Title

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Journal

Journal of Environmental Science and Health Part B, ahead-of-print(ahead-of-print)

ISSN

0360-1234

Authors

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et al.

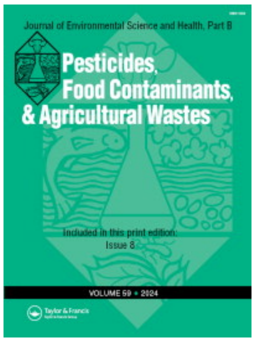
Publication Date

2024-09-20

DOI

10.1080/03601234.2024.2406123

Peer reviewed



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Journal of Environmental Science and Health, Part B
Pesticides, Food Contaminants, and Agricultural Wastes

ISSN: (Print) (Online) Journal homepage: www.tandfonline.com/journals/lesb20

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To cite this article: Matthew Fatino, Katie Martin, Franck Dayan & Bradley D. Hanson (20 Sep 2024): Adsorption of imazamox in California agricultural soils and implications for branched broomrape *(Phelipanche ramosa)* management, Journal of Environmental Science and Health, Part B, DOI: 10.1080/03601234.2024.2406123

To link to this article: https://doi.org/10.1080/03601234.2024.2406123

Published online: 20 Sep 2024.

JOURNAL OF ENVIRONMENTAL SCIENCE AND HEALTH, PART B
https://doi.org/10.1080/03601234.2024.2406123

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Adsorption of imazamox in California agricultural soils and implications for branched broomrape *(Phelipanche ramosa)* management

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ABSTRACT

Results of previous research on chemigated imazamox for control of branched broomrape (*Phelipanche ramosa*) in processing tomatoes suggested potential soil-type differences in imazamox availability. Over two years, there were differences in crop-injury between two sites less than 30-km apart: imazamox-treated tomatoes in the Davis location had relatively minor early season injury while tomatoes at the Woodland location were severely injured or killed. The following study was conducted to investigate imazamox sorption in four California soils to determine if differences in herbicide adsorption played a role in variable crop-injury observed in the field trials. To determine the sorption capacity of imazamox of each soil, a batch-equilibrium study was conducted. There were significant differences in sorbed imazamox: the clay soil had the highest adsorption (Robert's Island: 742.5 pg μL^{-1} sorbed), followed by the sandy loam soil (Ripon: 723.9 pg μL^{-1} sorbed), while the loam soils from both trial sites (Davis: 704.2 pg μL^{-1} sorbed; Woodland: 699.9 pg μL^{-1} sorbed) had the lowest adsorption and were not significantly different from one another. Results from this study illustrate only minor differences in imazamox adsorption among the soils tested which suggests that soil type was likely not a major factor contributing to differences in crop-injury.

ARTICLE HISTORY
Received 26 July 2024
Accepted 15 September 2024

KEYWORDS
Chemigation; herbicide fate; imidazolinones; processing tomatoes; California; agriculture; parasitic plants; broomrape; *Phelipanche ramosa*

Introduction

Processing tomato (*Solanum lycopersicum*) is a major cash crop grown in the central San Joaquin and Sacramento valleys of California. In 2023, it was a top 10 agricultural commodity in the state worth $1.2 billion dollars.[1] Branched broomrape (*Phelipanche ramosa*) is a parasitic noxious weed that can parasitize a wide range of agricultural crops.[2] Branched broomrape is an obligate holoparasite that parasitizes a host plant's root system, reducing plant vigor.[2] Tomatoes are highly susceptible to branched broomrape and yield loss in highly infested fields can be up to 80%.[3] Branched broomrape was first noted in California in the early 1900s.[4] After an industry driven eradication program utilizing methyl bromide fumigation that began in the 1960s, it was thought to have been eradicated from California by the 1980s.[5,6] It has reemerged in recent years, with several commercial fields having been reported since 2016.[7] Branched broomrape is currently an A-listed pest in the state requiring crop destruct and quarantine protocols which leads to massive economic losses to affected growers.[8]

Research began in 2019, to validate existing herbicide programs for broomrape management in processing tomato.[9] The programs were based on 20 years of research done by Israeli researchers for management of Egyptian broomrape (*Phelipanche aegyptiaca*) in their processing tomato systems.[10] These programs utilize acetolactate synthase (ALS) inhibitor herbicides in various combinations and application methods. There are several program regimes depending on broomrape infestation level; however, most of them utilize preplant incorporated sulfosulfuron followed by several in-season applications of imazapic applied *via* chemigation. Currently, neither sulfosulfuron or imazapic are labeled for use in tomatoes in California and there are differences in tomato production practices and primary broomrape species between California and Israel. Therefore, to validate and adapt the Israeli programs under California conditions and to generate the necessary data to support potential herbicide registration for this pest, a series of field studies were designed and implemented. In 2019 and 2020, experiments focused on evaluation of preplant incorporated sulfosulfuron and chemigated imazapic for their crop safety in California tomatoes and their efficacy for branched broomrape management.[9] After two seasons of field research, it became clear that imazapic faced insurmountable barriers to registration in California, and focus shifted from imazapic to imazamox as the chemigation component. Imazamox, a sister compound to imazapic in the imidazolinones class, already has a label in California on other crops such as alfalfa, making it a better candidate for potential registration on tomatoes in California. Beginning in 2021, field studies continued the evaluation of Israeli-based herbicide programs with imazamox as the chemigation component for crop safety on tomatoes and efficacy for branched broomrape management.[11]

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The mechanism of action of imazamox is the inhibition of acetolactate synthase, a critical enzyme in the synthesis of branched chain amino acids.[12] Imazamox uptake occurs in both leaves and roots, and it is translocated in both the xylem and phloem.[12] It was first marketed in the United States in 1997 for use in soybeans and subsequently received a reduced risk pesticide designation due to its low mammalian toxicity and its favorable environmental fate.[13] It is widely used in the United States for postemergence control of annual broadleaf and grass weeds in agricultural and aquatic systems, including imidazolinone-resistant (IR) crop systems. Currently, postemergence uses of imazamox are registered in California as Clearfield/Beyond on several IR crops, as well as Raptor in alfalfa.[14,15] Soil adsorption has been extensively studied in the imidazolinones herbicides, particularly those with preemergence soil activity such as imazethapyr, imazapyr, and imazaquin.[16–18] However, there are relatively few reports on imazamox soil adsorption, likely due to its primary use as a postemergence or aquatic herbicide. Among three imidazolinones evaluated, imazamox had the lowest soil sorption, followed by imazethapyr then imazaquin and that all three herbicides had lower sorption at pH 7 than at pH 5.[19] Soil amended with biochar did not increase the sorption of any of the herbicides tested, including imazamox.[20] The authors concluded this was likely due to the fact that biochar has a net-negative charge and all herbicides tested were anionic. Another recent publication by Hu et al.[21] also evaluated imazamox adsorption in lake sediment; however, herbicide fate could be substantially different in the aerobic and irrigated environment of an agricultural field.

After two field seasons of crop safety and efficacy studies, it became clear that chemigated imazamox had the potential to cause crop injury in tomatoes at our higher rate regimes.[11] Interestingly, tomatoes at the field site near Davis, CA, had only minor early season injury in 2021 and no injury in 2022, while tomatoes at the grower field site near Woodland, CA, were severely injured in 2021 and 2022 and did not recover by the end of the season. The discrepancy in crop injury observed in plots treated with chemigated imazamox across studies and study sites led us to investigate potential contributing factors. The differences between the two trial sites included soil type, irrigation tape depth, fertility, and irrigation practices. We hypothesized that soil type differences between the two sites may have influenced the amount of plant-available imazamox and led to differences in crop injury among trial sites. To evaluate this, batch equilibrium studies were conducted in late 2022 to determine if there were differences in imazamox soil adsorption among four California soils, including soil from the two trial sites. In addition, an experiment was conducted to determine the sorption coefficient of imazamox for each soil.

Materials and methods

Field collection

Soil was collected from four agricultural fields within the California tomato production region for batch equilibrium experiments. Collection sites included the Davis field site (38°31’48.1"N 121°47’01.1"W), a field adjacent to the Woodland field site that was not under broomrape quarantine control (38°45’29.1"N 121°46’15.0"W), a field near Ripon, CA, (37°43’03.4"N 121°12’05.0"W) to represent a sandy soil type, and a field near Roberts Island, CA (37°52’39.8"N 121°22’46.7"W) to represent a higher organic matter soil (Table 1). Soil was collected from the A-horizon in the top 7 cm, air dried, and sieved with a 2 mm screen. Field capacity and bulk density of each soil were calculated. Laboratory analyses of these soils were conducted at the Colorado State University Weed Science Lab in Fort Collins, CO.

Sorption capacity

Batch equilibrium methods were used to determine the sorption capacity of imazamox in each soil.[22] A stock solution was prepared with 0.6 µg mL^{-1} imazamox in a 0.02 M $CaCl_2$ solution. This concentration mimicked the field rate of imazamox used in chemigation applications in field trials conducted California and Chile.[11] Five grams of air-dried soil was added to a 50 mL centrifuge tube and brought to field capacity with the imazamox solution and was allowed to sit overnight at room temperature. The next day, 5 mL of 0.02 M $CaCl_2$ (aq) was added to the centrifuge tube, tubes were vortexed, then centrifuged for 10 min at 2,500 rpm (Legend X1R centrifuge, Thermo Fisher Scientific, Waltham, MA). A 2 mL aliquot of the supernatant was filtered through a 0.2 µm PVDF filter and injected into a Shimadzu 8040 LC-MS/MS system for quantification (Shimadzu Corporation, Kyoto, JP). This experiment had five 5 grams soil replications for each of the 4 soils, for a total of 20 samples.

Herbicide soil adsorption

To determine imazamox sorption coefficients for each soil, a batch equilibrium method was used.[22] Ten grams of each soil was placed in a 50 mL centrifuge tube, followed by 10 mL of 10 µg mL^{-1} imazamox and water solution.

Table 1. Soil properties of soil collected from four California processing tomato fields and used in imazamox batch equilibrium experiments.

Site	NO3-N	Olsen-P	Na	K	Ca	Mg	CEC (estimated)	OM (LOI)	pH	Sand	Silt	Clay	Soil Class
	ppm				meq 100 g^{-1}			%			%		
Davis	47.7	19.0	26	553	6.35	10.11	18.0	1.85	7.40	44	36	20	Loam
Woodland	179.3	83.2	101	296	9.55	7.91	18.7	2.13	7.20	48	33	19	Loam
Ripon	320.1	129.0	69	594	10.81	3.83	16.5	6.47	6.20	75	19	6	Sandy Loam
Robert’s Island	74.9	62.0	262	154	21.32	7.81	30.7	4.06	6.72	21	37	42	Clay

The centrifuge tubes were loaded on a reciprocal shaker and shaken for 24 h at room temperature. The tubes were centrifuged at 2500 rpm for 10 min. A 2 mL aliquot of the supernatant was filtered through a 0.2 µm PVDF filter and injected into a LC-MS/MS.[23] This experiment had five replications of each soil, for a total of 20 samples.

The adsorption coefficient (K_{d}) of each soil was calculated using Eq. (1).[22]

$$K = \frac{C_s^{ads}(eq)}{C_{aq}^{ads}(eq)} = \frac{m_s^{ads}(eq)}{m_{aq}^{ads}(eq)} \frac{V_0}{m_{soil}} (cm^3 g^{-1}) \quad (1)$$

Where:

$C_s^{ads}(eq)$ = content of the substance adsorbed on the soil at adsorption equilibrium

$C_{aq}^{ads}(eq)$ = mass concentration of the substance in the aqueous phase at adsorption equilibrium; this concentration is analytically determined taking into account the values given by the blanks.

$m_s^{ads}(eq)$ = mass of the test substance adsorbed on the soil at adsorption equilibrium

$m_{aq}^{ads}(eq)$ = mass of the test substance in the solution at adsorption equilibrium

m_{soil} = quantity of the soil phase, expressed in dry mass of soil

V_0 = initial volume of the aqueous phase in contact with the soil

K_{oc} (organic carbon-water partition coefficient) was calculated using Eq. (2).[24]

$$K_{oc} = (K_d / F_{oc}) x100 \quad (2)$$

F_{oc} (soil organic carbon mass-fraction 100 g soil^{-1}) was calculated using Eq. (3).

$$F_{oc} = SOM / 1.72 \quad (3)$$

Where 1.72 is a conversion factor to estimate organic carbon from soil organic matter.[24]

Quantification

Soil capacity and soil adsorption experiments were analyzed using a Shimadzu 8040 LC-MS/MS system. The LC was equipped with a C18 column (100 mm x 4.6 mm x 5 µm Phenomenex Corporation, Torrance, CA) heated to 40 C. A gradient mobile phase was run over an 8-min run time (Table 2). Solvent A was HPLC grade water with 0.1% formic acid and solvent B was HPLC grade acetonitrile with 0.1% formic acid. The ionization source was electrospray ionization. The flow rate was set to 0.4 mL min^{-1} and the injection volume was 1 µL. Under these conditions, imazamox retention time was 3.43 min. The mass spectrometer was run in positive mode with multiple reaction monitoring (MRM) optimized for imazamox analysis.[23]

Table 2. Quantification parameters from LC-MS/MS analysis used in imazamox soil adsorption studies.

Time (min)	% Solvent A†	% Solvent B†
0	70	30
4	10	90
6	10	90
6.1	70	30
8	70	30

MRM (m/z)	Dell Time (ms)	Q1 pre-bias (V)	Collision Energy (V)	Q-3 pre-bias (v)
306.05 > 69.1	100	−30.0	−35.0	−24.0
306.05 > 261.1	100	−14.0	−24.0	−27.0
306.05 > 86.15	100	−30.0	−31.0	−16.0

†Solvent A, 0.1% formic acid in water and solvent B, 0.1% formic acid in acetonitrile.

Soil analysis

All four soils were air dried and sieved with a 2 mm screen. Two samples of each soil were sent to the UC Davis Analytical Laboratory (Davis, CA) for analysis. Soils were analyzed for physical and chemical properties including sand/silt/clay percentages, pH, cation exchange capacity (estimated), and organic matter content (loss on ignition method) (Table 1).

Statistical analysis

Final concentrations of imazamox from each soil were analyzed with a one-way analysis of variance followed by means separations using Tukey's HSD test using the agricolae package in RStudio (R version 4.1.2).

Results and discussion

There were significant differences in imazamox availability in the Ripon and Robert's Island soils when compared to Davis and Woodland soils (Figure 1). However, there was no significant difference in imazamox sorption between the Davis and Woodland soils (Figure 1). Sorbed herbicide was calculated as the difference between the initial concentration of the imazamox solution applied (847 pg µL^{-1}) and the imazamox concentration in the extracted soil solution (847 pg µL^{-1}). The greatest amount of herbicide sorption was in soil from Robert's Island (742.5 pg µL^{-1}), followed by the Ripon soil (723.9 pg µL^{-1}). The Davis and Woodland soil had similar amounts of sorption (704.2 pg µL^{-1} and 699.9 pg µL^{-1}, respectively). The organic matter content of Davis and Woodland soils was very similar (1.85% and 2.13%, respectively) and much lower than the Ripon (6.47%) and Robert's Island (4.06%) soils (Table 1). Herbicide sorption was higher in soils with greater organic matter concentrations (Table 1). Soils from the two test sites had very similar organic matter concentrations and similar levels of imazamox adsorption

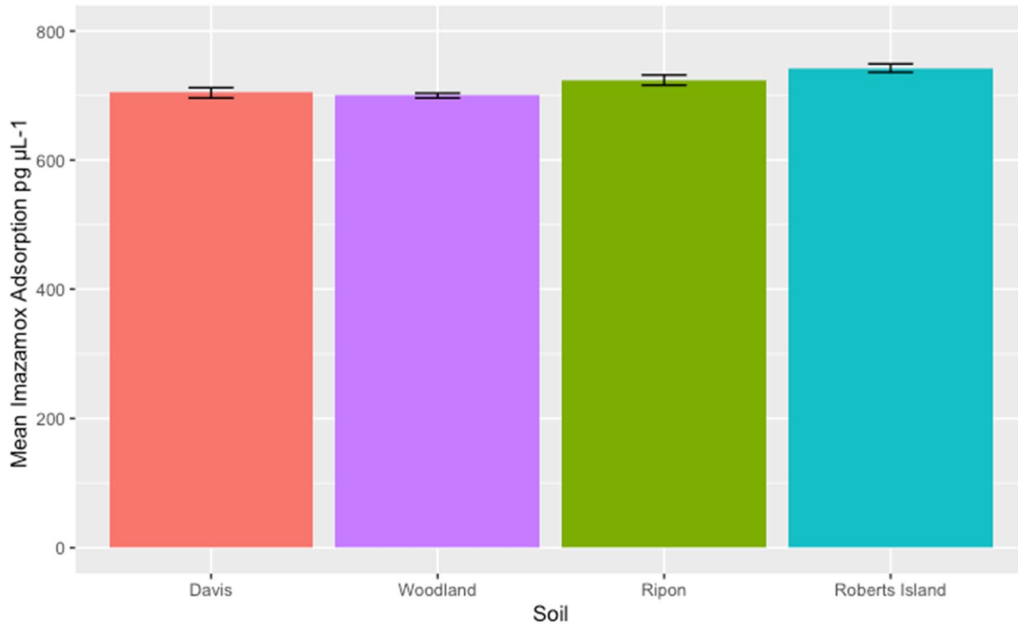

Figure 1. Sorbed imazamox in four California soils. Sorbed imazamox was calculated by the following: Initial imazamox solution concentration (847.7 pg μL^{-1}) - Final soil solution concentration. Error bars represent 95% confidence intervals.

(Table 1, Figure 1). The pH of the Ripon and Robert's Island soils (pH 6.2-6.7) was lower than Davis and Woodland (pH 7.2-7.4). However, because the pKa of imazamox is 3.3, the majority of the herbicide would be in similar ionic form in all the tested soils and sorption likely was not strongly affected by these pH differences.[19] Sorption coefficients (K_d) were higher for Robert's Island and Ripon soils, which had higher organic matter contents and higher CEC (Table 2). The Davis and Woodland site had similar K_d and K_{OC} values, likely due to their very similar soil organic matter and CEC.

These results did not support the original hypothesis that inconsistent imazamox injury between the Davis and Woodland field sites may have been related to differences in the amount of available imazamox due to soil binding characteristics. Additional factors differed between the two trial sites: the irrigation drip tape placement was between 15-25 cm at the Woodland site, below or at the low end of recommended depth for tomato production, while the Davis site was uniformly 30 cm deep.[25] The Davis field site was on a very intensively managed research farm, while the Woodland site was managed as its own 1.2-hectare subplot within a much larger 40+ hectare block with its own irrigation and fertigation system. Because of its standalone nature, the Woodland site was less intensively managed by the cooperating grower and differences in irrigation and fertilization frequency were noted. The Woodland site received less in-season fertigation applications and was irrigated more inconsistently compared to the ET_o-based irrigation schedule used at the UC Davis research farm, which has its own California Irrigation Management Information System weather monitoring site.[26] It is possible that non-uniform and shallow irrigation tape depth could have resulted in poor water and herbicide distribution uniformity; coupled with inconsistent irrigation and reduced post-chemigation line flush times chemigated herbicides may have been too shallow or too concentrated at the Woodland site.

Table 3. Imazamox adsorption coefficients from a 2022 study evaluating imazamox adsorption in four California agricultural soils.

Site	CEC (estimated) meq 100 g^{-1}	OM (LOI) %	pH	K_d 10.0 µg mL^{-1}	K_{oc} 10.0 µg mL^{-1}
Davis	18.0	1.85	7.40	0.21	19.89
Woodland	18.7	2.13	7.20	0.10	8.10
Ripon	16.5	6.47	6.20	0.31	8.17
Robert's Island	30.7	4.06	6.72	0.43	18.20

The deeper and more uniform irrigation tape depth and consistent irrigation sets and flush time at the Davis site could have led to better distribution uniformity of the chemigated herbicide, reducing the effective dose of imazamox to which tomato plants were exposed and leading to less serious injury.[27] Some or many of these factors could have led to differences in crop injury between the two sites and were not addressed with this research. Ultimately, due to the unpredictable and low margin of safety of imazamox in this use pattern, it will not be pursued as a chemigation material from branched broomrape management in California processing tomato [11] so the precise cause off differences in tomato injury may not be determined.

Conclusion

Our results fit within the limited existing literature on imazamox adsorption in both agricultural soils and sediment. Imazamox adsorption in sediment was dependent on the organic carbon content of sediment[21] and adsorption was more dependent on clay content than organic carbon content in Lithuanian agricultural soils.[28] In our results, K_d of imazamox among the four soils was as follows: Robert's Isla nd > Ripon > Woodland > Davis. Robert's Island had by far the highest CEC at 30.7 meq/100g, while Davis and Woodland had the lowest at 18.0 and 18.7 meq/100g, respectively (Tables 1, 3). These results support Sakaliene et al.[28]

findings that clay content impacted adsorption. Robert's Island and Ripon also had higher OM contents than Davis and Woodland, 4.06/6.47 and 1.85/2.13 respectively, which support Hu et al.'s[21] findings that imazamox adsorption was dependent on soil carbon content.

While there were statistical differences in imazamox sorption among the four soils tested, imazamox sorption in soils from the two experimental sites in Davis and Woodland were similar to one another. These results indicate that soil type likely was not a factor in the discrepancy in injury between the two trials. The low margin of crop safety observed in previous and ongoing field work for chemigated imazamox in processing tomato does not make imazamox a promising alternative as an in-season chemigation material.[9,11] Future research will focus on refining application protocols for a recently-approved chemigation protocol for another ALS-inhibiting herbicide, rimsulfuron, and continue to evaluate other chemistries and practices for managing and reducing the spread of branched broomrape.[29] While the initial research question remains unanswered, our results add to the limited literature available on imazamox adsorption and can help to inform management decisions regarding imazamox in agricultural soils.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

This work was supported by the California Tomato Research Institute and the University of California Henry A. Jastro-Shields Graduate Research Award.

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Data availability statement

The data that support the findings of this study are available from the corresponding author, [MF], upon reasonable request.

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