be 2,4-D, trifluralin and propanil (Dag et al., 2000).

In the research area of the Thrace region the amount of pesticides used reaches 8 to 10% of the pesticide amount used in Turkey. According to determination of pesticide amount per unit area, the total pesticide dose yearly used in Turkey is more than 1030 g/ha (Delen et al., 2015).

The Thrace region is an important area for Turkey's agricultural potential with wheat and sunflower rotation common in arable land. More than 56 % sunflower production in our country is provided by the experimental region.

According to pesticide classification based on formulation, trifluralin is in the emulsion concentrate (EC) group. This group is the most commonly used group, with the most important characteristic being immediate mixing with water and ability to be stored without degradation for long periods. Additionally due to the physicochemical characteristics of trifluralin, it remains in soil for long periods and can reach underground water sources.

After trifluralin is applied to the soil as a weed removing tool, it causes environmental pollution due to evaporation, leaching from the soil profile, accumulation in soil and contaminating underground water. In addition trifluralin applied to sunflower reduces germination of wheat seeds in crop rotation, causing abnormally low germination and sparse growth. This event causes economic loss of the significant economic input of seeds and significant falls in productivity.

Trifluralin is a carcinogenic material according to data from the EPA, FAO and International Cancer Research Center and is highly toxic for aquatic organisms even in low doses. According to the final declaration of the III North Sea Conference, a reduction of 50% in trifluralin was predicted from 1985 to 1995 due to these

characteristics. Again, due to these negative characteristics research investigating the migration and accumulation of residues of this pesticide and derivatives in soil and water was completed (Bengtson et al., 1990; Feagley and Kim, 1995; Kim and Feagley, 2002; Kodesova et al., 2011; Querejeta et al., 2014). Some researchers claim that when herbicides like trifluralin are applied to soil they are in contact with adsorbative soil surfaces. The adsorption causes the herbicide to be held by soil colloids, with direct effects or leaching from the surface to deeper soil, removal by evaporation and controlling of retention determining the movement of herbicide in soil and potential to create pollution (Tiryaki et al., 1997; Kim and Feagley, 1998). Recently scientific publications discussing the changes undergone by herbicides in soil and movement in water on a large scale can be found in the literature (Müller et al., 2007; Arias-Estevéz et al., 2008; Kanburoglu – Çebi et al., 2016).

There are many factors proposed to control the accumulation of residues in soil or leaching and transportation of pesticides applied in agriculture. Nelson et al., (2000) emphasized the importance of agricultural applications and pesticide applications before watering, while others (Gardner and Branham, 2001) proposed irrigation method, characteristics of the applied herbicide and dose.

The toxic effects of pesticides as trifluralin last for many years due to different biological degradation rates in the ecologic chain and residues in plants, and linked to this, in animal products. Because of the mentioned hazardous effects, these pesticides should be very carefully and consciously used and their application should be supported by scientific data obtained under controlled conditions. The transport, leaching and accumulation of herbicide in soil under application of different trifluralin doses and irrigation water amounts were investigated in the study conducted in lysimeters.

Materials and Methods

The research was completed using 15 drainage type lysimeters at the Atatürk Soil and Water Agricultural Meteorology Research Institute located 4 km west of

Kırklareli in the northern section of the Marmara Region. The area of the Research Institute is 655,034 ha, located at 41°42' latitude and 27°14' longitude with 233 m elevation.

Lysimeter Experiments

The lysimeter unit consists of 15 tanks in the form of independent fiberglass-reinforced cylinders in three blocks. The cylindrical tanks have a depth of 1.5 m, diameter of 1.20 m and the surface area of the tank is 1.13 m². These CPT (fiberglass) cylinder tanks are resistant to temperatures between -40°C to +112°C, with no water leakage from the inner surface structures. The base of the lysimeter was filled with 0.10 m of soil-free coarse sand-pebble mix. Above this mixture the layer of 1.0 m of experimental soil was placed, taking note of natural soil layering and following procedures published by Gungor (1985). In order to provide flooding during irrigation applications, the upper part of 0.40 m of each lysimeter was left empty. The soil in the lysimeter was non-calcareous brown soil from the Alfisol order of Soil Taxonomy, common in a 438,093 ha area (32%) of the Meric basin.

Soils

The basic chemical and physical properties of the soil placed in the lysimeter tanks are summarized in Table 1.

Characteristics of Trifluralin Used in the Study

The polluting potential of pesticides is linked to the resistance of the pesticide, evaporation characteristics and mobility in soil, in addition to factors like soil and climate. The resistance of a pesticide in soil varies according to persistence duration. The herbicide researched, within the commonly used pesticide classification in Turkey, is known to be one of the most resistant pesticide groups. According to evaporation constant (Henry constant-K_H) trifluralin is in the classification group with highest evaporation (K_H=2.5*10⁻⁵), with mobility in soil among the lowest of agricultural chemicals. The chemical structure and main characteristics of trifluralin are presented on Figure 1 and in Table 2, respectively.

Table1 Some physical and chemical characteristics of the studied soil

SL	pH	OM	EC	BD	FC	WP	S	L	C	TC
0-30	5.1	0.74	1.92	1.44	11.86	4.32	62.00	23.45	14.55	Sandy-loam
30-60	4.8	0.71	2.68	1.67	25.46	14.35	47.60	18.17	34.23	Sandy-clay-loam
60-100	5.2	0.63	3.09	1.67	33.16	21.83	37.70	13.80	48.50	Sandy-clay

SL: Soil layers (cm), OM: Organic matter (%), EC: EC (dS/m), BD: Bulk density (g/cm³), FC: Field capacity (pw), WP: Wilting point (pw), S: Sand (%), L: Loam (%), C: Clay (%), TC: Texture class

Table 2 Some chemical, physical and toxicological properties of trifluralin*

CAS RegistryNumber	1582-09-8
Chemical formula	C ₁₃ H ₁₆ F ₃ N ₃ O ₄
Molarmass	335.28 g/mol
Appearance	Yellowcrystals
Melting point	46 to 47°C (115 to 117°F; 319 to 320K)
Boiling point	139 to 140°C (282 to 284°F; 412 to 413 K) (at 4.2 mmHg)
Solubility in water	0.0024 g/100 mL
Lethal dose or concentration (LD, LC): LD ₅₀ (Mediandose)	500 mg/kg (rat, oral)

Source: Mackay et al., 2006

Experimental Design and Experimental Procedure

The lysimeter study was carried out using sunflower plants in which farming trifluralin herbicide is commonly used. Five experimental treatments in three replication were included in the 5-year study laid out in a randomized plot design. Irrigation scheduling was based on water depletion levels of 0-90 cm soil layer during the specific growth stages, while herbicide applications were done in way similar to that of the farmer applications in the region.

The experimental treatments applied in the lysimeter study were designed as follow:

T1S1- trifluralin applied in traditional dose used by the farmers and irrigation water amount adequate to replenish soil moisture deficit in 0-90 cm layer.

T1S2- trifluralin applied in traditional dose used by the farmers and 75% of water amount adequate to replenish soil moisture deficit in 0-90 cm layer (25% deficit water level)

T1S3- trifluralin applied in traditional dose used by the farmers and 125% of water amount adequate to replenish soil moisture deficit in 0-90 cm layer (25% excess water level)

T2S1- trifluralin applied in dose 1.5 times used by the farmers (50% herbicide excess level) and irrigation water amount adequate to replenish soil moisture deficit in 0-90 cm layer

T2S2- trifluralin applied in dose 1.5 times used by the farmers (50% herbicide excess level) and 75% of water amount adequate to replenish soil moisture deficit in 0-90 cm layer (25% deficit water level).

Irrigation Water and Herbicide Applications

Irrigation applications applied three times in a growing season were based on three known sunflower plant growth stages, namely heading, beginning of flowering and milk stage. All the experimental treatments were irrigated at the same time and various irrigation water amounts as described above were applied. Soil moisture content of the soil in lysimeters was monitored (for 0-30, 30-60 and 60-90 cm with a previously calibrated neutron probe (CPN, 503 DR), using one access tube located at the center of each lysimeter tank (Figure 2).

The trifluralin herbicide was applied each year approximately 10 days prior to seeding, at doses calculated on the basis of that used by the farmers in the region and in accordance of each experimental treatment. The dose of 2.0 L/ha of the herbicide commonly used by sunflower farmers, adjusted for the surface area of lysimeter tanks and 1.5 times of its amount were sprayed respectively on the surface of T1 and T2 tanks and incorporated in soil depth of 10-15 cm.

Soil Sampling

In order to prevent any disturbance of soil structure of the soil in the experimental tanks, disturbed and undisturbed soil samples from 0-30 cm, 30-60 cm and 60-90 cm soil layers were taken from the lands of origin (pasture with no herbicide) of the soil placed in lysimeter tanks in the beginning and from the tanks after

completing of the study. The soil samples obtained from the lysimeters were analyzed for trifluralin and its degradation product in the Residue Laboratories of the Ataturk Soil Water and Agricultural Meteorology Research Institute in Kırklareli- Turkey and that of the Chemistry Department of Ioannina University in Greece following methods described below.

Trifluralin analyses were completed using a PERICHRON PR 2100 brand gas chromatograph. The ECD detector (electron capture detector) had column doors of 0.32 mm inner diameter and 25 m length. The column, detector and injection temperatures were respectively 280°C, 230°C and 230°C, while the injection volume was 1 µl. Helium, nitrogen and hydrogen gases were used. Retention time of trifluralin was 8.84 min. Soil analysis was completed with EPA Method 3541: *Automated Soxhlet Extractio*. Soil samples of 5 g weight and 1:1 acetone-hexane extraction mixture were applied in the procedure (Lopez-Avila, 1991).

-2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole derivative was defined in the Ioannina University Chemistry Department in Greece using GC-MS. Later readings using GC determined the concentration from the area of the peak. The chromatographic conditions of GC-MS applied for the analysis in the laboratory are described earlier (Albanis et al., 1998).

Soil physical and chemical properties such as pH, EC, texture components, wilting point and organic matter were determined in disturbed soil samples, while bulk density and field capacity analyses were performed using undisturbed soil samples collected also from the lands of origin and following procedures given by Richards (1954) and Tuzuner (1990).

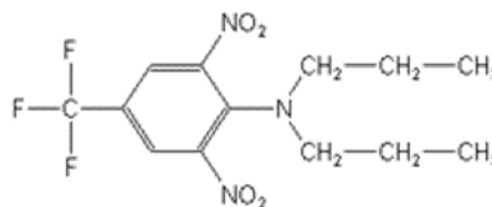


Figure 1 Chemical structure of trifluralin

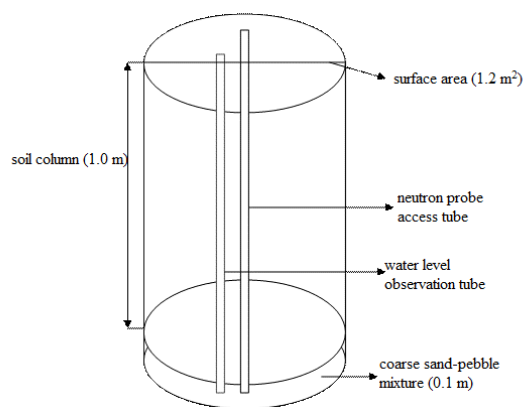


Figure 2 Schematic view of a lysimeter tank used in the study with neutron probe access tube

Statistical Evaluation

Laboratory data related to availability of trifluralin and its degradation product in the evaluated soil layers were subjected to statistical analysis procedure using JUMP 10 software program (SAS 2012) and the analysis of variance was determined.

In addition Duncan's multiple-range test procedure was applied to data indicating a significant effect of the pesticide and watering level on accumulation of trifluralin in the layers of the experimental soil, and Duncan's groups were determined.

Results

Soil sampling was completed at the beginning (2003) and end (2007) of the study. The soil used for the lysimeter initially was taken from a pasture without herbicide application, so initial soil samples in the research found no trifluralin residues.

Lasting five years, the research was completed in fall 2007 and trifluralin residue amounts in soil samples from the lysimeter are included in Table 3.

Evaluation of results from analysis of samples taken from different soil depths at the end of the study showed variations in the trifluralin residue amount in soil linked to the applied dose and applied irrigation water amount. Thus, the highest trifluralin residue amount was found in T2S2 lysimeter with 1.5 times normal herbicide dose and 75% irrigation water and T2S1 soil with 1.5 times normal dose and full water application. While the lowest pesticide residue levels were identified at different depths in the lysimeter (T1S3) with normal trifluralin dose and 1.25 times water applied.

Evaluation of analysis results revealed that with full application conditions, there were differences in the herbicide residue amounts with variations in soil depth. At full dose (farming application dose) trifluralin and irrigation water amount adequate to replenish soil moisture deficit (T1S1), for each of the three repetitions the highest residue amounts were of 1380, 1423 and 172 µg/L were detected in the 0-30 cm soil layer and lowest residue amounts of 800, 124 and 100 µg/L in the deepest 60-100 cm soil depth layer. When 25% less water is applied, in T1S2 lysimeter, the deepest level had less transport of pesticide so residue amounts intensified in the 0-30 and 30-60 cm layers. Compared to the full water (S1) and 25% more water (S3) applications, much lower amounts reached the deepest layer (60-100 cm) and there was much less accumulation.

Pesticides undergo chemical and microbial degradation process in the soil environment (Tiryaki et al., 2004). The result of these processes is that the pesticide is not the main material in soil, some pesticides leave residues in the form of degradation derivatives that are sometimes even more polluting than the pesticide itself. Research has shown that the degradation derivative of trifluralin is 2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole, defined by Prof Dr. A. Albanis of the Ioannina University Chemistry Department in Greece using GC-MS.

The amount of 2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole derivative determined in soil of lysimeter is given in Table 4. When the values in the table are investigated and the degradation derivative 2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole is compared with the amount of trifluralin residue, it appears that the concentration of the degradation derivative is higher than the main material.

Discussion

In our research, carried out under lysimeter conditions with different amounts of irrigation water applied it was determined that the increase in watering amount decreased the amounts of herbicide accumulated within the soil profile probably due to increased leaching rates as result of applied access water. With more irrigation water applied, the trifluralin residues in upper layers appeared to be lower than that in deeper levels. In this way, under conditions of the T1S3 treatment including application of 120 and 240 mm more irrigation water amount compared to S1 and S2 applications respectively, had greater accumulation of trifluralin residues at 60-100 cm soil depth. Thus, with the same dose of herbicide applied in the T1S2 and T1S1 applications, the amount of trifluralin residue at 60-100 cm depth was 341 and 986 µg/L respectively, while in the T1S3 application probably due to application of access water, the trifluralin concentration at this depth reached levels of 1485 µg/L. Kim and Feagley (2002) reported that the herbicides (trifluralin included) on the soil surface are subject to a downward movement into the soil profile due to leaching with water though trifluralin was strongly adsorbed on soil and showed negligible leaching with most of the herbicide accumulating in the top 15 cm of soil depth under rain fed conditions of the study. Much higher concentrations of trifluralin deeper layers were detected in our study which could be attributed to irrigation water applied in amounts adequate to replenish water deficit in 0-90 cm soil depth or excessive application. Another factor which is probably effective in movement of the herbicide into deeper layers in our study is the medium texture of the soil while the previous study was carried out under clay loam soil conditions in Louisiana. The behavior of pesticides in soil is governed by a variety of factors especially soil texture and clay content, and complex dynamic physical, chemical and biological processes including sorption-desorption, volatilization, chemical and biological degradation, uptake by plants, run-off, and leaching (Arias-Estevez et al., 2008).

The results obtained in our research concerning the effect of irrigation water amount on the movement and accumulation of trifluralin in the soil profile could be explained by some findings previously reported in the literature. A study by Johnstone et al., (1998) determined that in trial years with no rain trifluralin degradation was low enough to be ignored and they determined that the most important climatic factors in the leaching and degradation of trifluralin in soil were effective rain and temperature.

Table 3 Trifluralin residues in the soil layers of lysimeter tanks (µ/L)

LT	L	BS ¹	ES ¹	BS ²	ES ²	BS ³	ES ³	TA	TD*	IL*
T1S1	0-30	ND	1380±110	ND	1423±113	ND	1372±98	1392	b	b
	30-60	ND	1180±80	ND	1116±99	ND	1220±105	1172	ns	ns
	60-100	ND	800±8	ND	124.0±2	ND	100±1	341	ns	c
T1S2	0-30	ND	1256±982	ND	1781±120	ND	1350±110	1463	b	a
	30-60	ND	1744±132	ND	1482±105	ND	984±52	1403	ns	ns
	60-100	ND	1467±110	ND	869±11	ND	620±19	986	ns	b
T2S1	0-30	ND	1528±130	ND	1505±85	ND	2183±130	1739	a	b
	30-60	ND	819±25	ND	1444±113	ND	1532±101	1265	ns	ns
	60-100	ND	108±3	ND	1049±110	ND	1437.2±105	865	ns	c
T2S2	0-30	ND	1765±155	ND	2826±190	ND	3043±150	2545	a	a
	30-60	ND	1275±99	ND	1912±175	ND	1937±99	1709	ns	ns
	60-100	ND	1164±100	ND	1784±109	ND	837±11	1262	ns	a
T1S3	0-30	ND	1331±85	ND	1345±98	ND	1202±100	1293	b	c
	30-60	ND	1558±128	ND	839±65	ND	800±25	1066	ns	ns
	60-100	ND	1808±133	ND	1169±88	ND	1477±108	1485	ns	a

*Group of Duncan separation test, LT: Lysimeter Treatment, L: Layer (cm), BS: Beginning of study (2003), ES: End of study (2007), TA: Treatment average, TD: Trifluralin dose, IL: Irrigation level

Table 4 2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole derivative in soil layers (µ/L)

LT	L	BS ¹	ES ¹	BS ²	ES ²	BS ³	ES ³	TA
T1S1	0-30	ND	3404±42	ND	3376±45	ND	2620±34	3133
	30-60	ND	3244±44	ND	3660±41	ND	1176±18	2693
	60-100	ND	2708±14	ND	ND	ND	ND	903
T1S2	0-30	ND	2324±22	ND	4128±55	ND	2284±21	2912
	30-60	ND	2432±18	ND	3608±59	ND	1508±11	2516
	60-100	ND	1912±15	ND	3464±33	ND	1736±15	2371
T2S1	0-30	ND	1624±11	ND	3456±29	ND	972±5	2017
	30-60	ND	344±5	ND	3616±47	ND	2128±14	2029
	60-100	ND	ND	ND	3392±27	ND	ND	1131
T2S2	0-30	ND	1628±12	ND	3228±36	ND	1312±16	2056
	30-60	ND	2692±25	ND	3932±55	ND	1284±10	2636
	60-100	ND	2488±17	ND	3632±39	ND	792±4	2304
T1S3	0-30	ND	2248±19	ND	3196±32	ND	ND	1815
	30-60	ND	3844±39	ND	3120±28	ND	ND	2321
	60-100	ND	3940±51	ND	2952±19	ND	ND	2297

ND-not detected ns-differences are not statistically proved, LT: Lysimeter Treatment, L: Layer (cm), BS: Beginning of study (2003), ES: End of study (2007), TA: Treatment average, TD: Trifluralin dose, IL: Irrigation level

Stating that trifluralin degradation is linked to humidity and temperature, Jolley and Johnstone, (1994) found increased resistance of trifluralin in drought years. Additionally, Rohde et al., (1980) found that the trifluralin amount lost to surface flow was very limited, and that much higher trifluralin concentrations were reached under conditions of the sprinkler irrigation application compared to flow formed after natural rainfall.

In our study carried out in lysimeter conditions, increasing trifluralin dose was found to increase the trifluralin amount accumulated in different layers of the soil. Under conditions of traditional dose applications (farmer application) in T1S1 and T1S2, the trifluralin amounts accumulated at different depths of the soil were 342 -1392 µg/L and 986-1463 µg/L, while in the T2S1 and T2S2 applications with 50% excessive dose, the herbicide residue amounts reached high levels of 865-1739 and 1262-2541 µg/L respectively. Similar results were determined by Duseja and Holmes, (1978). The researchers determined higher amounts of trifluralin in soil when they applied trifluralin at doses above normal.

Laboratory analysis results on the soil samples from the lysimeter identified high amounts of the 2-ethyl-4-

nitro-6-trifluoromethyl-1H-benzimidazole in soil. Koskinen et al., (1984) identified total of 12 derivatives of trifluralin in soil, while Dimou et al., (2004) identified 7 derivatives. These researchers stated that only 2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole was the most commonly encountered derivative of the transformation derivatives in their research, forming the second main peak.

Conclusion

From the results obtained in a 3-year study, it can be concluded that the trifluralin leaching amounts increase with increase of water amounts applied to plants grown in the lysimeter trials. Under conditions of herbicide and irrigation water amount adequate to replenish water deficit in the soil (farmer application), the highest amounts of trifluralin are accumulated in the upper soil layer of 0-30 cm while the lowest amounts are detected in the soil layer at 60-100 cm depth.

Evaluations of data obtained for two application doses showed that increasing the trifluralin dose caused

different amounts of increase in the layers of the soil linked to watering levels.

The amount of 2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole, a trifluralin degradation derivative exceeded the amounts of trifluralin in different soil layers of the lysimeters used in the study.

Acknowledgements

This paper includes some of the results obtained from a research project (02210L01) funded by the Former General Directorate of Rural Services and General Directorate for Agricultural Research, Ankara

The authors deeply appreciate the valuable contributions of Prof Dr. A. Albanis of the Ioannina University Chemistry Department in Greece for detection of 2-ethyl-4-nitro-6-trifluoromethyl-1H-benzimidazole derivative using CG-MS techniques.

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