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Thiazopyr Behavior under Laboratory Conditions

Chandra Whitworth Milam
July 22, 1995
TN Scholar Report
Introduction

In today's society, environmental concerns are at the forefront of federal policy, and the general public is preoccupied with the future of Mother Earth. For this reason we have laws that protect the human population and the environment against harmful substances. Many different tests for every conceivable situation are run on a chemical before it reaches the market. Thiazopyr is one such chemical that is waiting to be approved. The purpose of this study was to see how two different temperatures affected the degradation of thiazopyr in the soil, and to determine the effect of microbial degradation on the herbicide. Thiazopyr is a herbicide in the pyridine family that will be used to control weeds during the growing season. Thiazopyr controls weed growth by interrupting spindle microtubule formation. It has little soil mobility and its half life in the soil is between 8 and 150 days. Thiazopyr has low water solubility, only 2.5 ppm. It also has low mammalian toxicity, but moderate fish toxicity. The following is an account of how the study was conducted and the results of the test.

Materials and Methods

The study began with the determination of soil moisture to compute how much soil would actually be needed to have 40g. The soil used was the Knoxville Sequatchie loam. This soil has a pH of 6.2, organic matter content is 1.6, and the CEC was 14. The use of the soil prior to this study was for growing soybeans, and the soil had never been treated with thiazopyr. Before determination of soil moisture, 8 kg of soil was collected from the 0 to 8 cm soil zone and the samples were passed through a 2 mm sieve while moist. The soil was then placed in plastic bags, and stored at 4°C for one week before the experiment. Determination of soil moisture was accomplished by drying a known
amount of soil, weighing it after drying at 140 degrees C, and computing the percentage water in the soil. Using the information obtained from drying the soils, I put 45.41 g of soil into sterile 250 ml polyethylene bottles that had screw top caps. The degradation of the herbicide was tested at 15 and 30 degrees Celsius for a period of eight weeks. Each week was done in triplicate for each temperature, and each bottle was considered an experimental unit. One and a half milliliters of a 28 ppm solution was added to the soil in the bottles so that the concentration added to the soil was approximately 1.05 ppm. The stock solution was made by dissolving 8.4 mg of herbicide in 180 ml of water and 120 ml of methanol. After applying the herbicide, each bottle was tightly capped and shaken by hand for two minutes in order to assure that the soil was evenly covered with thiazopyr. Time zero was immediately placed in the freezer at -10°C, and the rest of the bottles went into two incubators, half at 15°C and the others at 30°C. There was no light in the incubators. At the end of one week, 6 bottles were removed and placed in the freezer at -10°C until analysis.

At the end of the eighth week, the stock solution of 10500 ppb was diluted to make standards of 1000, 500, 250, 100, 50, 25, 10, and 5 ppb. The standards were then run on a gas chromatograph, and the method showed good recovery. In order to find the percentage absorbed to the soil and to determine the accuracy of the extraction method, 12 samples were run. The untreated soil samples were again placed in 250 ml polyethylene bottles. The first four bottles were filled with approximately 40 g of soil and treated with 4 ml of a 10.5 ppm solution of the herbicide. A second set of four bottles were filled with 40 g of soil and spiked with distilled water. The third set of four bottles was a straight solution of the 10.5 ppm stock solution. After weighing out the soil and spiking the bottles, the bottles were aired for approximately one hour. At the end of that time, all the bottles were shaken by hand for two minutes, and then they were capped and stood for approximately 24 hours. Then 80 ml of methanol was added to each bottle
to extract the herbicide, and the bottles were shaken overnight on a reciprocating shaker which agitated for 16 h at 240 excursions per minute. After the soil in the bottles settled, they were filtered using 4 pieces of qualitative filter paper to remove particulates. After each sample filtered the funnel was rinsed with methanol and dried with Kimwipes®, and an aliquot placed into a 1 ml GC autosampler vial. A similar extraction procedure was followed for the samples of the herbicide degradation at the two different temperatures once they were taken out of the freezer. After the samples had warmed up, they were extracted with 80 ml of methanol and shaken overnight. After the soil settled, they were filtered using 2 pieces of qualitative filter papers; again the funnels were rinsed with methanol and wiped dry with Kimwipes® after each sample was filtered. Once extracted, the samples were run on the gas chromatograph. Several sets of temperature programs were investigated, and the optimum analytical conditions consisted of injection port at 230°C, initial column oven temperature at 170°C for 1 minute followed by a 6 minute ramp at 7 degrees per minute to reach 210°C, and the nitrogen-phosphorus detector was operated at 250°C. A 30 m Alltech SE-30, 0.54 mm ID, with 1.2 micron film thickness column was used. The retention time under these conditions was 9.78 minutes with a peak width of .095 to 0.098 min.

Data analysis. The data were empirically fit to first order kinetics (Walker). The soil thiazopyr concentration in ppbw was converted to the natural logarithm and then linearly regressed against time in days to best fit the equation:

\[ C = C_i \cdot e^{(k \cdot DAT)} \]

\( C \) = thiazopyr concentration
\( C_i \) = initial thiaizopyr concentration
\( k \) = first-order rate constant
\( DAT \) = days after treatment

The first-order dissipation rate and the 95% confidence interval were calculated. To separate treatment effects, confidence intervals were compared. If the confidence
intervals overlapped, then dissipation rate was not different. Conceptually, this is a conservative approach because there are two standard deviations from each rate constant. For two treatments to be different, four standard deviations are necessary, so the significance level is $\approx 1\%$ (Zar). The half-life values in days were based on the first-order regression equations, and were calculated by the relationship: half-life in days $= 0.693/k$ (Walker). A "corrected" $r^2$ value was determined by the formula $r^2$ is equal to one minus the residual sums of squares divided by the corrected total sums of squares.

**Results and Discussion**

The results of thiazopyr dissipation under laboratory conditions are shown in Figure 1. Thiazopyr dissipation under laboratory conditions was much lower than in the field, and was slower at 15°C than at 30°C. Calculated initial half-lives were three times greater at 15°C than at 30°C and were ten times greater at 15°C when compared with the field results. This laboratory test system greatly reduced or eliminated the dissipation processes of photo-degradation, volatilization, and leaching into subsurface soil zones. Because temperature had a major effect, microbial degradation is suggested to be the significant component of the total dissipation process, but other dissipation mechanisms play a role in thiazopyr degradation under field conditions.
Figure 1. Thiazopyr concentration over time in soil as influenced by incubation temperature under controlled conditions. Datapoints are means of four replications ± one standard error. The line represents the first-order regression equation. Rate constants, calculated half-lives, and $r^2$ are in Table 1.
Table 1. Herbicide dissipation rate constants (K) and calculated initial half-lives for thiazopyr in 0-8 cm soil.

<table>
<thead>
<tr>
<th>Year</th>
<th>Location</th>
<th>K</th>
<th>Confidence Interval</th>
<th>$r^2$</th>
<th>half-life</th>
</tr>
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<tr>
<td></td>
<td></td>
<td>per d</td>
<td></td>
<td></td>
<td>d</td>
</tr>
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<td>1993</td>
<td>Knoxville</td>
<td>.057</td>
<td>(.0399, .0742)</td>
<td>0.97</td>
<td>12</td>
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<tr>
<td>1994</td>
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<td>.051</td>
<td>(.0371, .0664)</td>
<td>0.97</td>
<td>13</td>
</tr>
<tr>
<td>1993</td>
<td>Jackson</td>
<td>.049</td>
<td>(.0196, .07843)</td>
<td>0.92</td>
<td>14</td>
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<tr>
<td>1994</td>
<td>Laboratory-</td>
<td>.005</td>
<td>(.0038, .0065)</td>
<td>0.92</td>
<td>133</td>
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<td></td>
<td>15 degrees</td>
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<tr>
<td>1994</td>
<td>Laboratory-</td>
<td>.016</td>
<td>(.0091, .0233)</td>
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<td>43</td>
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<td></td>
<td>30 degrees</td>
<td></td>
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Literature Cited
