

Effects of Crop Management on the Fate of Three Herbicides in Soil

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The effects of the management of straw, nitrogen fertilization, and application rates on the dissipation of metsulfuron-methyl, methabenzthiazuron, and trifluralin were investigated in a 3-year field crop rotation study, complemented with laboratory studies. The dehydrogenase activity in soil was measured as an indicator of the soil microflora. The effects were specific for the herbicides investigated. The amendment of straw accelerated the dissipation of trifluralin and methabenzthiazuron. Furthermore, the movement of trifluralin, but not methabenzthiazuron, in the top 30 cm of the soil was reduced. Nitrogen fertilization and high application rates significantly decreased only the dissipation of methabenzthiazuron in the laboratory. Dehydrogenase activity in soil was influenced mainly by the amendment of straw. There was no evidence for cumulative effects on herbicide dissipation due to crop management within the crop rotation.

Keywords: *Herbicides; degradation; straw incorporation; nitrogen fertilization; application rate*

INTRODUCTION

Crop management can influence soil properties (organic material, structure, pH, nutrients, microflora, etc.) and thereby the degradation, sorption, and leaching of pesticides. Effects of nitrogen fertilization, organic matter, and application rates on the dissipation of herbicides have been reported previously (Hurle, 1982; Domsch, 1992). However, there is little published information on the effect of different crop management in a complete crop rotation. The addition of organic matter and fertilizer will increase biological degradation of herbicides if they encourage growth of degrading microorganisms. Furthermore, the addition of organic substances can catalyze chemical hydrolysis of certain pesticides (Shea, 1985) and increase the adsorption of the compounds to the soil. This results in decreased leaching. For herbicides that impede their own metabolism, reduced application rates should lead to faster degradation.

Our investigations were part of a project to investigate the effects of different crop management and reduced inputs in agricultural systems in Göttingen, Germany. We chose three herbicides on the basis of their dissipation and mechanisms of degradation. Trifluralin is considered to dissipate by volatilization as well as chemical and microbial change of the parent molecule (Laanio et al., 1973). Methabenzthiazuron is degraded predominantly by soil microorganisms (Bunte, 1991). Metsulfuron-methyl dissipates from soil both by microbial action and by chemical hydrolysis (Beyer et al., 1988). We investigated the dissipation of these herbicides and their distribution in the top 30 cm layer of the soil in a 3-year crop rotation (oilseed rape, wheat, and barley). The results from field trials were verified in laboratory studies. In order to investigate the effects of crop management and herbicides on the soil microorganisms, dehydrogenase activity was measured as an indicator of soil microbial activity.

MATERIALS AND METHODS

Field Trials. Two field trials, the first (I) to investigate the effects of application rates and straw amendment with a plot size of 16 m × 4 m and the second (II) to investigate the influence of nitrogen fertilization with a plot size of 2.5 m × 2.5 m, were undertaken at the same site in 1990, 1991, and 1992, on a field at the experimental farm at the University of Göttingen. There were four randomized replicates. The soils (coarse-silty Typic udifluent) had 3.0% (I) and 28.2% (II) sand, 60.4% (I) and 34.2% (II) silt, and 36.6% (I) and 37.6% (II) clay. A pH of 7.2 (I) and 7.1 (II) was measured in 0.01 M CaCl₂. The soil in trials I and II had organic contents of 1.65 and 1.63 g per 100 g of soil, respectively. Field capacities were 33.9% (I) and 29.9% (II). Herbicide applications (with a small plot sprayer), additional treatment with nitrogen fertilizer (manual application), and management of the organic material are listed in Table 1. All other treatments were done in accordance with normal practice. Straw was left on the field after harvesting the previous crop or removed manually and incorporated later. For technical reasons, this could not be achieved in 1989. To investigate the fate of the herbicides and the activity of the soil microflora, 12 soil samples were taken from the center of the plots. The investigated depths were 0–5 cm for metsulfuron-methyl and methabenzthiazuron and 0–10 cm for trifluralin residues. In the spring, further samples were taken from the soil layers 5–15 cm, 15–30 cm (metsulfuron-methyl and methabenzthiazuron), and 10–20 and 20–30 cm (trifluralin). Mixed samples were made from every plot. The soil was sieved (mesh size of 2 mm), dehydrogenase activity investigated immediately as described previously (Berger and Heitefuss, 1991), and herbicide analysis done after storage at –18 °C.

Laboratory Studies. Soil was taken after harvest from untreated plots of the field trials, passed through a 2 mm mesh size sieve, and stored at 4 °C. In order to accustom the soil microorganisms to the conditions of the experiment, the soil was stored at 20 °C for 7 days.

Dissipation of methabenzthiazuron was determined after thorough mixing of soil (50 g dry weight) with a solution of Tribunil (700 g of methabenzthiazuron/kg) in water (3 mL, with concentrations of 88.9 and 889.0 mg of methabenzthiazuron/mL, corresponding to application rates of 4 and 40 kg of Tribunil/ha, presuming a distribution of the herbicide in the soil between 0 and 5 cm). To investigate the effects of organic material and nitrogen, ground, untreated straw (0.53 g), or an aqueous solution of NH₄NO₃ (0.5 mL, 60.2 mg/mL) was added. These amounts correspond to normal practice (800 kg of straw/ha and 158 kg of N/ha), presuming a distribution in the soil layer between 0 and 5 cm. The final soil moisture

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Table 1. Herbicide Application, Nitrogen Fertilization, and Management of Organic Material

	1989/1990 winter wheat	1990/1991 winter barley	1991/1992 oilseed rape
herbicides	Gropper (DuPont)	Tribunil (Bayer AG)	Elancolan (DowElanco)
active ingredient (AI)	metsulfuron-methyl	methabenzthiazuron	trifluralin
concentration of AI	200 g/kg	700 g/kg	480 g/L
full application rates	20.0 g/ha	4.6 kg/ha	2.0 L/ha
reduced application rates	10.0 g/ha	2.3 kg/ha	1.0 L/ha
date of application	March 13, 1990	October 1, 1990	August 21, 1991
	Nitrogen Fertilization (Calcium-Ammonium Nitrate)		
full and reduced application rates (kg/ha)	80, 40 40, 20 30, 15	48, 24 34, 17 50, 25	50, 25 100, 50 —
dates of application	March 15, 1990 April 6, 1990 May 8, 1990	March 13, 1991 April 11, 1991 May 23, 1991	February 28, 1992 April 6, 1992 —
	Management of Straw		
removal	—	August 7, 1990	July 29, 1991
incorporation	—	August 20, 1990	August 2, 1991

was 70% of field capacity and was maintained at that level by the addition of distilled water. The soil was placed in glass bottles, incubated at 20 °C, and analyzed after extraction at different time intervals. Each treatment was performed in triplicate.

Trifluralin dissipation from soil was measured after adding a solution of Elancolan (480 g of trifluralin/L) in water (0.83 mL, 48.0 and 480.0 mg of trifluralin/mL, corresponding to application rates of 2.5 and 25.0 L of Elancolan/ha, calculated for a distribution in the soil layer between 0 and 10 cm, as trifluralin is usually incorporated into the soil). Addition of straw and nitrogen was performed as described for methabenzthiazuron. Soil moisture was 65% of field capacity and was maintained at that level by the addition of distilled water. Incubation temperature was 20 °C. Trifluralin has a high vapor pressure. Its volatilization was determined by conducting wet air through the glass bottles into a gas-washing bottle filled with *n*-decane following the procedure described by Kearney and Kontson (1976). The treatments were performed in duplicate (effect of nitrogen) and in triplicate (effect of straw and application rate). An aliquot of the *n*-decane was sampled in regular intervals and mixed with an internal standard solution. Soil samples were analyzed at the beginning and at the end of each experiment.

Effects of straw and nitrogen fertilization on the soil microflora were investigated after addition of ground, untreated straw (4.26 mg/kg), or an aqueous solution of NH₄-NO₃ (10.0 mL, 20.0 mg/mL) to 1 kg of untreated soil. The soil was placed in plastic containers allowing air exchange and incubated in the dark at 20 °C. The soil moisture was adjusted to 65% of field capacity and maintained at that level by the addition of distilled water. Each treatment was performed in triplicate. After sampling in regular intervals, dehydrogenase activity was measured according to the procedure described earlier (Berger and Heitefuss, 1991).

Methods of Analysis. The concentration of metsulfuron-methyl was determined with a biotest with sugar-beet (*Beta vulgaris* L. ssp. *vulgaris* var. *altissima*) corresponding to the procedure described previously (Berger and Bernd, 1993), similar to the method of Blair et al. (1989) for chlorsulfuron. Methabenzthiazuron and its metabolite 1-(1,3-benzothiazol-2-yl)-1-methylurea were analyzed following a method for phenylurea herbicides by high-performance liquid chromatography (Berger and Heitefuss, 1989). Recoveries between 0.04 and 4.00 mg/kg of soil were higher than 91% for the metabolite and 97% for the herbicide. Trifluralin was determined with gas chromatography (Berger et al., 1992). Recoveries were 87% at a concentration of 0.005 mg/kg and higher than 93% in the concentration range 0.028–0.670 mg/kg.

Standard compounds of the herbicides and their metabolites were kindly provided by DuPont, Bayer AG, and DowElanco.

Statistical Processing. DT-50 values (disappearance time for 50% of the initial residues) of the herbicides were obtained by calculation according to the procedure of Timme et al. (1986) and by linear interpolation from the degradation curves for laboratory and field trials, respectively. Results from the

Table 2. Interpolated DT-50 Values of Metsulfuron-methyl, Methabenzthiazuron, and Trifluralin in Field Trials

	DT-50 values (days)		
	metsulfuron-methyl	methabenzthiazuron	trifluralin
trial I			
full application rate without addition of straw	33	294	285
reduced application rate without addition of straw	23	251	249
trial II			
full nitrogen fertilization	33	268	314
reduced nitrogen fertilization	32	248	320

investigations on the activity of the soil microflora, from the laboratory trials with trifluralin, and from the residues in deeper soil layers were subjected to an analysis of variance followed by an *F* test. Where significant variations were present, differences of means were approved by the Scheffé test for comparing means of both single and combined variants.

RESULTS AND DISCUSSION

Metsulfuron-methyl. Soil samples collected from the plots sprayed with metsulfuron-methyl were analyzed after 1, 7, 14, 28, 56, 128, and 163 days. The DT-50 values of the herbicide are shown in Table 2. Only a slight reduction in herbicide concentration occurred during the first week after application, as the temperature was low. After 7 days, we observed a steady decrease of the residues. There was no herbicide detectable either in deeper soil layers (5–15 and 15–30 cm) or at the end of the vegetation period 128 and 163 days after application (detection limit of 0.001 mg/kg).

The DT-50 values for metsulfuron-methyl determined in our investigations are similar to those observed earlier (Nicholls et al., 1987; Walker et al., 1989), whereas Smith (1986) reported DT-50 values of metsulfuron-methyl of 178 days. These discrepancies can be explained mainly by different pH values of the soils, as the degradation of this herbicide is highly pH sensitive.

There were no significant differences between the degradation curves and interpolated DT-50 values of the two application rates. The concentration obviously had no influence on herbicide dissipation in soil. Similar observations were reported from Ritter et al. (1988) and Wadd and Drennan (1989). Dehydrogenase activity in the soil is shown in Figure 1. There was no influence

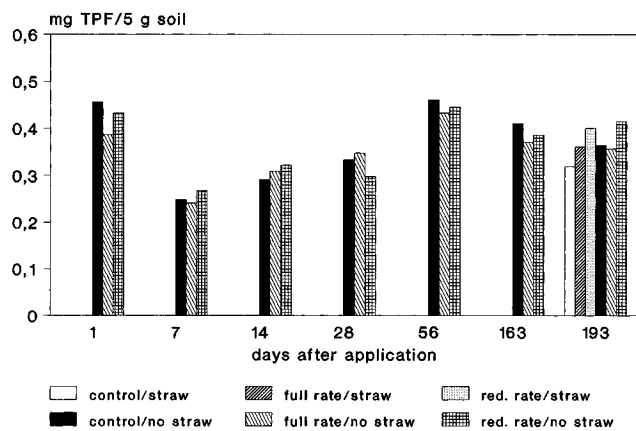


Figure 1. Dehydrogenase activity in soil after application of the full and the reduced (red.) rate of metsulfuron-methyl (40.0 and 20.0 g/ha), with and without incorporation of straw in a field trial.

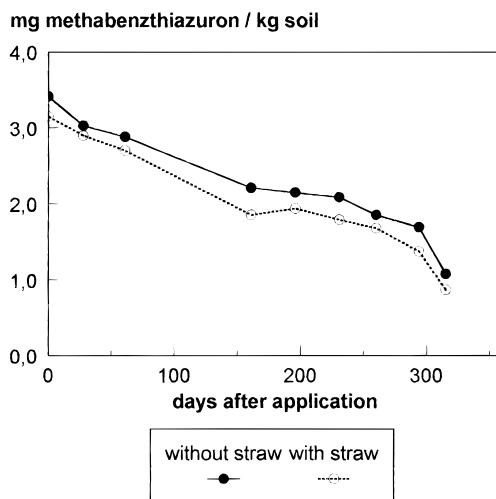


Figure 2. Dissipation of methabenzthiazuron (full rate = 4.6 kg/ha, soil layer 0–5 cm) in soil with and without incorporation of straw.

of the herbicide at the two concentrations on microbial activity. These results are in agreement with laboratory studies from Malkomes (1990), who found little or no effect of this herbicide on different parameters of the soil microflora.

Nitrogen fertilization did not influence the microflora able to degrade metsulfuron-methyl in field trials. There was no effect on the dehydrogenase activity either (data not shown). An influence of nutrients on the bacteriostatic effects of sulfonylurea herbicides in pure cultures (Blair and Martin, 1988) could therefore not be confirmed for nitrogen fertilization in our studies.

As effects were not significant in the field, no additional laboratory experiments were performed with metsulfuron-methyl.

Methabenzthiazuron. Soil samples from the plots sprayed with methabenzthiazuron were collected from the field trials 1, 28, 61, 161, 196, 231, 266, 294, and 315 days after application. The DT-50 values are shown in Table 2. The degradation curves of methabenzthiazuron in soil with and without the amendment of straw are demonstrated in Figure 2. Reduction of herbicide concentration started without delay in all plots. The results of our investigation are consistent with those of other studies, where DT-50 values of methabenzthiazuron between 62 and 300 days were reported from field experiments and between 146 and 521 days from laboratory studies (Pestemer and Auspurg, 1987; Bunte,

Table 3. DT-50 Values of Methabenzthiazuron in the Laboratory Trial with Confidence Intervals and Correlation Coefficients (r), Calculated for First-Order Reaction Kinetics

	DT-50 values (days)	
	calculated	r
full application rate	148.5 ± 23.8	0.9599
10-fold application rate	265.5 ± 62.2	0.9955
full application rate with amendment of straw	93.4 ± 8.9	0.9839
full application rate with nitrogen fertilization	223.6 ± 54.9	0.9228

1991). The highest concentrations of the herbicide in the soil layer 5–15 cm were measured in April with 0.33 mg/kg. Residues in the soil layer 15–30 cm were below the detection limit in all cases.

In the field, lower application rates led to a slightly improved dissipation of the herbicide, where DT-50 was shortened from 294 to 251 days. The effect of the herbicide concentration on the dissipation was confirmed in laboratory studies, where the concentration of the herbicide was measured 0, 7, 21, 35, 70, 105, and 161 days after addition of the chemical to the soil. Table 3 demonstrates that the DT-50 value of methabenzthiazuron significantly increased from 149 days for the full rate to 266 days for the 10-fold concentration used in agricultural practice. Hance and McKone (1971) obtained similar results with linuron, another herbicide of the phenylurea type. Bunte (1991), however, found no effect of the concentration on the degradation, but she used lower application rates in her investigations.

Our results from the degradation studies were supported by investigations on the dehydrogenase activity. The results are shown in Figure 3. In plots with incorporated straw, dehydrogenase activity was significantly reduced 28 days after application of the full rate compared to the control (no herbicide application). A stimulating effect of straw and the inhibitory influence of methabenzthiazuron accumulated to significantly reduced microbial dehydrogenase activity in plots without straw incorporation 28, 61, 196, and 231 days after application of the full rate. Transient inhibitory effects of the herbicide on dehydrogenase activity and straw decomposition have been described previously (Malkomes and Pestemer, 1981). This gives evidence that methabenzthiazuron could impede its own degradation by reducing the activity of the degrading soil microflora.

The dissipation rate of the herbicide was decreased after a supplementary nitrogen fertilization in the field and laboratory, but these effects were not significant (Tables 2 and 3). A similar result was observed in the laboratory, where dehydrogenase activity was reduced after nitrogen amendment (Figure 4). A reduction of the dehydrogenase activity after an additional nitrogen fertilization could also be observed in the field trial and was significant 266 days after application (data not shown). This indicates only a slight influence of nitrogen on herbicide dissipation and dehydrogenase activity. To our knowledge, similar investigations with methabenzthiazuron have not been published previously. Hance (1973), however, found no effect of a nutrient solution on the degradation of linuron, another herbicide of the phenylurea type. Bakalivanov and Hlebarova (1977) reported accelerated detoxification of linuron and metobromuron after fertilizer application, but the authors did not provide statistical significance.

In field trials, herbicide dissipation and dehydrogenase activity were slightly stimulated by the incorporation of straw (Figures 2 and 3). The results are more

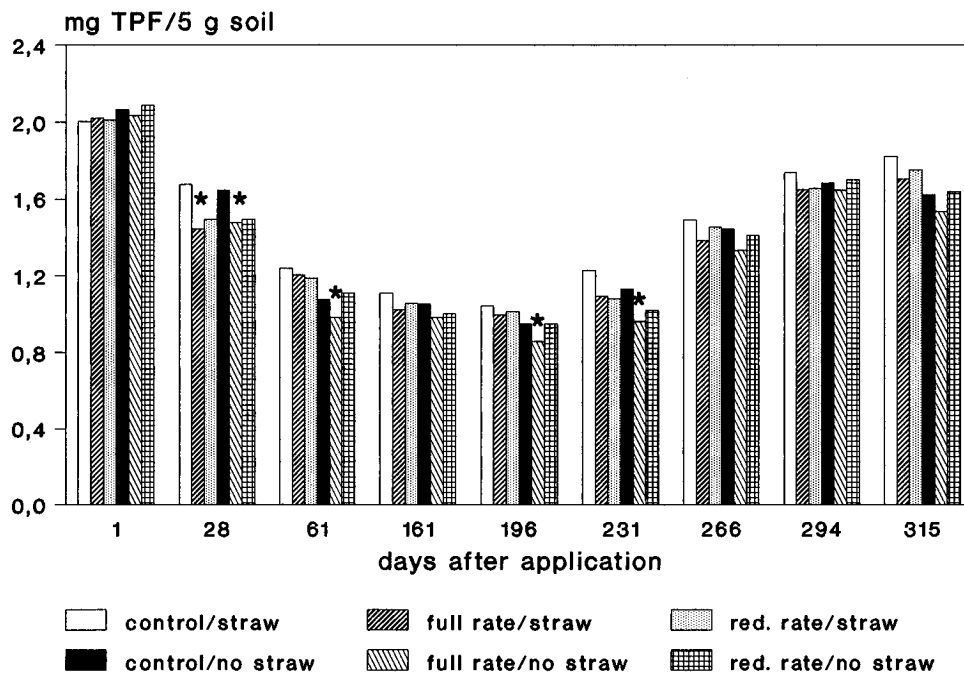


Figure 3. Dehydrogenase activity in soil after application of the full and the reduced (red.) rate of methabenzthiazuron (4.6 and 2.3 kg/ha), with and without incorporation of straw in a field trial. * indicates significant differences at $p \leq 0.05$ (Scheffé) compared to the control with straw incorporation (no herbicide application).

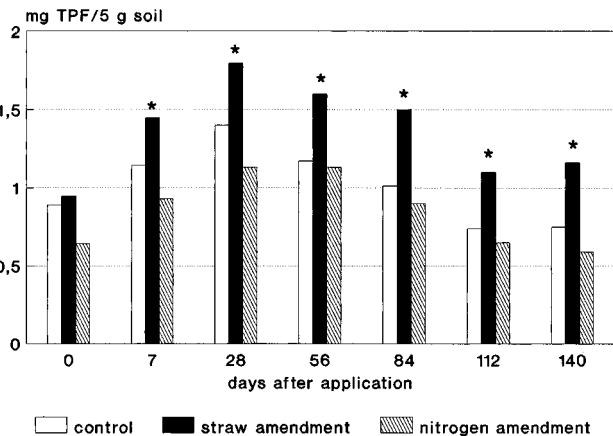


Figure 4. Dehydrogenase activity in soil after amendment of nitrogen and straw, laboratory trial. * indicates significant differences at $p \leq 0.05$ (Scheffé) compared to the control (no straw and no nitrogen amendment).

pronounced in laboratory studies, where decreased DT-50 values of the herbicide (Table 3) and increased dehydrogenase activity (Figure 4) could be observed after straw amendment. High organic matter content can reduce the degradation of methabenzthiazuron due to adsorption (Bunte, 1991) or incorporation into the nonextractable humin fraction (Azam et al., 1988). On the other hand, increased herbicide degradation due to a stimulation of the soil microflora by straw has been reviewed earlier by Hurle (1981) and could have caused the effect. The distribution of the herbicide in the top 30 cm of the soil was not influenced by the management of the organic material (data not shown). This stresses the fact that adsorption of methabenzthiazuron by straw was probably less important.

The concentrations of the metabolite 1-(1,3-benzothiazol-2-yl)-1-methylurea, the main degradation product in soil (Mittelstaedt et al., 1977), were below 0.04 mg/kg in all cases. Even the application of the 10-fold rate in our laboratory studies did not result in significantly higher amounts of the metabolite. According to studies of Führ and Mittelstaedt (1980), the low concentration

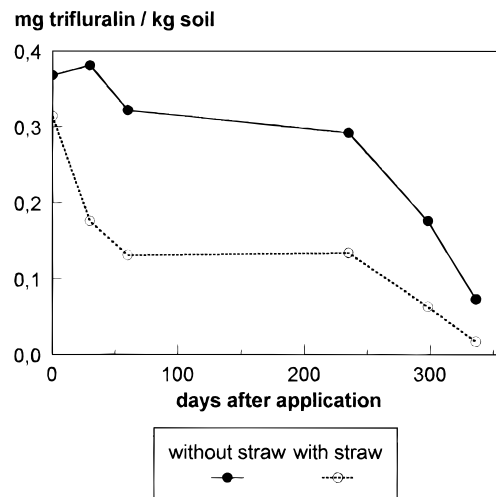


Figure 5. Dissipation of trifluralin (full rate = 2.0 L/ha, soil layer 0–10 cm) in soil with and without incorporation of straw.

of the metabolite can be explained by further degradation and incorporation into the organic fraction of the soil.

Trifluralin. Following herbicide application, soil samples from the two field trials in 1991/1992 were collected after 0, 30, 60, 235, 298, and 336 days (for residues only) and 0, 235, and 336 days for the field trials I and II, respectively. The herbicide dissipates from soil by volatilization, chemical degradation, photodecomposition, and microbial action (Laanio et al., 1973). The DT-50 values are listed in Table 2. The dissipation curves of the herbicide in soil with and without the addition of straw are shown in Figure 5.

Linear interpolation resulted in DT-50 values of trifluralin between 250 and 320 days, indicating a higher persistence of the herbicide in our trials compared to that from studies from Walker and Eagle (1983) and Gaynor (1985). This can be explained by a reduced dissipation due to the low temperatures in winter. Prolonged persistence of trifluralin in periods of low temperatures was also observed by Hayden and Smith

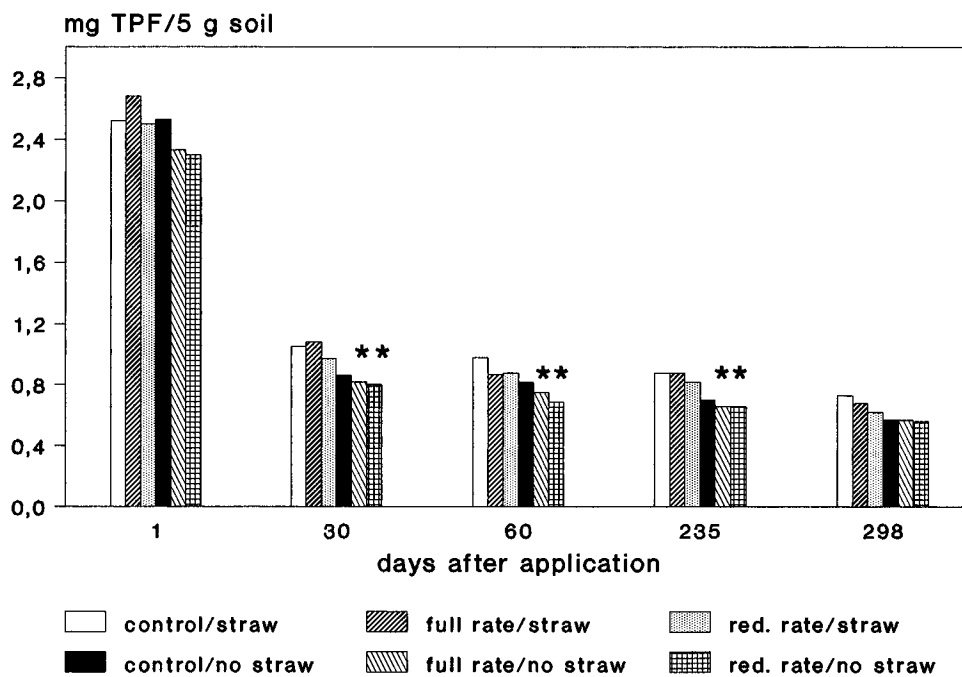


Figure 6. Dehydrogenase activity in soil after application of the full and the reduced (red.) rate of trifluralin (2.0 and 1.0 L/ha), with and without incorporation of straw in a field trial. * indicates significant differences at $p \leq 0.05$ (Scheffé) compared to the control with straw incorporation (no herbicide application).

(1980) and Pritchard and Stobbe (1980). As was the case for methabenzthiazuron, the stimulating effect of straw and a slight inhibitory influence of the herbicide accumulated to reduced dehydrogenase activities. Those were significant 30, 60, and 235 days after application compared to the control with straw incorporation (Figure 6).

Application rates had no influence on the persistence of trifluralin in our field trials (Table 2). This was confirmed in laboratory studies, both for the degradation and for the total volatilization of the chemical (data not shown). Effects of the two concentrations of the herbicide on soil dehydrogenase activity in plots with and without incorporation of straw were not significant when compared to the appropriate control (Figure 6). This result is in accordance with other studies, where herbicide concentrations were used as in normal practice. Drastically enhanced concentrations, however, led to significantly increased persistence (Domsch, 1992).

We found no effects of nitrogen fertilization on the dissipation of the herbicide or on the dehydrogenase activity in our studies (data not shown). Similar investigations have not been undertaken previously, but sewage sludge was shown to lead to a slight inhibition of trifluralin degradation (Doyle et al., 1978).

Dissipation of the herbicide was increased after the incorporation of straw (Figure 5). Significant differences of the dehydrogenase activity between combined variants with and without straw incorporation could be observed 30 days after application (Figure 6) and in the laboratory (Figure 4). The results from these studies were also confirmed in a laboratory trial. Volatilization, as an important process of trifluralin dissipation from soil, was significantly reduced in the soil after incorporation of straw, probably due to adsorption to the organic material (Table 4). Still, residues of trifluralin were lower in that soil 24 days after application. This indicates enhanced dissipation of the herbicide after incorporation of the organic material. Increased degradation rates of trifluralin after addition of organic material (dairy manure) in the laboratory under aerobic conditions had been reported previously (Doyle et al.,

Table 4. Dissipation (in Milligrams of Trifluralin per Kilogram of Soil) of Trifluralin from Soil in a Laboratory Trial 24 Days after Application, with and without Incorporation of Straw

treatment	amount volatilized (mg/kg)	residues (mg/kg)	total amount dissipated (mg/kg)
without straw	0.1088	0.3259	0.2189
with straw	0.0866	0.3072	0.2376
LSD ^a ($p \leq 0.05$, Scheffé)	0.0221	0.0821	— ^b

^a Least significant difference. ^b No LSD, as the values were calculated.

1978) and explained by assuming stimulation of microbial activity (Shea, 1985). In June, no residues in the soil layer 10–20 cm were detectable in those plots where straw had been incorporated. Concentrations of 0.013 mg of trifluralin/kg of soil, however, were measured in soil without amendment of organic material. This stresses the importance of straw incorporation for the adsorption and thereby distribution of trifluralin in soil. No herbicide could be observed in the soil layer 20–30 cm.

CONCLUSION

Our results indicate that nitrogen and straw fertilization as well as different application rates can have a significant influence on the fate of herbicides and the dehydrogenase activity as an indicator of the microbial activity in soil. Management of organic material, e.g. incorporation of straw, was the most important factor to influence pesticide dissipation and the dehydrogenase activity in soil. The results stress the importance of an appropriate management of organic material in order to reduce concentration and movement of the parent herbicide in soil, but no cumulative effect of crop management on the fate of herbicides could be observed.

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