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### Permalink

<https://escholarship.org/uc/item/0vg61414>

### Journal

Agrosystems Geosciences & Environment, 7(1)

### ISSN

2639-6696

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### Publication Date

2024-03-01

### DOI

10.1002/agg2.20475

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Peer reviewed

## ORIGINAL ARTICLE

Agrosystems

# Dissipation of pendimethalin in a water-seeded rice field and implications for water management

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Assigned to Associate Editor Kurt Vollmer.

## Funding information

BASF Corporation; California Rice Research Board; UC Davis Jastro-Shields Research Award

## Abstract

Water-seeded rice (*Oryza sativa* L.) in California is produced near growing urban centers and a variety of neighboring high-value crops, which make water quality a paramount concern because of potential herbicide residue contamination in downstream surface waters. Pendimethalin is a potential herbicide for use in California water-seeded rice. A study was conducted to characterize pendimethalin's dissipation in water of a water-seeded rice field. A capsule suspension (CS), emulsifiable concentrate (EC), and granule (GR) pendimethalin were applied onto flooded rice plots at 1.1, 2.3, and 3.4 kg ai ha<sup>-1</sup> rates. Water samples were collected periodically and analyzed with an high pressure liquid chromatography tandem mass spectrometry system for residues. Pendimethalin dissipation differed across formulations. The initial sampled concentrations recorded values from 3.0 to 125.6 parts per billion (ppb). First-order dissipation resulted in half-lives for the CS from 2.3 to 3.5 days, the EC from 0.6 to 0.7 days, and the GR from 3.5 to 6.9 days. Pendimethalin use in water-seeded rice is at low risk of contaminating downstream surface waters; however, early sampled residue concentrations could be concerning. The results can assist in generating management tactics like water-holding periods to avoid potential downstream off-target effects and ensure herbicidal activity in the applied area after a pendimethalin application in a water-seeded rice field.

## 1 | INTRODUCTION

Rice (*Oryza sativa* L.) is a major crop, valued for its nutritious components as a food crop and produced worldwide (Rao et al., 2017). In the United States, rice production is centered in Arkansas, California, Louisiana, Texas, and Mississippi producing nearly 2 million t of grain for the export market in 2022 (United States Department of Agriculture (USDA), 2023). Weed management is a major challenge to achieve

economically viable production levels. Cultural practices to achieve an integrated weed management program in California rice include use of certified seed, proper land preparation, and water management (University of California Division of Agriculture and Natural Resources (UCANR), 2023). However, to reach the economically viable rice yields, herbicides are necessary to control weeds (Brim-DeForest et al., 2017; Hill et al., 2006).

The limited number of available herbicides and continuous rice cultivation year after year in California have selected for herbicide-resistant weeds and have caused a reduction in weed control from the available herbicides (Becerra-Alvarez

**Abbreviations:** CS, capsule suspension; DAT, days after treatment; EC, emulsifiable concentrate; GR, granule.

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et al., 2023; Hill et al., 2006). The lack of crop rotations makes water management and herbicide use the most important tools to manage weeds (University of California Division of Agriculture and Natural Resources (UCANR), 2023). Therefore, new herbicide modes of action are needed to help manage herbicide-resistant weed populations.

California rice is uniquely different from the other US rice-producing states because nearly 90% of the production is medium-grain rice and produced in a water-seeded system (University of California Division of Agriculture and Natural Resources (UCANR), 2023). The water-seeded production system in California is a common method to suppress weedy grasses and non-aquatic weed species. In California, pregerminated rice seed is air-seeded onto fields with a 10- to 15-cm standing flood, and the fields are typically maintained continuously flooded throughout the growing season (Hill et al., 2006).

The California rice cropping system is again unique because of its presence near growing urban communities and a variety of neighboring high-value crops. Surface water used for rice production is mainly derived from reservoirs that capture water in the Cascade Mountain Range and Sierra Nevada from the Sacramento River and the Feather River, respectively (University of California Division of Agriculture and Natural Resources (UCANR), 2023; Hill et al., 2006). Much of that reservoir water also goes toward municipal potable water and irrigation for other crops in the area. There is potential for contamination of drinking water and water for wildlife by herbicide use in California rice fields, which has historically been documented with the rice herbicides thiobencarb and molinate (Hill et al., 2006; Wagner et al., 2019). Production lands further away from the water sources will also use drainage water downstream as irrigation (Hill et al., 2006). Many neighboring crops can be susceptible to pesticide residues at low concentrations, and this can be of concern if herbicide residues are present in the irrigation water (Starner et al., 2005).

Historically, regulatory agencies and the California rice industry have collaborated to implement successful programs to manage and reduce off-target pesticide effects by mandating reports of pesticide use, monitoring water quality, and water-holding periods after chemical applications (Hill et al., 2006; Wagner et al., 2019). Pesticide use reporting and monitoring encourage stewardship of chemical use among agencies and applicators (Wagner et al., 2019). Water-holding periods prevent the pesticide active ingredient from becoming runoff in the tail water and contaminating non-target areas and organisms. The water-holding period can differ among pesticides based on their physicochemical properties and degradation pathways (University of California Division of Agriculture and Natural Resources (UCANR), 2023). Therefore, it is important to understand the behavior of herbicide active ingredients in the water-seeded system to successfully

### Core Ideas

- Herbicide use is an integral component for weed management in California water-seeded rice.
- The water quality of tailwater from a water-seeded rice field is of concern for downstream uses.
- Pendimethalin did not persist to levels of concern in the water of a water-seeded rice field.
- Water management such as water-holding periods will be important to prevent off-target contamination.

characterize them in support of sustainable stewardship and efficacious use of chemicals.

Herbicide products can be developed in various formulations to assist with weed control, for instance, to achieve longer soil residual activity, reduce crop injury, affect dissipation or for applicator safety (Hatzinikolaou et al., 2004; Daneshvari et al., 2021). Formulation is also suggested to influence the potential of the active ingredient to contaminate surface waters (Michael & Neary, 1993).

Pendimethalin is a mitotic inhibiting herbicide from the dinitroaniline chemistry; it is a selective pre-emergent that ceases seedling growth shortly after germination of susceptible plants (Appleby & Valverde, 1989). The physicochemical properties of pendimethalin are presented in Table 1. Pendimethalin has been proposed for use in water-seeded rice, since it controlled herbicide-resistant grass populations and, if labeled, would provide an additional tool for management over herbicide-resistant grasses in California rice. However, there has been no work characterizing pendimethalin's behavior in water from a water-seeded rice field. It is hypothesized, based on the physicochemical properties, that pendimethalin will not persist in surface water; however, product formulation could affect dissipation in water. Therefore, the objectives of this study were to evaluate the dissipation behavior of pendimethalin across three formulations in rice flood water after an application in a water-seeded rice field.

## 2 | MATERIALS AND METHODS

### 2.1 | Field site

A field study was carried out at the Rice Experiment Station in Biggs, CA (39°27'8.0964" N, 121°43'14.6532" W) because of scrupulous quality assurance for each experimental unit to meet regulatory standards, which led to extensive costs associated with the analysis and labor, the study was only conducted in 2021 with three replications. Individual plots

**TABLE 1** Physicochemical properties of pendimethalin.

Properties	Value
Molecular formula	C <sub>13</sub> H <sub>19</sub> N <sub>3</sub> O <sub>4</sub>
Molecular weight (g mole <sup>-1</sup> )	281.31
Water solubility (mg L <sup>-1</sup> )	0.275
Vapor pressure (Pa)	1.25 × 10 <sup>-3</sup>
pK <sub>a</sub>	Non-ionizable
Log K <sub>OC</sub>	4.11
Log K <sub>OW</sub>	5.20
Henry's Law constant (atm m <sup>3</sup> mol <sup>-1</sup> ) 25°C	2.68 × 10 <sup>-5</sup>

Source: Vighi et al. (2017) and Shaner et al. (2014).

were arranged in a randomized complete block design across the field. Soils at the site are characterized as Esquon-Neerdobe (fine, smectitic, thermic Xeric Epiaquerts and Duraquerts), silty clay, made up of 27% sand, 39% silt, and 34% clay, with a pH of 5.1, and 2.8% organic matter. Irrigation waters at the research site on average have a pH of 7.81 and electrical conductivity of 0.12 ds/m. Individual 3-m wide by 6-m long plots surrounded by 2.2-m wide shared levees were made to prevent contamination from adjacent treatments. Water temperature, when delivered from the irrigation canal, can average as low as 13°C, and in the field, it is recommended for the water to not be below 18°C for appropriate rice growth and development (University of California Division of Agriculture and Natural Resources (UCANR), 2023). Irrigation water was first delivered on June 2, 2021 into a warming field basin, where it circulated before traveling to the field basin with the plots. To move water inside each individual plot, 5-cm diameter by 1.5-cm length single bend aluminum siphon irrigation tubes (Empire Irrigation Inc.) were placed over the 2.2-m wide levees. The plots were flooded to 4 in. by June 4, 2021, and maintained at that depth for the duration of the study. “M-206” rice was air-seeded at a rate of 170 kg ha<sup>-1</sup> onto the field with a standing flood on June 5, 2021.

## 2.2 | Herbicide applications

BAS 455 48H (BASF), a capsule suspension (CS) with 0.46 kg L<sup>-1</sup> of active ingredient, BAS 455 39H (BASF), an emulsifiable concentrate (EC) with 0.40 kg L<sup>-1</sup> of active ingredient, and BAS 455 20H (BASF), a granule (GR) with 2% of active ingredient per weight, were applied onto the flooded plots at three- to four-leaf stage rice on June 15, 2021. The application rates for each treatment were 1.1, 2.3, and 3.4 kg ai ha<sup>-1</sup>. The selected rates are within the range of the appropriate field rates used in dry-seeded rice (Bond et al., 2009). The CS and EC were applied at 187 L ha<sup>-1</sup> onto the

**TABLE 2** Pendimethalin residue concentration in water after an application onto a water-seeded rice field as effected by formulation and sampling time averaged over three application rates.

Formulation	Sampling time (DAT)	Concentration (ppb)
Capsule suspension	1	10.9b
	3	6.4bcd
	5	4.3cd
	10	0.6fg
	15	0.3h
Emulsifiable concentrate	1	73.0a
	3	8.2bc
	5	3.3d
	10	0.3gh
	15	0.1i
Granule	1	4.2cd
	3	4.8cd
	5	4.8cd
	10	1.3e
	15	0.8ef

Note: Means with the same letter do not differ by Tukey's  $\alpha = 0.05$ . Data were log-transformed for analysis and back-transformed for presentation. 1 ppb = 1  $\mu\text{g L}^{-1}$ .

Abbreviation: DAT, days after treatment.

flood with a 3-m boom sprayer equipped with a CO<sub>2</sub> backpack at 206 kPa and six flat-fan 8003VS tips (TeeJet Technologies). The GR was evenly spread by hand onto the flooded plots.

## 2.3 | Sample collection

Rice flood water was sampled at 1, 3, 5, 10, and 15 days after treatment (DAT) application for each plot and replication separately. At each individual plot, a composite water sample was collected with a glass beaker from four areas in each plot near the center and quickly homogenized in an approximately 1-L plastic container (Ngim & Crosby, 2001a). Then, 3 oz was poured in a 4-oz tight-sealed jar and stored at 0°C immediately until delivered inside the lab within 4 h. For each individual plot, new containers were used to sample each time. In the lab, water samples were cleaned and 50 mL of samples were allocated from the filtered sample and stored at -20°C until analysis.

Daily temperature, relative humidity, and solar radiation data were obtained from the California Irrigation Management Information System (CIMIS), Biggs, CA, weather station number 244 (California Department of Water Resources [CDWR], 2023).

**TABLE 3** First-order dissipation kinetics and time until 50% dissipation of pendimethalin in flood water after an application on a water-seeded rice field across three formulations and three rates.

Formulation	Application rate (kg ai ha <sup>-1</sup> )	C <sub>0</sub> (ppb)	Dissipation rate (constant k day <sup>-1</sup> )	T <sub>1/2</sub> (days)
Capsule suspension	1.1	8.5 ± 1.8	0.3 ± 0.1	2.3
	2.3	22.1 ± 2.5	0.3 ± 0.1	2.3
	3.4	19.2 ± 2.3	0.2 ± 0.04	3.5
Emulsifiable concentrate	1.1	75.7 ± 15.9	1.0 ± 0.2	0.7
	2.3	353.0 ± 71.9	1.2 ± 0.2	0.6
	3.4	360.1 ± 133.8	1.0 ± 0.3	0.7
Granule	1.1	4.0 ± 0.6	0.1 ± 0.03	6.9
	2.3	6.3 ± 0.9	0.1 ± 0.03	6.9
	3.4	18.3 ± 5.1	0.2 ± 0.1	3.5

Abbreviations: C<sub>0</sub>, initial concentration; ppb, parts per billion; T<sub>1/2</sub>, time for 50% of initial concentration of the herbicide to dissipate.

## 2.4 | Residue and data analysis

Liquid-liquid extraction methods were modified from USEPA (United States Environmental Protection Agency (USEPA), 2013). High pressure liquid chromatography tandem mass spectrometry was employed to analyze for residue in water samples. A standard for pendimethalin was obtained as a reference to quantify residue in samples. The recovery in water samples was on average 79%. See supplementary material (Supporting Information 1) for details on method.

Data analysis was performed using R v4.1.2 (R Core Team, 2022). Linear regression analysis and analysis of variance were used to determine associations on the concentrations across formulations, rates, and sampling time with LMERTEST R package (Kuznetsova et al., 2017). Means separation with Tukey's honestly significant difference at  $\alpha = 0.05$  was then used where appropriate with EMMEANS R package (Lenth et al., 2018). The data were log transformed to fulfill homogeneity and linearity requirements for a linear regression (Kuznetsova et al., 2017). Furthermore, pendimethalin dissipation for each formulation at each rate was fitted to the first-order kinetic equation as follows:

$$C_t = C_0 e^{-kt}$$

where  $C_t$  is the concentration at time  $t$ ,  $C_0$  is the initial concentration,  $t$  is the time, and  $k$  is the rate constant. The NLS: NONLINEAR LEAST SQUARES R package was used to fit the data and create models, and then the NLSTOOLS R package was used to evaluate and select the most appropriate model (Baty et al., 2015). Half-lives ( $T_{1/2}$ ) were calculated from the following equation:

$$T_{1/2} = \frac{\ln 2}{k}$$

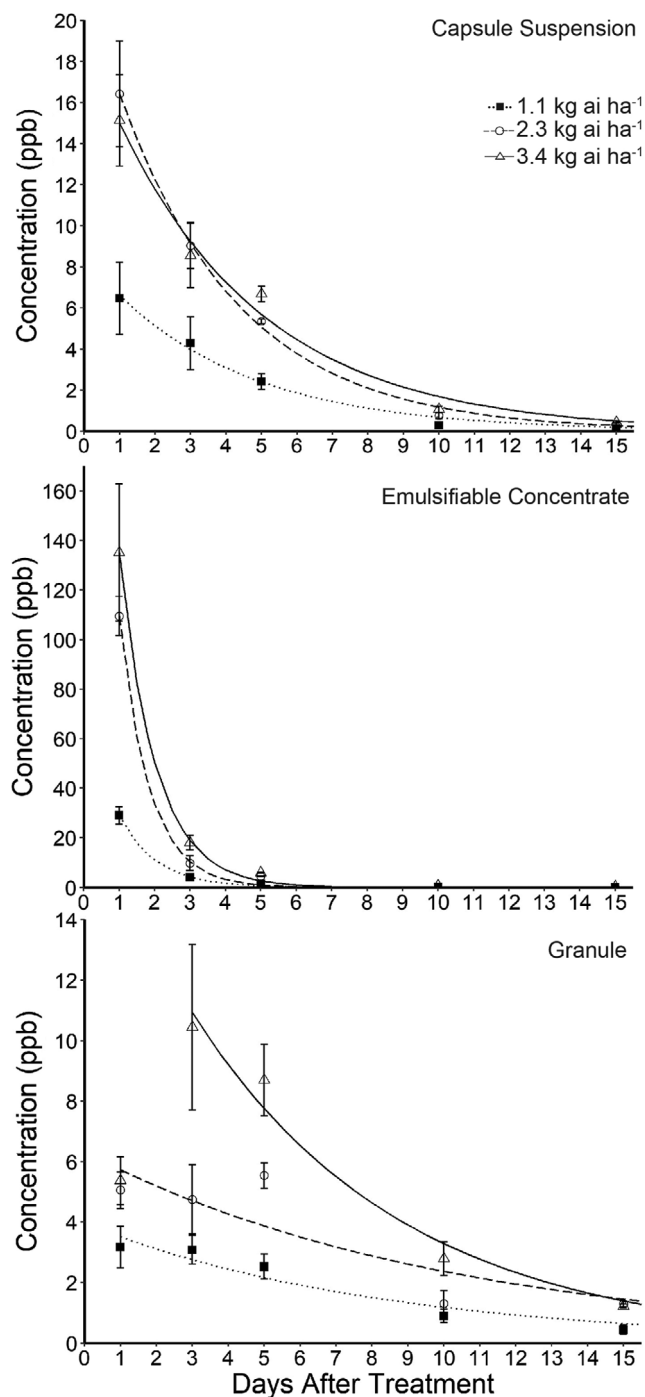
where  $T_{1/2}$  is the time for 50% of the herbicide concentration to dissipate and  $k$  is the rate constant.

## 3 | RESULTS AND DISCUSSION

There were differences in concentrations recovered from water samples across rates ( $p < 0.001$ ), sampling time ( $p < 0.001$ ), and formulation by sampling time ( $p < 0.001$ ). At 1 DAT sampling, the EC had the highest concentrations at 73.0 parts per billion (ppb) (1 ppb = 1  $\mu\text{g L}^{-1}$ ) averaged over rates (Table 2). The CS and EC formulations maintained similar concentrations throughout sampling times after the 1 DAT (Table 2). The GR maintained the greatest concentrations at 10 and 15 DAT compared to the CS and EC (Table 2).

The differences in dissipation across formulations could be attributed to the formulation properties. The EC is constructed of an oil-water emulsion with organic solvents, while the CS encapsulates the active ingredient in layers of water-soluble polymers (Rao et al., 2021). As an oil-based formulation, the EC would make pendimethalin persist in suspension on the water at higher concentrations early on because of the inactive carriers being not water soluble. The encapsulating polymers in the CS would allow the compound to be water soluble and extend the amount of time the compound is suspended in water (Rao et al., 2021). These characteristics can explain the higher concentrations early on from the EC formulation compared to the other two formulations.

GR herbicide formulations tend to have the active ingredient adsorbed to inert material, allowing slow and continuous release of the active ingredient (Hatzinikolaou et al., 2004). This characteristic of the GR formulation may help explain the increases of concentration in water 3 days after the application of the 3.4 kg ha<sup>-1</sup> rate (Figure 1). The delayed increase in concentration was rate dependent, however. Similarly, Ngim and Crosby (2001b) observed formulation affected



**FIGURE 1** Dissipation of pendimethalin in capsule suspension (top), emulsifiable concentrate (middle) and granule (bottom) in water after applications onto a flooded water-seeded rice field at 1.1, 2.3, and 3.4 kg ai ha<sup>-1</sup> rates. The first-order dissipation equation  $C_t = C_0 e^{-kt}$  was fit to the data, where  $C_t$  is the concentration at time  $t$ ,  $C_0$  is the initial concentration,  $t$  is the time, and  $k$  is the rate constant. 1 parts per billion (ppb) = 1  $\mu\text{g L}^{-1}$ . Error bars are standard error.

dissipation of the insecticide fipronil in water-seeded rice, with the GR formulation being most persistent. A GR pendimethalin application onto a water-seeded rice field may need a longer water-holding period than the liquid formulations.

Dissipation generally followed first-order kinetics (Figure 1). The GR demonstrated half-lives up to 6.9 days. The CS had half-lives 3–4 days less than GR and the EC had half-lives nearly 7 days less (Table 3). The average daily temperature for the duration of the study was 25°C, with a low of 16°C and a high of 34°C. Daily solar radiation averaged 346 W m<sup>-2</sup>, with a low of 341 W m<sup>-2</sup> and a high of 366 W m<sup>-2</sup>. Relative humidity averaged at 50%, with a low of 30% and a high of 80%. These are the typical conditions during the early rice growing season in California and are important to note as factors that can affect the pendimethalin degradation.

Half-lives of pendimethalin in water were reduced in this study, probably due to greater degradation occurring in a field environment stimulated by microorganisms, photolysis degradation, and partitioning onto organic sediments from the soil (Vighi et al., 2017). Pendimethalin residue half-lives in water have been previously reported at 12.7 and 13.7 days after an application of an EC pendimethalin formulation at 0.5 parts per million (ppm) and 1.0 ppm (1 ppm = 1 mg L<sup>-1</sup>), respectively, onto irrigation canal water (Chopra et al., 2015).

Degradation pathways can be inferred based on the physicochemical properties of pendimethalin. The pendimethalin molecule is not highly water soluble, non-ionizable, and not hydrolyzed in water and possesses a high affinity for organic matter (Table 1); therefore, sediment partitioning is most likely the significant degradation pathway. Partitioning of pendimethalin onto sediment in water/sediment investigations in dark demonstrated to be within 0.4–1.6 days for 50% allocation onto sediments (Vighi et al., 2017). Pendimethalin is moderately volatile, and volatilization is an important dissipation pathway in dry and moist soil; however, as soil moisture increases over soil field capacity, volatilization decreases due to lower movement of the vapor phase in wetter soils (Barrett & Lavy, 1983; Weber, 1990). Solar radiation was high in the study area and can be a significant degradation pathway. Both photolysis and sediment partitioning are most likely the important pathways of pendimethalin degradation. While this study negates the pendimethalin metabolites, it is important to note that there are three metabolites that can form in water (United States Environmental Protection Agency (USEPA), 2013). Nevertheless, the pendimethalin residues in the water indicate the importance of holding flood water in the field after an application to allow the herbicide molecule to settle on the soil surface when applied to a flooded rice field.

## 4 | PRACTICAL IMPLICATIONS

The US EPA has recorded an observed maximum level of pendimethalin in surface water at 17.6 ppb, probably contaminated by spray drift, and expressed the risk of pendimethalin contaminating surface waters to be less than 2% (United States Environmental Protection Agency (USEPA), 1997). While there is no water quality criteria level for pendimethalin, residues of pendimethalin have been observed in surface water tributaries near agricultural regions with concentrations up to 0.02 ppb (Lehotoy et al., 1998; Zimmerman et al., 2000). Additionally, pendimethalin residues as low as 30.0 ppb in soil have shown to cause injury to tomato (*Solanum lycopersicum* L.), a common crop grown near California rice fields (Angeles et al., 2020). Despite observed concentrations above these levels from the EC and CS formulations early on, pendimethalin dissipated quickly below levels of concern (Figure 1).

Apart from preventing potential herbicide runoff, water-holding periods can be useful for increasing herbicide efficacy. Some pesticides currently used need water for activation or to evenly distribute in the field and holding water in the field is common practice for California growers when using GR pesticides in rice (University of California Division of Agriculture and Natural Resources (UCANR), 2023; Ngim & Crosby, 2001b; Ngim & Crosby, 2001a). The concentrations observed from this study also suggest pendimethalin could benefit from a water-holding period to increase the efficacy when applied to the flood. However, an increase in efficacy can also develop greater rice crop injury and should be balanced through application rates and timings. The rates used in this study were the typical use rates in dry-seeded rice, which are known to provide adequate weed control. This study did not focus on weed control, but ongoing work is examining this aspect to enable efficacious and safe use of pendimethalin for water-seeded rice.

Pendimethalin did not persist to levels of concern in the surface water of a water-seeded rice field and was detected at very low concentrations, in general. The results from this study can assist regulatory agencies and registrants in articulating a water-holding period for pendimethalin in water-seeded rice, which can help prevent potential contamination of municipal drinking waters, prevent damage to downstream high-value crops, and ensure efficacious use, therefore promoting responsible stewardship of chemical use in California rice.

### AUTHOR CONTRIBUTIONS

**Aaron Becerra-Alvarez:** Conceptualization; data curation; formal analysis; funding acquisition; investigation; methodology; visualization; writing—original draft; writing—review and editing. **Kassim Al-Khatib:** Conceptualization; data

curation; formal analysis; funding acquisition; investigation; methodology; project administration; resources; software; supervision; validation; visualization; writing—original draft; writing—review and editing.

### ACKNOWLEDGMENTS

The authors acknowledge partial funding by the California Rice Research Board, BASF, and UC Davis Jastro Research Award. The authors also acknowledge the California Rice Experiment Station for providing the site and assisting with field management, the Multi-User Analytical Lab in Clemson University which performed the analytical analysis and Ekrem Uludag who assisted with the water sampling in the field. The authors also acknowledge the D. Marlin Brandon Rice Research Fellowship by the California Rice Research Trust, and the Department of Plant Sciences, UC Davis for the award of a GSR scholarship funded by endowments, particularly the James Monroe McDonald Endowment, administered by UCANR, which supported the student during the time of this study.

### CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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## SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

**How to cite this article:** Becerra-Alvarez, A., & Al-Khatib, K. (2024). Dissipation of pendimethalin in a water-seeded rice field and implications for water management. *Agrosystems, Geosciences & Environment*, 7, e20475. <https://doi.org/10.1002/agg2.20475>