Effects of long-term irrigation with reclaimed wastewater on the efficacy and fate of trifloxsulfuron-sodium in the soil

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Summary

Long-term irrigation with reclaimed wastewater (RWW) is known to affect the physical, chemical and biological properties of the receiving soil. The sulfonylurea herbicide trifloxsulfuron-sodium (TFX), an acetolactate synthase inhibitor, is stable in the soil for several months and can carry over to rotational crops; however, its activity in RWW-irrigated soils is much lower. In this study, we monitored the activity/dissipation of TFX applied on RWW-irrigated soil. High weed infestation in the treated soil in field studies along with a decrease in the soil activity as determined with test plant was observed, suggesting fast dissipation of the herbicidal activity of TFX in the soil. Dose–response curves of Sorghum bicolor to TFX planted in RWW- and in fresh water-irrigated soils showed higher and more significant damage for both shoots and roots with the fresh water-irrigated soil. Sterilised RWW-irrigated soil exhibited higher TFX activity versus non-sterilised soil. This implies that reduction in TFX activity in RWW-irrigated soil is probably affected by the microbial activity.

Keywords: Sorghum bicolor, sulfonylurea, dissipation, microbial activity, recycled water.

Introduction

In semi-arid zones, water is a limited resource for intensive agriculture. The use of reclaimed wastewater (RWW) for irrigation in agriculture allows freshwater use to be directed away from the agricultural sector to domestic and industrial sectors. The use of RWW for crop irrigation in Israel has started in the early seventies and has dramatically increased since 1985, now reaching about 50% of the total irrigation water (Navon et al., 2011).

Long-term irrigation with RWW is known to affect the physical, chemical and biological properties of the soil. These effects include reduction in the soil’s hydraulic conductivity, permeability and infiltration, an increase in the sodium adsorption ratio (SAR), and changes in the soil’s microbial community and its attendant activities (Ovreas et al., 1998; Levy et al., 1999; Beaulieu et al., 2000; Mamedov et al., 2000). Moreover, the influx of relatively high concentrations of dissolved organic matter (DOM) into the soil (Jueschke et al., 2008) might have a major influence on the interactions between soil-applied pesticides (mainly herbicides) and the soil matrix (Graber et al., 1995; Seol & Lee, 2000; Drori et al., 2005, 2006; Ilani et al., 2005; Muller et al., 2007). Numerous studies have shown that...
the addition of easily degradable organic compounds can cause an increase in the microbial population (Friedel et al., 2000; Kuzyakov et al., 2000). DOM may be the most important carbon source in soils, because all microbial uptake mechanisms require an aqueous environment (Metting, 1993); continued influx of DOM to soils by RWW irrigation is expected to affect the soil’s microbial activity (Arye et al., 2008).

Acetolactate synthase (ALS)-inhibiting herbicides are widely used in many crops, because they are effective in controlling a broad spectrum of weed species at low use rates, resulting in reduced environmental chemical loading (Wilcut et al., 1996; Wright & Penner, 1998; Porterfield & Wilcut, 2006), and are residual in the soils of Israel (pH, 7.5–8.4). Most sulfonylurea herbicides remain stable in the soil for several months and can carry over to rotational crops (Johnson et al., 1995; Seifert et al., 2001). The sulfonylurea herbicide trifloxysulfuron-sodium (TFX) is an ALS inhibitor registered for post-emergence weed control in cotton (Gossypium spp.), sugarcane (Saccharum officinarum L.) and turf (Minton et al., 2008). Namenek et al. (2001) reported that TFX applied pre-emergence provided more than 80% control of Amaranthus palmeri S. Watson (Palmer amaranth), Ipomoea lacunose L. (pitted morning glory), Sesbania herbacea (Mill.) McVaugh (hemp sesbania) and Senna obtusifolia (L.) Irwin & Barneby (sicklepod).

Until the introduction of TFX, pyrithiobac was the only selective post-emergence (POST) herbicide for annual broad-leaved weed control that could be applied over the top in cotton and did not risk the yield (Culpepper et al., 2000; Porterfield & Wilcut, 2006). Pyrithiobac and TFX are the only options for over the top POST application in tank mixtures for selective Cyperus rotundus L. (purple nutsedge) annual broad-leaved weed control in non-transgenic cotton cultivars.

Trifloxysulfuron-sodium degradation in the soil is primarily controlled by abiotic hydrolysis, which increases with increasing temperature and in acidic pH condition (Hudetz et al., 2000; Matocha & Senseman, 2007). Soil half-life time of TFX was reported to vary between 22 and 52 days. Minton et al. (2008) have found that maize grown in soil collected 60 days after TFX application (7.5 g ha⁻¹) resulted in 48% injury 21 days after application (DAP). These results and current label restriction indicate long-lasting soil activity (Anonymous, 2003). Microbial degradation of herbicides has been broadly reported in the last two decades (Freund et al., 1994; Braschi et al., 2000; He et al., 2006; Krutz et al., 2008; Mandelbaum et al., 2008). These reports have shown that there is an accelerated degradation of herbicides in the soil because of increased microbial activity. TFX effect on microorganisms in the soil has been studied, and it was found that when added to soil it increased soil respiration rate and CO₂ emission rate, indicating growth enhancement of the microbial population (Reis et al., 2008). However, studies reporting pyrimidinyl sulfonylurea-accelerated dissipation in the soil are scarce (Valle et al., 2006; Xu et al., 2009; Zhang et al., 2009). The objective of this study was to evaluate the effect of long-term irrigation with RWW on TFX persistence in the soil.

Materials and methods

Field studies

Three field studies were conducted near Kibbutz Na’an (31°53’31.34”N, 34°52’05.13”E), Israel, during 2007–2009. The field has been pivot-irrigated with secondary-treated RWW from ‘Kolchei Ayalon’ reservoir. The experiment was conducted in a field growing maize (Zea mays L.) for silage in a randomised plot design with five replicates; plot size was 2 m wide by 12 m long. Herbicides (Table 1) were applied using a motorised backpack sprayer equipped with a five Tee-Jet 11001

Table 1 Herbicides applied during field studies 2007–2009

<table>
<thead>
<tr>
<th>Year</th>
<th>Herbicide(s)</th>
<th>2007</th>
<th>2008</th>
<th>2009</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>g ha⁻¹</td>
<td></td>
<td>g ha⁻¹</td>
</tr>
<tr>
<td>2007</td>
<td>TFX</td>
<td>11.25</td>
<td>TFX</td>
<td>11.25</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2008</td>
<td>TFX</td>
<td>22.5</td>
<td>TFX</td>
<td>22.5</td>
</tr>
<tr>
<td></td>
<td>Flufenacet +</td>
<td>27 + 6.8</td>
<td>TFX</td>
<td>7.5 + 22.5</td>
</tr>
<tr>
<td></td>
<td>metribuzin</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2009</td>
<td>Pyrithiobac-sodium</td>
<td>42.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Rimsulfuron</td>
<td>5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

TFX, trifloxysulfuron-sodium.
Dose–response curves

Soil samples were collected from the non-treated plots in the RWW-irrigated field and from a fresh water (FW)-irrigated nearby field. The soils were air dried, sieved (5 mm), placed in 7 × 7 × 7-cm plastic pots and planted with sorghum. The pots were sprayed with different rates of the commercial formulation of TFX (rates are given in active ingredient throughout): 0, 1.875, 3.75, 7.5, 15 and 30 g ha\(^{-1}\) using a chain-driven laboratory sprayer, equipped with a flat-fan nozzle (8001E) calibrated to deliver 300 L ha\(^{-1}\) at 206 kPa. These rates are equivalent to 0, 0.25X, 0.5X, X, 2X and 3X, where X is the recommended rate for TFX application in cotton in Israel. Then, the sprayed pots were transferred to the nethouse. The pots were irrigated with FW as needed for 21 days, and the fresh weight of the sorghum’s shoots and roots was recorded. The data were analysed using a nonlinear curve model (exponential rise to maximum, three parameters) using SigmaPlot software (SigmaPlot, Jandel Corporation, San Rafael, CA, USA):

\[
Y = y_0 + a(1 - e^{-bx})
\]

where \(Y\) represents % inhibition of TFX-treated root/shoot biomass, \(y_0\) represents the % inhibition of UTC plants, \(a\) represents the amplitude, and \(b\) represents the rate constant.

Soil sterilisation

The dissipation rate of TFX was examined in a sterilised (autoclave × 2 for 20 min 24 h apart) and non-sterilised RWW- and FW-irrigated soils. Different rates of TFX (equivalent to 0, 7.5 (X) and 75 (10X) g ha\(^{-1}\)) were aseptically added and thoroughly mixed into the sterilised and non-sterilised soils, and their efficacy was examined using the Petri dish bioassay as described earlier.

Results

Field studies

In 2007, plots treated with TFX and pyrithiobac-sodium were heavily infested mainly with \(A.\) palmeri and 40 days after treatment (DAT) were significantly more infested than the rest of the treatments, including UTC (Fig. 1).

Season 1 (2007) Data presented in Table 3 are based on the Petri dish bioassay and indicated a rapid and significant reduction in TFX herbicidal activity in the soil. The soil activity of the applied herbicide was evaluated by measuring the sorghum root elongation. The first soil sampling collected four DAT and the following irrigation have shown that only 64% and 59%
of the applied herbicide remained active when the herbicide was applied at the recommended rate X and 2X respectively. When TFX was applied at a double rate (2X), the dissipation was slower and it took 28 days until a significant difference between 0 and 28 DAT was observed. Hence, no herbicidal activity in soil was observed 28 DAT, regardless of the applied herbicide rate.

**Season II (2008–2009)** The TFX herbicidal activity in soil was practically dissipated within 4 weeks (Table 4). Furthermore, the other sulfonylurea herbicides, namely iodosulfuron and iodosulfuron + mesosulfuron (iodo + meso), exhibited a similar trend of rapid activity dissipation. The soil activity of iodosulfuron was reduced significantly after 28 days at 2008 and 2009 (from 67% and 58% inhibition to 1% and 8% inhibition respectively). With iodosulfuron, no significant effect of soil depths was detected. The activity of the iodo + meso mixture declined only 14 DAT, from 81% to 17% inhibition (Table 4). In all three herbicide treatments presented in Table 4, there were negative results, which were possibly due to hormesis (Cedergreen, 2008).

The possibility that the dissipation of the herbicidal activity was caused by enhanced leaching of the herbicide to a deeper soil layers because of the irrigation was examined. At selected times after herbicide application during 2008 and 2009, soil was sampled from depths of 0–15 cm and from a depth of 15–30 cm and bioassayed as described earlier. The results clearly demonstrated that the herbicide was well distributed through the examined soil profile and no significant difference in TFX activity in the two soil layers was detected (Table 5).

### Dose–response curves

The sorghum shoot development in the soil with freshwater irrigation history was severely inhibited, while the development of the sorghum shoots grown in the soil with RWW irrigation history was less affected. Even in the highest TFX rate (4X), the sorghum plants that grew in the soil with RWW irrigation history survived and their shoot biomass was reduced only by 60% (Fig. 2).

### Effects of soil sterilisation

The use of the Petri dish bioassay for herbicide residual activity was further validated in the soil sterilisation experiment. Data analysis of the root length indicated differences in TFX activity in the soils with different

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**Table 3** Effect of trifloxysulfuron-sodium in the soil on sorghum root lengths. In 2007, soil samples were collected from the plots (0–15 cm depth) into Petri dishes. The Petri dishes were planted with *Sorghum bicolor*, and after 5 days of incubation, root lengths were measured. Data presented as % inhibition of UTC ± SEM. The root lengths of seedlings grown in UTC soil were 7.6, 6.3, 7.1 and 8.1 cm for 4, 14, 28 and 42 DAT respectively.

<table>
<thead>
<tr>
<th>Root length (% of inhibition)</th>
<th>Trifloxysulfuron-sodium applied (g ha⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DAT</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>11.25</td>
</tr>
<tr>
<td>14</td>
<td>64 ± 6</td>
</tr>
<tr>
<td>28</td>
<td>56 ± 8</td>
</tr>
<tr>
<td>42</td>
<td>7 ± 5</td>
</tr>
<tr>
<td></td>
<td>8 ± 5</td>
</tr>
</tbody>
</table>

**Fig. 1** Effects of herbicide application on weed infestation in the different treatments. Herbicides were applied post-emergence, and weed infestation was evaluated every 2 weeks. Infestation was estimated as percentage of plot coverage.

**Fig. 2** Trifloxysulfuron-sodium (TFX) effect on *Sorghum bicolor* plant growth (shoot and root) in pots containing soil with different irrigation history. Soils were collected from the field, planted with *S. bicolor* and sprayed with different doses of TFX. After 21 days, the plants were harvested and shoot/root fresh weight was measured. RWW, reclaimed wastewater; FW, fresh water. Vertical bars represent standard error of the mean values (n = 5, P ≤ 0.05).
irrigation history at the highest (10X) applied rate (75 g ha\(^{-1}\)). The growth of the sorghum roots was the same in all irrigation histories, regardless of the sterilisation, at 0 and 7.5 g ha\(^{-1}\) treatments. In the 10X treatment, there was a significant difference in root growth between RWW-sterilised soil and non-sterilised soil, and no significant difference between FW-sterilised and non-sterilised soil. The response of the plants that were grown in the freshwater soil to TFX did not significantly differ because of the sterilisation (from 22% to 35% inhibition), but there is a great difference between the response of the plants that grew in RWW-irrigated soil and sterilised RWW-irrigated soil (from 5% to 53% inhibition respectively) (Fig. 3).

### Discussion

The aim of this study was to evaluate the effect of long-term RWW irrigation on the fate of TFX activity in the soil. TFX is considered a residual herbicide in soils with neutral–basic pH. We demonstrated that under field conditions in soil with extended RWW irrigation period, there was very high infestation with weeds 30 days of application as compared with other herbicides. The high infestation with *A. palmeri* in the field was also because of its resistance to TFX and at lower level to pyrithiobac-sodium (Manor, unpubl. obs.); we assumed that this developed because of exposure to lower than recommended levels of TFX in the soil, because of its rapid dissipation. This exposure to lower TFX doses may also cause hormesis, resulting in higher plant biomass of the *A. palmeri*. Weed infestation (higher than the control) observed in the TFX and pyrithiobac-sodium plots was also because of the severe herbicide damage to the crop plants, causing higher light interception and thus enhancing weed growth. However, higher light interception alone does not explain the very high infestation

### Table 4

<table>
<thead>
<tr>
<th>Fresh shoot biomass (% inhibition)</th>
<th>Trifloxysulfuron-sodium</th>
<th>Iodo + Mefenpyr</th>
<th>Meso + Iodo + Mefenpyr</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2008</td>
<td>2009</td>
<td>2008</td>
</tr>
<tr>
<td>g ha(^{-1})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DAT</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>82 ± 3</td>
<td>62 ± 5</td>
<td>85 ± 3</td>
</tr>
<tr>
<td>14</td>
<td>10 ± 9</td>
<td>62 ± 6</td>
<td>29 ± 16</td>
</tr>
<tr>
<td>28</td>
<td>−37 ± 23</td>
<td>−30 ± 15</td>
<td>31 ± 19</td>
</tr>
<tr>
<td>42</td>
<td>40 ± 11</td>
<td>3 ± 18</td>
<td>29 ± 1</td>
</tr>
</tbody>
</table>

DAT, days after treatment; UTC, untreated control.

### Table 5

<table>
<thead>
<tr>
<th>Shoot biomass (% of inhibition)</th>
<th>Trifloxysulfuron-sodium applied (g ha(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0–15 cm</td>
</tr>
<tr>
<td>2008</td>
<td>22.5</td>
</tr>
<tr>
<td>2009</td>
<td></td>
</tr>
</tbody>
</table>

DAT, days after treatment; UTC, untreated control.

Fig. 3 Effect of soil sterilisation and trifloxysulfuron-sodium (TFX) doses on the activity of the herbicide in soils with different irrigation history (reclaimed wastewater and fresh water). Soils were collected from field, brought to 40% field capacity and sterilised twice within 24 h. The TFX was applied, and *Sorghum bicolor* was planted. After 5-day incubation, root lengths were measured. Vertical bars represent standard error of the mean values (\(n = 5\), \(P \leq 0.05\)).
with *A. palmeri* in the treated plots. Therefore, there has to be another element involved in this phenomenon. At slightly higher rate than the recommended (7.5 < 11.25 g ha⁻¹), a rapid dissipation of the herbicide from the soil was involved. A similar trend was observed with other sulfonylurea herbicides (Table 4), which implies that the phenomenon is much broader and is not limited to TFX. These results are different from the results reported by Minton *et al.* (2008), who found that injury to maize planted in soil treated with TFX 60 DAT was 48%. At the higher rates of TFX, there was a difference in the pattern of the dissipation between 2007 and 2008–2009, which could be explained by the differences in the irrigation during the season, as well as from the different evaluation methods (Petri dishes versus pots). The difference in the dissipation pattern between 2008 and 2009 could be due to difference in soil humidity on the day of application. In 2008 (and 2007), the soil was already wet at the time of application, while in 2009 irrigation started only 3 days after herbicide application, which probably delayed the initiation of microbial population growth and activity. The differences in irrigation might also suggest a reason for the relatively low growth inhibition of the test plants observed at 0 DAT in 2007 and 2008. The fact that there was no significant difference in the activity of TFX in two soil depths indicates that leaching is not a major element in the activity dissipation of TFX. In the dose–response curves, soil irrigation history was the only significant factor effecting TFX herbicidal activity, because the soils did not differ in other major qualities (mechanical structure and irrigation during the experiments were the same). The major difference between sterilised and non-sterilised soil is the presence/absence of microorganisms.

As there was no difference in the response of FW-sterilised and non-sterilised soil to the herbicide, we assume that the presence of microorganisms in this soil has a minimal effect on the TFX activity. However, the differential response between sterilised and non-sterilised RWW soils to TFX was significant, so we can assume that it is because of the absence of microorganisms in the sterilised soil. As we used 10 times the recommended dose as the highest rate, we expected inhibition level higher than 22% in the non-sterilised freshwater soil. This can be explained by the fact that the exposure time of the plants to the herbicide was only 5 days, and even though the roots of the plants that grew in non-sterilised freshwater soil were thinner and redder (characteristics of ALS-inhibiting herbicides), their length was not affected by the herbicide’s presence, at the lower rates examined. Adsorption of TFX to RWW- and FW-irrigated soils was examined using very high TFX rates (>>10X) and HPLC analysis, as well as ¹⁴C-TFX, which allowed us to lower the rates to 10X. In both experiments, no significant difference between the soils was detected (data not shown). Thus, we propose that in the RWW soil, the microorganisms have a significant role in TFX soil activity dissipation. Furthermore, because the experiment was conducted in Petri dishes, the leaching factor was eliminated and the assumption of microorganism’s activity became clearer.

**Conclusion**

The results presented in this article indicate that there is a strong link between long-term irrigation with RWW and fast dissipation of TFX (and other sulfonylurea herbicides) from the soil. There is also reason to believe that this dissipation is not because of leaching of the herbicides to the soil depth and involves microbial activity. As Israel is one of the only countries where irrigation with RWW has been common for more than 40 years, this research may be of importance for identifying problems that may occur following long-term usage of RWW for crop irrigation. However, more research is needed to determine the extent of this phenomenon, as well as to identify the microorganisms and the mechanism involved in the TFX dissipation.

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**References**


