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Surface Irrigation Reduces the Emission of Volatile 1,3-Dichloropropene from Agricultural Soils

D. J. ASHWORTH* AND S. R. YATES

USDA/ARS, United States Salinity Laboratory, 450 West Big Springs Road, Riverside, California, 92507

Low-cost, practicable techniques are required to limit the release of volatile organic compound-containing fumigants such as 1,3-D to the atmosphere. In this study, we aimed to quantify 1,3-D diffusion and emission from laboratory soil columns maintained under realistic conditions and thereby assess the efficacy of soil irrigation as a technique for reducing emissions. In two soils (one relatively high, and one relatively low, in organic matter), irrigation led to a limiting of upward diffusion of the fumigant and to the maintenance of higher soil gas concentrations. Therefore, rather than being emitted from the column, the 1,3-D was maintained in the soil where it was ultimately degraded. As a consequence, emission of 1,3-D from the irrigated columns was around half of that from the nonirrigated columns. It is concluded that surface irrigation represents an effective, low-cost, and readily practicable approach to lessening the environmental impact of 1,3-D fumigant use. In addition, the higher organic matter soil exhibited emissions of around one-fifth of the lower organic matter soil in both irrigated and nonirrigated treatments, due to markedly enhanced degradation of the fumigant. Organic matter amendment of soils may, therefore, also represent an extremely effective, relatively low-cost approach to reducing 1,3-D emissions.

Introduction

In the United States, the use of 1,3-dichloropropene (1,3-D) as a preplant fumigant for the control of soil pests in the cultivation of high-cash crops is increasing in response to the recent (2005) ban on the use of methyl bromide. 1,3-Dichloropropene comprises two isomers, cis and trans, and is a liquid at low temperatures. However, it is readily converted to a gas once injected into warm soil. Henry's constants (at 25 °C) for the cis and trans isomers are 0.074 and 0.043, respectively. Vapor pressures (at 25 °C) are 4.5 and 3.1 KPa, respectively. The relatively high vapor pressure of 1,3-D gas ensures a high degree of diffusion within the soil and, hence, a desirably extensive pesticidal effect. However, this property of 1,3-D also facilitates its potential transfer from the soil into the air above the soil surface and potentially poses a toxicological inhalation risk to bystanders and agricultural workers. In addition, since the constituent volatile organic compounds of fumigants such as 1,3-D are thought to contribute near-surface ozone formation (smog), measures are required to limit soil−air transfer and, hence, limit the environmental impact of these emissions. Ideally, such measures should be both low-cost and readily practicable.

Recent research has shown the potential of a number of soil treatments to reduce emissions of 1,3-D from soil. For example, it has been shown that a thiourea reactive barrier at the soil surface rapidly transformed 1,3-D to nonvolatile products and reduced cumulative emissions of 1,3-D by more than 80% when compared to a bare soil surface (*1*, *2*). Similarly, surface application of thiosulfate fertilizers has reduced emissions of 1,3-D by dramatically enhancing chemical degradation of the fumigant (*3*, *4*). Enhanced degradation also explained reduced 1,3-D emissions from soils amended with a variety of organic materials (e.g., composted animal manures) (*5*−*7*). It has been further shown (*8*) that yard waste compost reduced soil diffusion, and retarded the emission, of 1,3-D. In addition to enhancing chemical (*6*) and biological (*6*, *9*) degradation, organic matter (particularly the fulvic acid fraction) is also capable of sorbing 1,3-D residues (*10*); a process that may further serve to reduce emissions to air. The use of plastic films over the soil surface reduces emissions by acting as a physical barrier. For example, a virtually impermeable film retained 1,3-D in the soil pore space and hence better retarded 1,3-D emissions than polyethylene film cover or a bare soil surface (*8*, *11*). Similar findings have been reported by other workers (*12*, *13*).

Clearly, these various methods have the potential to reduce 1,3-D emissions from soil to air. However, in many cases, these methods are not as low-cost, nor as readily practicable, as the use of surface irrigation waters. The formation of a "water seal" potentially limits the emission of gases by slowing their rate of diffusion to the soil−atmosphere interface; the rate of diffusion of a gas through water being $\sim 10^4$ times slower than through air (*14*). Thus, it has been also observed (*8*, *15*) that as soil water content of field-based "microplots" increased, slower subsurface dispersion and a longer soil residence time were observed for 1,3-D. As a consequence, lower 1,3-D emissions from soil columns having received surface irrigation following fumigation treatment have been observed (*16*). In concurrence, it has been noted (*17*) that, as the surface of soil columns dried out, the emission of 1,3-D from soil to air increased. However, other workers have noted that the reduction in emissions associated with irrigation treatment can be low (*13*).

In order to further develop and assess numerical models for regulatory purposes, good quality experimental data pertaining to the soil emission of 1,3-D are required. Ideally, such data would be generated using field experiments, but these are expensive, time-consuming, and subject to a large number of variables, meaning that laboratory studies are often favored. It is therefore critical that laboratory-based studies are established in a way that represents field conditions. This is rarely achieved; for example, variations in soil temperature, organic matter content, bulk density, moisture content, and depth of the soil profile are usually not all adequately represented in a single study. Here, we attempt to address these issues and thereby quantify soil diffusion and soil−air emission of 1,3-D in laboratory-based soil columns but under field-representative conditions. This approach is used to test the hypothesis that application of a water seal is an effective method for significantly reducing the release of 1,3-D gas from the soil to the atmosphere.

Experimental Section

Soil Column Experiment. The general nature of the soil column experiments has been described previously (*18*). However, several departures from this previous approach were made and are thus detailed here. The length of the stainless steel columns was extended from 70 to 150 cm to allow for a more adequate representation of a field soil profile. Soil gas sampling ports were placed at 10 cm intervals over

* Corresponding author e-mail: dashworth@ussl.ars.usda.gov.

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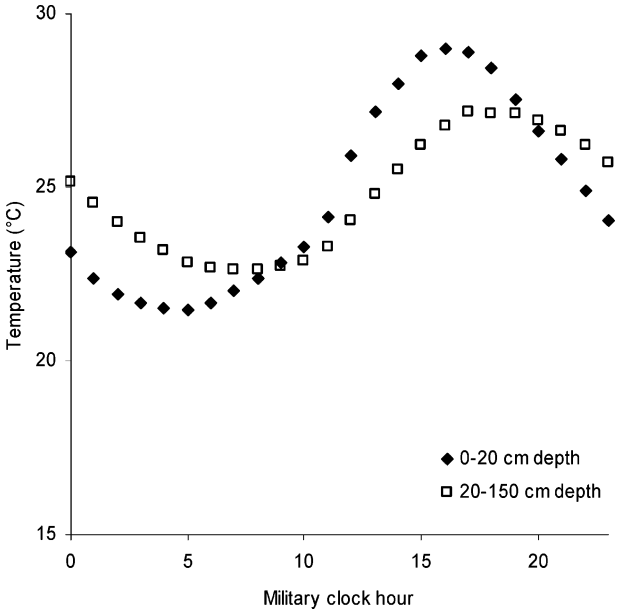

FIGURE 1. Twenty-four hour temperature regime for the soil columns.

the entire column length. Two soils were used in the study. Both were a sandy loam (about 60% sand, 30% silt, and 10% clay) of pH 7.8 collected from the same farm site near Buttonwillow, CA (thermic Typic Haplargids; Milham series). Both soils were collected to a depth of 60 cm, and each soil was mixed thoroughly, but not sieved, before use. The primary difference between the two soils was organic matter content because one of the soils had previously been amended with composted green and municipal waste material in the field. Therefore, the relative terms lower organic matter (LOM) and higher organic matter (HOM) are used to distinguish the soils that had organic matter contents of 2.09 and 3.16%, respectively. Although a 1% increase in organic matter content does not appear large, this nevertheless represents a 50% increase in the total mass of organic matter in the HOM soil.

Six columns of each soil were prepared. The soil bulk densities and moisture contents used throughout the soil column profile were the same for each set of columns and were based on values determined for the soil profiles in the field. For the bulk density, a value of 1.0 g cm^{-3} was used from 0 to 15 cm depth, 1.2 g cm^{-3} from 15 to 25 cm depth, and 1.5 g cm^{-3} from 25 to 150 cm depth. For the moisture content, a value of 5% was used from 0 to 2 cm depth, 12% from 2 to 4 cm depth, 17% from 4 to 25 cm depth, and 12% from 25 to 150 cm depth. The top of each packed column was capped with stainless steel emission chamber allowing the headspace air to be channeled through charcoal filters (ORBO 32, Supelco, Bellefonte, PA) via Teflon tubing. The columns were housed in a controlled temperature room and placed on a wooden stand to dampen vibrations, which may affect diffusion of the 1,3-D gas. It was aimed to maintain soil temperatures close to hourly averages of those observed through early September 2005 at the site from which the soils were collected. Generally, in the field, soil temperatures ranged from around 19 to 32 °C in the upper 20 cm of soil (due to diurnal variation) and were consistently around 25 °C below this. However, it was not possible to precisely simulate these throughout the entire soil profile despite insulating the columns with 1 cm thick foam below 20 cm depth. The 24 h soil temperature regime that most closely matched the field observations is shown in Figure 1 and was that used in the column study (repeated each day of the study).

In order to simulate a shank injection of 1,3-D, the columns were injected with 140 μL (equivalent to 124 L ha^{-1}; a typical agricultural application rate) of Telone II (Dow AgroSciences, Indianapolis, IN) through an injection port at 46 cm depth. Telone II is a commercial mixture of *cis*- and *trans*-1,3-D (53.25 and 42.65%, respectively; total purity 95.9%). Injection was carried out at 13:00 h on day 1 of the experiment. At 16:00 h on day 1, the soil surfaces of three columns of each soil were irrigated with 1 cm depth (113 mL) of deionized water by temporarily removing a plug in the emission chamber and inserting a "pronged" irrigation device attached to a syringe. Thus, irrigation water was slowly and evenly spread over the soil surface. The added water infiltrated the soil rapidly and did not cause "ponding" on the soil surface. Irrigation was repeated at 10:00 h on days 2−5. At 17:00 h on day 1, and at 11:00 h on days 2−5, 8, 11, and 14 or 15, soil gas sampling was carried out by removing the cap on each sampling port and slowly removing 0.5 mL of soil gas using a gastight syringe. This was immediately dispensed into a 10 mL glass vial and capped with a Teflon-faced rubber septum and aluminum crimp seal. Samples were stored at −70 °C prior to analysis.

Immediately after fumigant injection, the pulling of headspace air through the charcoal filters was initiated. Typically, each charcoal filter was used for 4 h, although this time period was increased toward the end of the experiment when emission of 1,3-D was greatly reduced. An automated, 21X data logger-controlled (Campbell Scientific, Inc, Logan, UT) system of solenoid valves allowed for four successive filters to be used without human intervention (e.g., overnight). The flow rate across the soil surface was maintained at an average of 140 (standard deviation 5) mL min^{-1}. Used filters were stored at −19 °C prior to extraction and analysis. Extraction was carried out by removing the charcoal material into a glass vial, adding 3 mL of acetone, shaking for 30 min, and then taking 1.5 mL of supernatant solution for analysis. Separate experiments showed that the recovery of *cis*-1,3-D using this method of extraction was 86%.

Degradation Experiment. Within the soil columns, sorption onto soil solids was assumed to play only a very minor role in controlling 1,3-D fate, due to its high volatility (*19*). The dominant processes were therefore assumed to be emission and degradation. Since the columns do not allow quantification of the latter of these two processes, a separate experiment was carried out in which the time-dependent kinetics of 1,3-D degradation were studied in both soils used in the column experiment. Into 21 mL glass vials, 10.5 g of moist (5% gravimetric moisture content) soil was weighed. After addition of 193.5 μg of 1,3-D (equivalent to 19.35 mg kg^{-1} of dry soil) the vials were capped with a Teflon-faced rubber septum and aluminum crimp seal. The vials were then placed at a constant 25 °C. At 0, 2, 5, 10, 24, 48, 96, and 168 h, duplicate vials were placed at −19 °C to prevent any further 1,3-D degradation from taking place. At the end of the study, the frozen vials were decapped and 10 g of anhydrous sodium sulfate and 10 mL of ethyl acetate were quickly added before immediate recapping. The vials were then shaken for 1 h before 1.5 mL of supernatant solution was removed for analysis. The degradation rate constant and, hence, 1,3-D half-life in each soil were determined by fitting first-order kinetic theory to the data.

Analysis. The *cis*- and *trans*-1,3-D concentrations of the charcoal filter (soil column experiment) and soil (degradation experiment) extracts were determined using a Hewlett-Packard HP6890 gas chromatograph equipped with a microelectron capture detector. The column was a 30.0 m × 0.25 mm × 1.4 μm capillary column (Agilent Technologies) running at a flow rate of 1.6 mL min^{-1} and using He as the carrier gas. The oven temperature was fixed at 90 °C. The inlet temperature was 240 °C, and the detector temperature 280 °C. Five 1,3-D standards encompassing the range of concentrations observed in the samples were prepared in

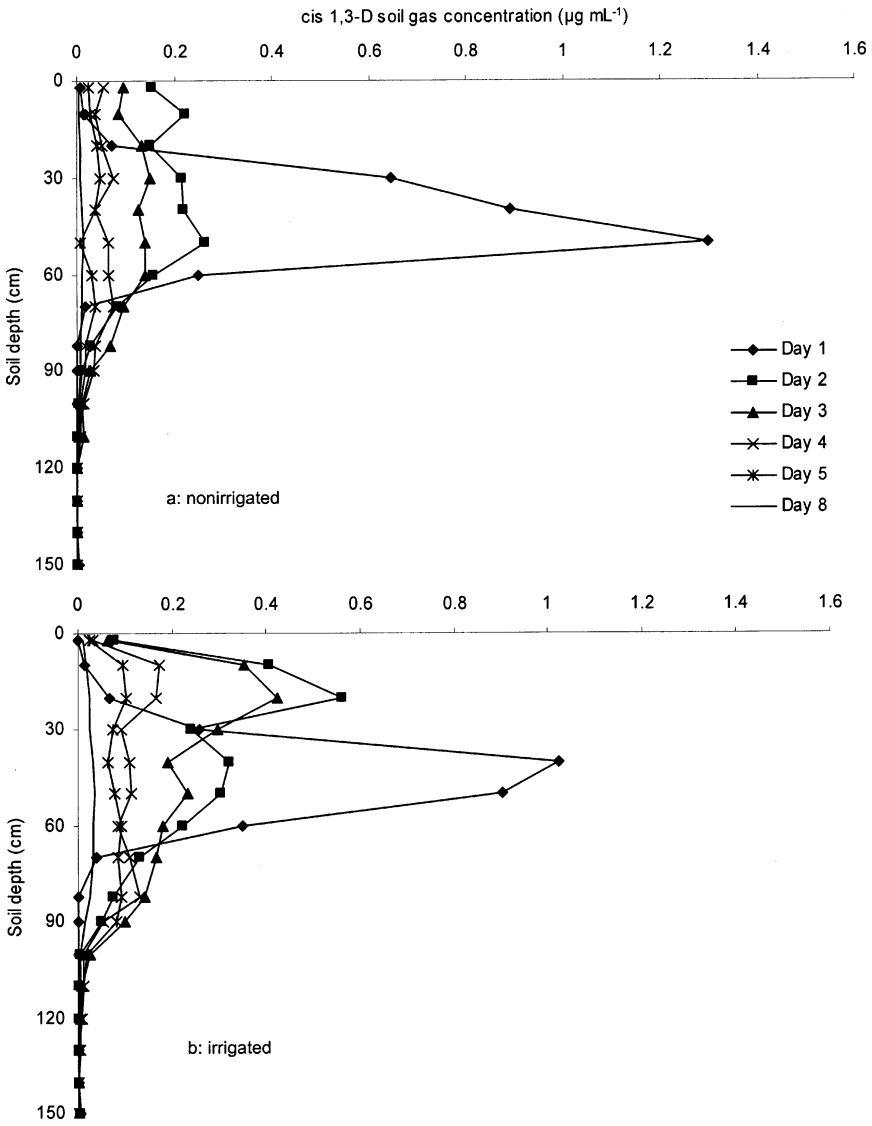

FIGURE 2. Mean ($n = 3$) *cis*-1,3-D soil gas concentrations with depth and over time for the LOM soil: (a) nonirrigated treatment; (b) irrigated treatment.

the relevant solvent. For the soil gas samples, the GC system described above was used in conjunction with a G1888 Network Headspace Sampler (Agilent Techologies). The GC conditions were as described above except that the flow rate was 1.5 mL min^{-1} and the oven temperature was 80 °C. The operating conditions for the headspace sampler were as follows: oven temperature 80 °C, loop temperature 90 °C, transfer line temperature 100 °C, vial equilibration time 5 min, and sample loop volume 0.2 mL. Five 1,3-D standards encompassing the range of concentrations observed in the soil gas samples were prepared in hexane. Chromatogram analysis was performed using Chemstation Rev.A.10.02 (Agilent Technologies).

Results

Soil Column Experiment. For both the soil gas sampling and the surface emissions, the cis and trans isomers exhibited time-course trends that were very similar to one another, although the trans concentrations were consistently lower. Only data for the cis isomer are presented here.

Mean soil gas *cis*-1,3-D concentrations over the course of the experiment are shown in Figures 2 and 3 for the LOM and HOM soils, respectively. Across both soils and treatments, the peak of 1,3-D concentration (initially around the point of injection) greatly declined between days 1 and 2 of the experiment. Diffusion of the gas through the soil was rapid and even on day 1 (4 h after injection) the gas was detected up to ~25 cm either side of the injection point. Maximum depth of gas penetration was generally reached on day 3 and occurred at depths of 90−110 cm. Very low (commonly nondetectable) concentrations of 1,3-D were consistently observed throughout the soil profiles by day 11 of the experiment.

The effect of irrigation on the soil gas concentrations was evident in both soils from day 1, with the irrigation treatment having a reduced peak 1,3-D concentration at this time. Thereafter, concentrations did not decline as rapidly in the irrigated treatment as in the nonirrigated treatment. In the HOM soil, the most noticeable effect of the irrigation occurred on day 2 when the peak of 1,3-D concentration was about twice that of the nonirrigated treatment. After this time, concentrations were similar in both treatments. In the LOM soil, further differences due to irrigation were observed. Here, on days 2 and 3, maximum 1,3-D concentrations occurred between 10 and 20 cm depth. Over the course of the experiment, soil gas 1,3-D concentrations were greater in the irrigated LOM soil than in the nonirrigated treatment. In addition to the effect of irrigation, the soil appeared to also affect soil gas concentrations after day 1, the HOM soil generally giving lower values than the LOM soil. Overall, it

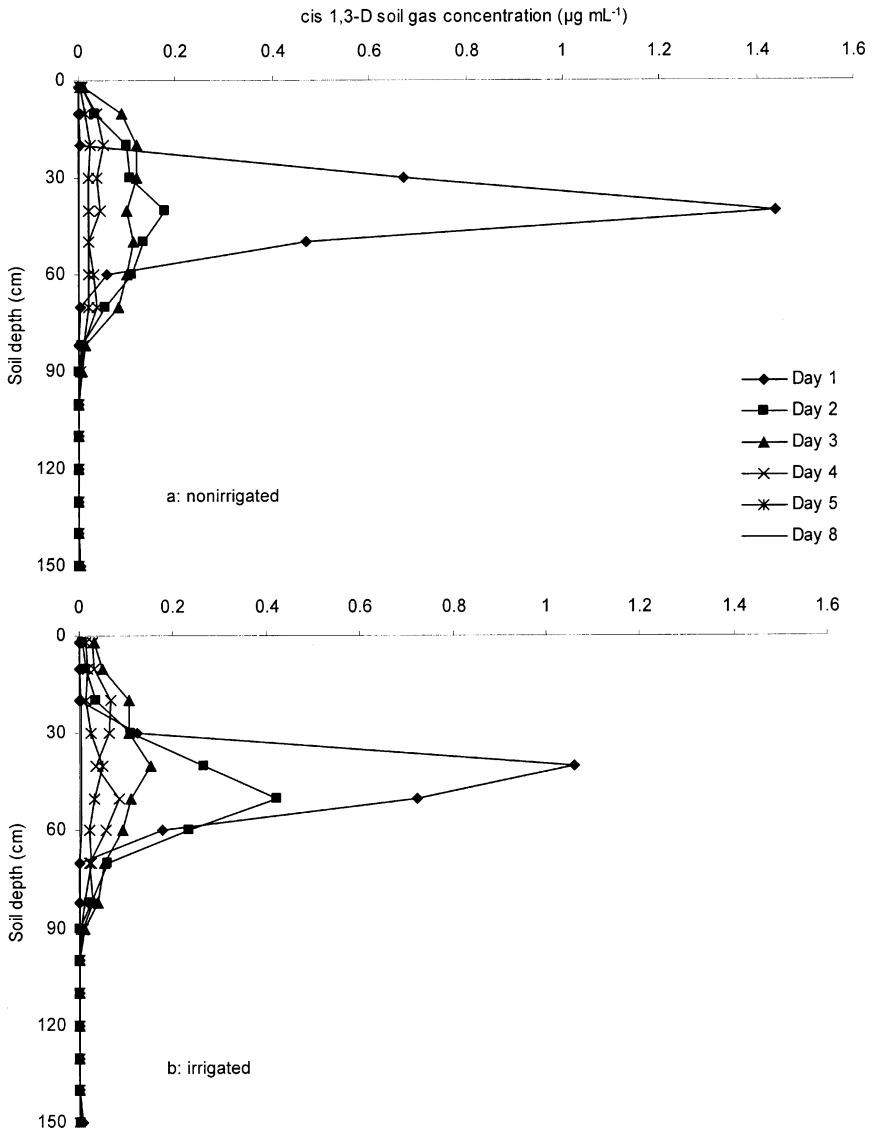

FIGURE 3. Mean ($n = 3$) *cis*-1,3-D soil gas concentrations with depth and over time for the HOM soil: (a) nonirrigated treatment; (b) irrigated treatment.

is clear that of the four treatment/soil combinations, irrigation of the LOM soil led to the greatest, and nonirrigation of the HOM soil the lowest, retention of 1,3-D gas within the soil.

Emission fluxes of *cis*-1,3-D over the course of the experiment for the LOM and HOM soils are shown in Figure 4. The principal peak in emissions occurred at ~25 h in the LOM soil and at ~50 h in the HOM soil. This peak was ~6 times greater in the LOM soil than in the HOM soil, for the same treatment. Several smaller peaks were consistently observed at subsequent times. Detectable emissions in both soils occurred over approximately the first 150 h of the experiment. In both soils during this time, irrigation markedly reduced flux rates. The total emissions (percentage of that amount added) of *cis*-1,3-D over the entire experiment were, in the LOM soil, 33.1 and 17.1% for the nonirrigated and irrigated treatments, respectively and, in the HOM soil, 5.7 and 2.7% for the nonirrigated and irrigated treatments, respectively. These values indicate that, for each soil, irrigation approximately halved the total emissions. Comparing soils, emissions from the HOM soil were around one-fifth of the emissions from the LOM soil for each treatment.

Degradation Experiment. Results from the degradation experiment (not shown) indicated that, compared to the LOM soil, much more rapid degradation occurred in the HOM soil, with almost all of the added 1,3-D in this soil becoming degraded over the week-long experiment. The fitting of first-order kinetics to the degradation curves produced half-lives ($t_{1/2}$) of 5.3 and 1.2 days for the LOM and HOM soils, respectively.

Discussion

In this study, a significant departure from previous fumigant soil column experiments was the use of long, 1.5 m, columns. This potentially allowed for a more realistic approximation of a field soil profile since there was less chance of the fumigant hitting the base of the column, rebounding, and, thus, compromising the accuracy of subsequent soil gas concentration measurements. The penetration of the 1,3-D to a maximum depth of ~110 cm suggests that a column of at least this length is required to entirely mitigate such an effect. This should be borne in mind in future experiments although, given the low concentrations occurring at such depth, the effect may only be small. Quantification of this effect, by comparing fumigant behavior in tall (1.5 m) and short (0.7 m) columns, represents a future research objective.

Within the closed column system, only two processes were assumed to control the fate of the fumigant, i.e., emission from the soil surface and degradation (chemical and biological) within the soil. Clearly, emission from the soil surface

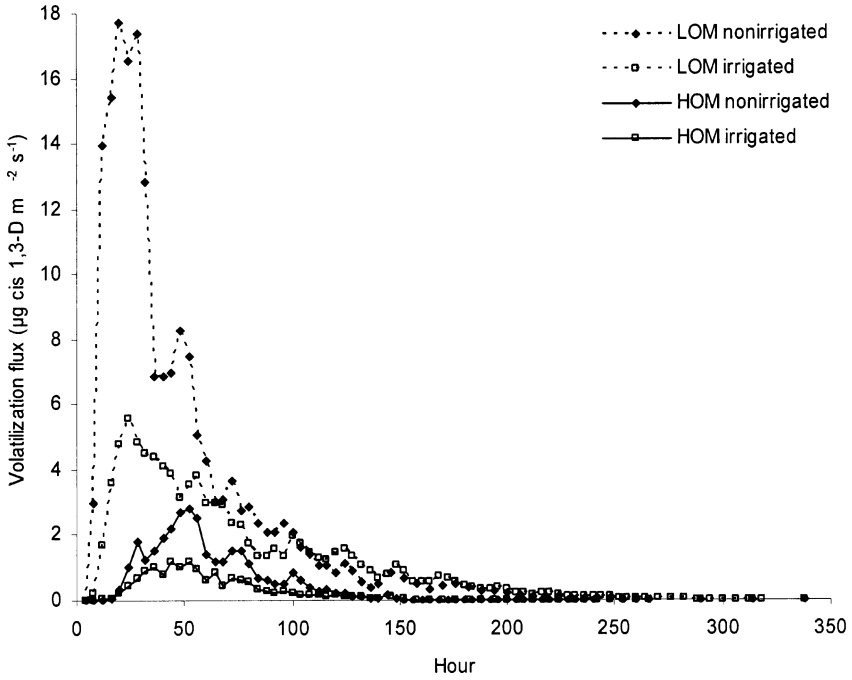

FIGURE 4. Mean ($n = 3$) *cis*-1,3-D volatilization fluxes for each soil treatment over the course of the experiment.

is dependent upon diffusion of the gas through the soil from the point of injection. The physical rate of this diffusion is controlled primarily by the bulk density and moisture content of the soil, since these variables control the amount of pore space available for gas transport. Here, effects of these variables can be most easily determined by considering the nonirrigated treatment (since these columns were not further complicated by the effect of added irrigation water). In these columns, diffusion of the 1,3-D occurred more readily above the point of injection than below, presumably as a result of the lower bulk density (and hence greater pore space) of the soil toward the soil surface. The enhanced diffusion of fumigant gas at low bulk density has been reported previously (*20*). In our study, this effect seemed to outweigh any potential retardation of gas diffusion resulting from the region of increased moisture content from 4 to 20 cm soil depth.

Nevertheless, increasing the moisture content of the surface soil by irrigation treatment did have a number of effects on the behavior of the 1,3-D gas within the soil. One such potential effect is for the 1,3-D to partition into the added water, thus reducing the soil gas concentrations. This may explain the lower soil gas concentrations observed in the irrigated treatments of both soils at day 1. Once further diffusion of the 1,3-D gas had taken place, the irrigation, particularly in the LOM soil, led to greater soil gas concentrations compared to the nonirrigated treatment. This is likely due to the limiting of upward gas diffusion by the added surface water; as evidenced by the accumulation of 1,3-D in the 10−20 cm layer of soil, i.e., emission loss, and a consequential reduction in soil gas levels, was prevented.

At day 1, little difference in the soil gas concentrations between the LOM and HOM soils was observed. Beyond this time, the HOM soil generally showed lower soil gas concentrations than the LOM soil, and on each subsequent day, the total amount of 1,3-D in the HOM soil compartment was markedly lower than in the LOM soil, for the same treatment. This was probably due to enhanced 1,3-D degradation in the HOM soil over time. Degradation occurs via biological and chemical processes and is accelerated in the presence of soil organic matter (*5*, *6*). Specifically, the addition of organic materials to soil increases specific microbial populations capable of accelerating the biological degradation of 1,3-D (*9*). Little is known about the specific processes governing the chemical degradation of 1,3-D in soil. In the present experiment, the greater level of organic matter in the HOM soil (albeit small in terms of the total percentage) was apparently sufficient to induce significantly enhanced degradation. This is further borne out by the results of the degradation study, where, at a constant temperature (25 °C) and moisture content (5%), degradation of *cis*-1,3-D occurred more than four times faster in the HOM soil than in the LOM soil. While differences in degradation rate between the HOM and LOM soils in the column study may not have been identical (due to differences in temperature and soil moisture content between the two experiments), the degradation study data provide strong evidence for the role of organic matter in increasing the degradation of 1,3-D and, hence, a clear explanation for the lower soil gas 1,3-D concentrations observed in the HOM soil columns. In addition, values obtained for the half-life of 1,3-D in the LOM and HOM soils compare relatively well to those reported elsewhere (*6*). These workers found *cis*-1,3-D half-lives for a sandy loam soil of 6.3 (unamended) and 1.8 days (amended with composted steer manure (5% w/w)) at 20 °C.

The enhanced degradation of the 1,3-D in the HOM soil led, in both treatments, to smaller amounts of the fumigant being available for emission from the soil surface. Hence, dramatically reduced flux rates were observed in the HOM soil treatments, and the total amount of 1,3-D emission from the HOM soils was around one-fifth of that from the LOM soil (for both nonirrigated and irrigated treatments). This indicates that the organic matter treatment of the soil had a much more marked effect on reducing emissions than the irrigation treatment (which approximately halved emissions). In addition, the primary 1,3-D emission peak from the HOM soil occurred later than in the LOM soil, suggesting that the higher level of organic matter also delayed the emission release of the 1,3-D. Again, the role of the organic matter in the enhanced degradation of the 1,3-D is likely to be important here, but also, since organic matter is known to chemically complex 1,3-D (*10*), a possible explanation for this delayed emission may be a slower movement of the 1,3-D from the point of injection toward the soil surface, i.e., a greater degree of interaction with the soil solids.

In both soils, total emissions of 1,3-D from the column surfaces were substantially reduced (halved) due to irrigation treatment. As mentioned above in relation to the greater soil gas concentrations observed in the irrigated treatments, this was likely a result of much slower diffusion of the gas through the wet surface soil, which in turn gave the 1,3-D a longer contact time with the soil and a greater potential to degrade rather than be emitted. Reductions in emission due to irrigation were much greater than those recently reported by Gao and Trout (*13*) who, using soil columns, found values of 41−46% emission (as a percentage of the total added) for irrigated treatments, compared to 51% for the control. Their greatest irrigation-induced emission reduction was found at the highest level of water addition (a total of 13.2 mm over the first 24 h). They concluded that "frequent" addition of water would be required to substantially reduce emissions. Here, application for each of the first 5 days appears to have achieved this, presumably because this is the period over which the vast majority of the added 1,3-D is emitted. The fact that by 5 days most of the 1,3-D had either been emitted or degraded suggests that further application of water would not offer any greater benefit to emission reduction. It is considered that application of water for the first 5 days following fumigation is a practicable irrigation regime for a grower to carry out. The reductions in 1,3-D emissions were not as marked as have been noted for other types of treatment. For example, thiourea and thiosulfate amendments (*2*, *3*) produced emission reductions of 80−90%. Similarly, organic matter treatment appears to have a much greater effect on reducing emissions than irrigation (e.g., data in this paper). However, these approaches are not as cost-effective or as easily carried out as irrigation. Irrigation may therefore represent an effective compromise between reducing the environmental impact of fumigant use and practicability. In addition, the balance of rapid degradation of 1,3-D under conditions of high organic matter (and its beneficial effect in terms of emission reduction) with the time required for pesticidal efficacy must be taken into account.

The importance of simulating realistic conditions in a laboratory-based experiment are illustrated by the fluctuations in 1,3-D emissions over time. The many peaks in emission over the course of the experiment occurred during the warmest parts of the day, i.e., in the afternoons. Such relationships are not seen in laboratory-based experiments run at a constant temperature. The role of temperature in controlling the volatilization rate and, hence, emissions of fumigants such as 1,3-D has been reported previously (*21*, *22*). Therefore, both the daily and overall emission trends are likely to be less well represented when more simple, constant temperature, experiments are undertaken. If accurate trends in the release of fumigants from soil are required from laboratory-based experiments, it is suggested that a diurnal temperature fluctuation regime is likely to help achieve this. Given this observation, and the noted effects of using extended-length columns and realistic soil bulk densities, future studies should aim to further understand how well laboratory studies can simulate field conditions with regard to fumigant fate and transport, since the economic benefit of running laboratory-based, rather than field-based, studies is considerable.

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