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1,3-Dichloropropene Distribution and Emission after Gelatin Capsule Formulation Application

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The gelatin capsule (gel cap) formulation of 1,3-dichloropropene (1,3-D) was developed as a potential means of reducing 1,3-D emissions. The objective of this study was to determine the distribution, emission, and leaching of 1,3-D after applying the gel cap in soil columns. Comparable 1,3-D soil gas concentrations were obtained between a conventional liquid injection control and the gel cap application with film treatment. When the soil surface was irrigated with 39.6 mm water per day during the first 4 days, 1,3-D soil gas concentration was higher than the film treatments at depths below 20 cm, but lower concentrations were observed at 0−15 cm depth. The application of 1,3-D gel cap relatively reduced total 1,3-D emission by about 41% compared to liquid injection with film cover, and total 1,3-D emission was only 0.13% for 1,3-D gel cap application with 4 days of irrigation without a tarp. The results indicated that 1,3-D gel cap could be a promising new technology for reducing environmental emissions and potential human exposure.

KEYWORDS: 1,3-Dichloropropene; distribution; emission; leaching; gelatin capsule

INTRODUCTION

The use of methyl bromide (MeBr), which is a stratospheric ozone depleting compound in preplant soil fumigation for controlling soil-borne pests, will be phased out in China in 2015. Alternatives to MeBr are needed for soil fumigation to control soilborne pests and diseases. A potential alternative to MeBr is 1,3-dichloropropene (1,3-D). Emission of 1,3-D from soil can be significant and result in off-site air pollution, and thus agricultural use of 1,3-D in some regions of the world is currently restricted when applied with the shank injection method (*1*, *2*). For example, in some counties in Florida, 1,3-D use is limited to soils with an impeding layer, while in Prince Edward Island, Canada, 1,3-D use has been discontinued (*3*). To minimize the negative effects of 1,3-D on humans and the environment, it is necessary to develop efficient management strategies to control 1,3-D emissions and leaching.

Many field and laboratory studies have been conducted to evaluate strategies of reducing the emission of 1,3-D. Some studies showed that 1,3-D emission could be decreased by increasing application depth (*4*). Wang et al. found that using subsurface drip irrigation with lower dosage of 1,3-D has the potential to reduce emission (*2*). Many studies showed that fumigant emissions were reduced by covering the soil surface with polyethylene (PE) or virtually impermeable film (VIF) (*5*−*10*). However, agricultural films are expensive and difficult to dispose. Dungan et al. reported that applying composted animal manure to fields could reduce the 1,3-D emission (*11*). Some experiments showed that 1,3-D emission could be reduced by using thiourea to construct a reactive surface barrier (RSB) on the soil surface (*12*). Gan et al. reported that surface application of thiosulfate fertilizers may be a feasible and effective method to decrease 1,3-D emissions (*13*). The application of a surface water seal has shown the potential to reduce 1,3-D emissions by forming a high water content soil surface layer which serves as a diffusion barrier for fumigants gases (*8*−*10*, *14*−*18*). This technique may cost less than plastic films in areas where irrigation water is available. One possible limitation of the surface water seal is the potential of fumigant leaching, which could contaminate groundwater that may be close to the soil surface.

The gelatin capsule (gel cap) formulation of 1,3-D is a new concept that is being explored to reduce 1,3-D emissions and thus reduce worker and bystander exposure to 1,3-D. 1,3-D gel cap is easy to use in the soil without using special equipment. 1,3-D is a strong skin irritant and is a potential inhalation hazard, requiring personal protective equipment when applied in liquid form, which can be a barrier to its adoption. The gel cap formulation can offer a good solution to these application constraints. Field and laboratory studies have shown that the 1,3-D gel cap formulation is a promising new formulation with good efficacy to control soilborne pests (*19*).

There is no information in the literature on the environmental behavior of 1,3-D when applied as gelatin capsules in the soil. The objective of this study is to determine the soil distribution, emission, and leaching of 1,3-D when applied as a gel cap formulation in soil columns.

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MATERIALS AND METHODS

Soil, Chemicals, and Plastic Materials. Agricultural sandy loam soil samples were collected at a depth of 0−20 cm in a greenhouse around a suburb of Beijing (Malianwa district, Tujing village). The soil has a pH of 8.3 and a soil organic matter content of 2.76% and consists of 58.8% sand, 37.3% silt, and 3.9% clay. The soil was air-dried at room temperature and sieved through a 4 mm screen.

The analytical standard of 1,3-D (98% purity, cis:trans = 3:1) was obtained from Sigma-Aldrich Co. Technical 1,3-D (purity of 95%) for use in the gel cap was obtained from Beijing Zhongzhikehua Agricultural Technology Co. Ltd. The gel cap was manufactured on a capsule machine. The common capsule machine was modified by adding sealing equipment to eliminate 1,3-D emission during gel cap production. The outer film (0.8 mm thickness) of the gel cap is gelatin which was supplied by Qinghai Gelatin Co. Ltd. The average volume of a gel cap is 1 mL, and the average weight of 1,3-D (purity of 95%) in a gel cap is 0.8 g. The gel cap is designed to be applied to field soil at a 15−20 cm depth, by forming a hole and inserting a gel cap without special application tools, similar to planting a seed.

Hexane (analytical grade) and sodium sulfate anhydrous were obtained from Beijing Chemical Reagent Co. (Beijing, China). Polyethylene film (PE, thickness, 0.08 mm) was obtained from Baoding Juxing Plastic Factory (Hebei Province, China).

Soil Column Experiment. A repacked column system was constructed from a section of PVC pipe, an emission flux chamber, and a leachate collection system, as described by Zhang and Wang (*20*). The PVC column was 72 cm (height) by 15 cm (internal diameter) fitted with nine sampling ports which were placed at 5, 10, 15, 20, 25, 30, 40, 50, and 60 cm, respectively, from the soil surface. The sampling ports were made by inserting 10 cm long steel needles with noncoring-deflected tip (20 gauge, Dongguan Shengnuo Electronic Co, Ltd., Guangdong Province, China) through predrilled holes on the PVC column at the preselected depths.

The emission flux chamber was constructed with a 6.5 cm section of the same diameter PVC pipe by sealing one end with a transparent glass sheet. After the column was assembled and a 1,3-D gel cap treatment was applied, a continuous flow rate of 100 ± 10 mL min^{-1} of air through the chamber was maintained using a vacuum pump to sweep the volatilized 1,3-D into charcoal sorbent tubes (Tongzhou Jinnan Glass Instrument and Hardware Factory, Jiangsu Province, China). A gas flowmeter (Model RMA-11-SSV, Dwyer Instruments Inc., Michigan City, IN) was used to monitor the rate of air flow. The internal surface of the PVC column and emission chamber was lined with a Teflon film (thickness 0.08 mm, Teflon technology center, Beijing) to prevent adsorption of 1,3-D.

The leachate collection system consisted of a funnel fitted to the bottom of the column for directing water, through a glass U-tube, to a collection flask.

Air-dried soil was preconditioned by adding deionized water and then adjusting the water content to 15% (w/w), close to field capacity. The moist soil was kept in closed containers for 24 h to redistribute and equilibrate soil−water. A total of 20.4 kg of soil was packed in each column to give a bulk density of 1.6 g cm^{-3}.

To evaluate the distribution, emission and leaching of 1,3-D from the gel cap, and to compare to convential 1,3-D application methods, the following treatments were applied to soil columns:

(a) 1,3-D liquid injection with LDPE tarp
(b) 1,3-D gel cap application with LDPE tarp
(c) 1,3-D gel cap application with 4 days of irrigation with no tarp

For treatments a and b, a piece of 0.08 mm thick LDPE tarp was placed on the soil surface, and then the emission sampling (flux) chamber was installed over the tarp. In treatment a, 0.8 g of 1,3-D liquid was injected into the column center through an injection port connected with a steel needle extending to the center of the column. In treatment b, a single 1,3-D gel cap (containing 0.8 g of 1,3-D) was put into the column center through a 1 cm diameter predrilled hole at the 20 cm depth and repacked with the same soil to the original bulk density. A Teflon-faced silicone rubber septum (3 mm thick; Agilent) was installed to seal the port. Sampling for 1,3-D began after the connection was sealed with silicon sealant.

For treatment c, prior to inserting the 1,3-D gel cap, the column was pretreated by adding water at the soil surface until approximately 1 L of leachate was collected from the bottom. The final 250 mL of leachate was kept for analysis and used as background fumigant concentration of the soil. Exactly 710 mL of water was applied daily at approximately 2 mL min^{-1} rate during the first 4 days to simulate 39.6 mm of daily irrigation under field conditions. The leachate was collected from the outlet at the column bottom at 6−12 h intervals. The volume of leachate was measured for each collection event, and the collected leachate was kept at 4 °C before GC analysis.

Each treatment was repeated three times. Monitoring and sampling were done for 32 days. The experiments were conducted at room temperature (20−33 °C).

Sampling and Analysis. The charcoal tubes were connected with the outlet of the flux chambers at the top of the soil column in order to absorb fumigants evaporating from the soil surface. Charcoal tubes were replaced every 30 min during the first 48 h and then every 1−2 h throughout the next 48 h. Incrementally, longer sampling intervals were used later in the experiment until 32 days after fumigant application. At night, a chain of the tubes (four to six) was connected to ensure trapping of all emissions. The last charcoal tube in the chain always showed no detection of 1,3-D, indicating breakthrough of 1,3-D had not occurred within detection limits of the instruments. The charcoal tubes were stored at −20 °C before analysis using gas chromatography (GC).

Charcoal sorbent tubes were broken, and all materials were transferred into a 20 mL clear headspace vial. Two milliliters of hexane was added, and the vials were immediately sealed with Teflon-lined septa and an aluminum cap. The headspace of the vials was analyzed using an Agilent 7890A gas chromatograph coupled with an Agilent 7694E headspace sampler and a micro electron capture detector (Agilent Technologies, Inc., Palo Alto, CA). An AB 0525-3002 capillary column (30 m length × 0.25 mm i.d. × 5 μm film thickness, Abel Industries, Inc. Wilmington, DE) was used for analysis. 1,3-D was quantified through a four-point external calibration within expected ranges, using the blank charcoal tube contents spiked with standard chemicals. The detector temperature was 250 °C. The oven temperature was held at 120 °C for 6 min. The autosampler headspace conditions were as follows: 1.0 mL sample loop; 90, 93, and 95 °C for sample equilibration and loop and transfer line temperature, respectively; 5 min vial equilibration time; 0.5 min loop filling time; 0.05 min loop equilibration time; 0.1 min pressurize time; 0.5 min injection time; 8.2 PSI vial pressurization; low shake mode for 1 min.

For the determination of 1,3-D concentration of the soil-gas phase, 0.5 mL of soil gas was collected from the sampling ports with a gastight syringe at 1, 3, 6, 12, 24, and 48 h and at 3, 4, 5, 6, and 11 days after 1,3-D application. The gas sample was injected into a 20 mL clear headspace vial which was immediately crimp-sealed with an aluminum cap and Teflon-faced butyl-rubber septum (Agilent). This method has been shown to be accurate and reproducible (*8*, *21*). To avoid a moisture effect on the fumigant stability, 0.2 g of sodium sulfate was added in each vial before sample injection. If analysis could not be finished immediately, the vials were stored in a freezer at −70 °C. All samples were analyzed within 72 h (*9*, *22*). Soil air samples in crimp-sealed 20 mL vials were analyzed under the same conditions as the emission samples, except that no solvent was added into vials. A calibration curve was established by analyzing vials containing known amounts of 1,3-D standard under the same conditions as for soil air samples.

1,3-D residue in the leachate samples was determined using the automatic static headspace GC analysis method (*20*). A 10 mL leachate sample was transferred into 20 mL headspace vials, and 2.5 g sodium chloride was added to create a salt solution. The vials were placed on the Agilent-7694E Headspace Sampler for GC analysis after the salt dissolved. The solution was thermally equilibrated in the closed headspace vials within the autosampler oven for 15 min, and then an aliquot of the headspace was introduced into the GC column for analysis. The GC conditions were the same as for the emission samples. Calibration curves were created by spiking known amounts of 1,3-D into 10 mL leachate background samples and analyzed with the same GC settings as mentioned above.

Upon termination of the experiment, soil samples from each column were taken from seven depth increments: 0−10, 10−20, 20−30, 30−40, 40−50. 50−60, and 60−72 cm. Soil−water content and the 1,3-D residue in the soil were determined. The extraction procedure for soil samples followed the methodology described by Gao et al. (*8*). An equivalent dry weight of 8 g of soil was added in a 20 mL clear vial that contained Na_2SO_4

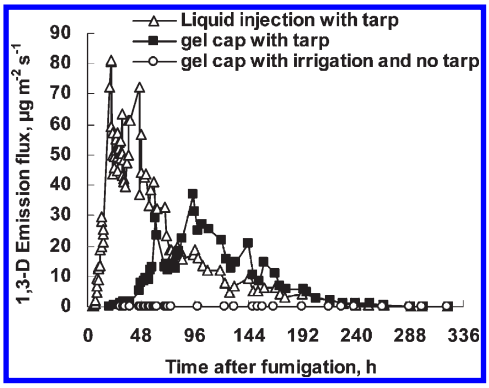

Figure 1. 1,3-D emission fluxes from soil column treatments.

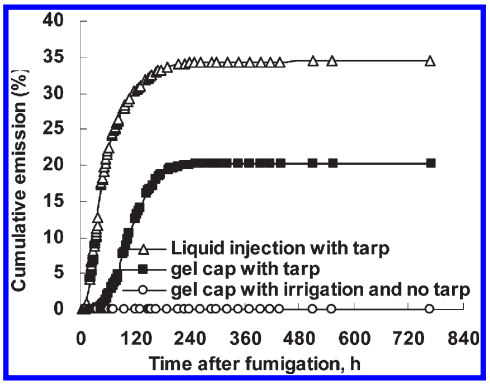

Figure 2. Cumulative 1,3-D emission (percent of applied 1,3-D) from soil column treatments.

Table 1. Maximum Flux and 1,3-D Emissions from Soil Column Treatments

treatment	max emission flux (μg m^{-2} s^{-1})	total emission (% of applied)
injection application with tarp	81.37 ± 1.45 a[a]	34.40 ± 1.89 a
gel cap application with tarp	36.82 ± 5.19 b	20.26 ± 0.63 b
gel cap application with irrigation and no tarp	0.03 ± 0.005 c	0.13 ± 0.011 c

[a] Figures in the same column with the same letter are not statistically different according to Duncan's multiple range test (P = 0.05).

(amount depended on soil−water content at a 7:1 ratio of Na_2SO_4 to water). Eight milliliters of ethyl acetate was added in the vial, which was then crimp-sealed with aluminum caps and a Teflon-faced butyl-rubber septum, and then the sample was extracted with a shaker for 30 min. After centrifugation at 2000 rpm for 3 min, the supernatant was separated and transferred into a 20 mL vial for fumigant analysis using the chromatographic conditions described above.

The data were statistically analyzed according to Duncan's multiple range test with the Statistical Analysis System (SAS) computer program.

RESULTS AND DISCUSSION

1,3-D flux. The emission fluxes for 1,3-D are shown in **Figure 1** and summarized in **Table 1** for the various treatments. Emission of 1,3-D began at 3.5 h after application for the liquid injection treatment; the flux increased with time and reached a maximum value of 81.4 μg m^{-2} s^{-1} at 22 h after injection. The 1,3-D flux remained over 30 μg m^{-2} s^{-1} during the 12−68 h after injection and gradually decreased thereafter. The 1,3-D gel cap application delayed fumigant emissions for at least 16−17 h. The peak flux of 36.8 μg m^{-2} s^{-1} occurred at 95 h after gel cap application with tarp treatment and was less than 50% of the maximum of the injection treatments. The flux values were significantly reduced by the 4 days of irrigation, and the maximum 1,3-D flux was only 0.026 μg m^{-2} s^{-1}, which occurred at 157 h after the gel cap application without a tarp. The flux remained low (< 0.03 μg m^{-2} s^{-1}) during the experimental time. The results indicated that the gel cap application resulted in lower maximum emission rates and later emission than for a liquid injection treatment. The addition of a surface water application provided an effective means of further reducing the 1,3-D emissions.

Cumulative Emissions. Cumulative emission of 1,3-D from the column treatments during the 32 day experimental period are shown in **Figure 2** and summarized in **Table 1**. Emission percentages (percent of total 1,3-D applied) over the entire 32 day study were 34.4% for the 1,3-D liquid injection, 20.3% for 1,3-D gel cap application, and only 0.13% for 1,3-D gel cap application with 4 days of irrigation without tarp. About 90% of total emission occurred in the first 5 days after the liquid injection treatment, and over 80% of cumulative emission occurred during 3−8 days after gel cap application. There was no significant emission of 1,3-D from the gel cap application with the 4 day irrigation treatment.

The 1,3-D gel cap application relatively reduced emissions by about 41% compared with 1,3-D liquid injection. There was no emission during the first 20 h after the gel cap application. The results indicated that gel cap formulation effectively reduces 1,3-D emissions and thereby reduces risks to workers and bystanders during fumigation.

Concentrations of 1,3-D in Soil-Gas Phase. The distribution of 1,3-D in the soil-gas phase over time is shown in **Figure 3**. The highest concentration of 1,3-D was detected near the application port (20 cm depth) or at the 15−25 cm depth range. For the 1,3-D liquid injection treatment, the highest concentration (137.0 μg cm^{-3}) occurred at 1 h after fumigant injection because 1,3-D had not dispersed to other depths yet. Eventually the concentration of 1,3-D decreased at the 20 cm depth, but it first increased and then declined at the other depths. After 144 h, 1,3-D concentrations in the soil-gas phase had decreased to extremely low levels (< 0.3 μg cm^{-3}) in the soil columns.

Concentrations of 1,3-D at all depths increased initially and then decreased with time for both gel cap application treatments. The concentration of 1,3-D in the soil column increased abruptly at 48 h after fumigant application, indicating that 1,3-D in the gel cap was slowly released into the surrounding soil initially, but rapid breakdown of the gel cap occurred at 24−48 h after gel cap application. Upon complete degradation of the gel cap, 1,3-D was released into the soil rapidly and subsequently diffused throughout the soil column. The highest concentration of 1,3-D detected for gel cap application with tarp treatment and gel cap application with 4 days of irrigation treatment were 24.6 and 97.3 μg cm^{-3}, respectively, at 20 cm depth. The fumigant concentration reached the highest level at the bottom sampling port at approximately 72 h, or later, for both treatments. The average 1,3-D concentration in the soil-gas phase throughout the columns was 3.69 μg cm^{-3} for the gel cap application at 72 h and 4.64 μg cm^{-3} for the injection treatment at 48 h. Also, field and laboratory studies showed that the efficacy of gel caps was same as for the injection treatment (*19*), though the highest 1,3-D concentration for gel cap application with a tarp was lower than that for injection treatment.

The movement of 1,3-D may be hindered by the 4 day irrigation of 39.6 mm per day, and so the peak of concentration at the 20 cm depth for the irrigation treatment was significantly higher than that of nonirrigated treatment, whereas the concentrations at 5 and 10 cm depth were very low (< 0.3 μg cm^{-3}). This suggests that excessive water in soil slows down diffusion of 1,3-D to the soil surface, which could result in poor 1,3-D efficacy. The gel cap could be applied closer to the soil surface (e.g., 5 cm depth) with irrigation to ensure fumigant efficacy and emission reduction concurrently.

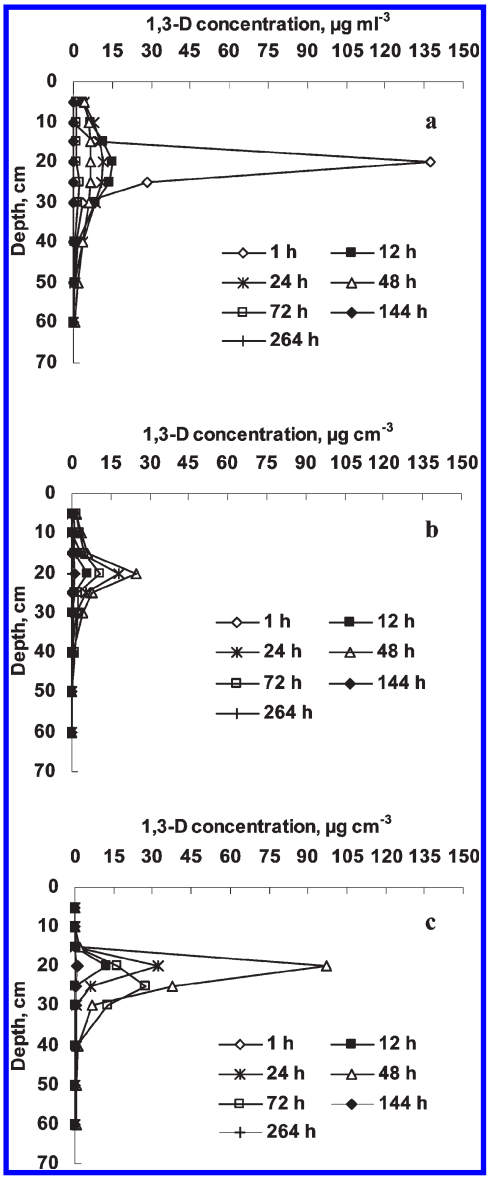

Figure 3. Distribution of 1,3-D in the soil-gas phase in soil columns: (a) 1,3-D liquid injection with PE tarp; (b) 1,3-D gel cap application with PE tarp; (c) 1,3-D capsule application with 4 days of irrigation and no tarp.

Gel caps release 1,3-D into the soil slowly as the capsules degrade, which decreases soil-gas phase concentrations but increases the soil residence time. These factors combine to decrease the potential for volatilization from soil.

Leaching and Residual Fumigation in Soils. The limits of detection (LOD), which are considered to be the concentrations that produced a signal-to-noise (S/N) ratio of 3 for *cis*- and *trans*-1,3-D, were all 0.07 μg L^{-1}. In the first 4 days, the volume of collected leachate from the 4 day irrigation with no tarp treatment was about 600−650 mL per day, and the concentration of 1,3-D in leachates was lower than the LOD. The results indicated that the application of a 1,3-D gel cap formulation may be able to reduce the potential contamination of groundwater if the fumigant was applied in areas where the groundwater was close to the soil surface. This conclusion should be validated under additional experimental conditions (e.g., different types of soil textures and irrigation amounts).

The residue of 1,3-D in soil was not detectable at the end of each column experiment, exactly 32 days after fumigation. This indicated that 1,3-D had degraded in the soil within 32 days and is consistent with the reported soil half-life of 1,3-D of 2−17 days in a range of soils (*23*).

The results showed that application of 1,3-D gel cap could reduce total 1,3-D emission by about 41% compared to conventional liquid injection with film cover. The data from laboratory bioassay and field trials indicated that 1,3-D gel cap can effectively control nematodes and is partially effective for pathogens, similar to 1,3-D injection application (*19*). The results indicated that 1,3-D gel cap is a promising new formulation which can reduce negative environmental impact and reduces potential risks to workers or bystanders during fumigation. The gel cap formulation can provide advantages in handling and application compared to the traditional liquid formulations of 1,3-D. Further research is required to determine the optimal irrigation water and application depth of gel cap in soil to provide the maximum efficacy and environmental benefit.

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