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Clomazone dissipation, adsorption and translocation in four paddy topsoils

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Abstract: Laboratory experiments about the dissipation, adsorption and translocation in four paddy topsoils were conducted in this paper. From the results it can be concluded as follows: the dissipation rate of clomazone differed greatly in different paddy soil derived from different parent materials. The half-lives for clomazone degradation in paddy soils ranged from 5.7 to 22.0 d. The order of clomazone dissipation rate was reddish yellow paddy soil > alluvial sandy paddy soil > yellow clayey paddy soil > purple sandy paddy soil. Clomazone sorption quantity was significantly correlated with organic carbon (R^2 = 0.62) and clay content (R^2 = 0.67) in the tested paddy soils. Positive correlation was found between apparent K_d value and cation exchange content (CEC). The consequences for the adsorption of different soils were purple sandy paddy soil > yellow clayey paddy soil > reddish yellow paddy soil > alluvial sandy paddy soil. Under the simulated rainfall of 200 mm through four different unsaturated soil lysimeters over 24 h, clomazone was readily to be leached into lower surface soil and there was about 2.6%—4.2% of applied clomazone leached out of 20 cm cultivated soil layer. Translocation experiments showed that the order of clomazone leaching ability was: alluvial sandy paddy soil > reddish yellow paddy soil > yellow clayey paddy soil > purple sandy paddy soil. Simple regression results manifested that factors like CEC, organic carbon, clay, and adsorption rate constant had been negatively correlated with the percentage of clomazone loss from soil lysimeters.

Keywords: clomazone; dissipation; adsorption; translocation; paddy topsoils

Introduction

Clomazone, (2-[(2-chlorophenyl) methyl]-4, 4-dimethyl-3-isoxazolidinone), is a soil-applied isoxazolidinone herbicide produced by FMC Corp. It is introduced for annual grass and broad-spectrum weeds in soybeans (Rani, 1998; Vyas, 2000). It is also currently registered for use in pumpkins [*Glycine max* (L.) Merr.] and has potential for use in other crops such as cotton, tobacco, and various vegetable crops (Warfield, 1985; Scott, 1992; Madiwalar, 1998; Miller, 1999; Brown, 2002). In susceptible species, clomazone interferes synthesis of chlorophyll and carotenoids and prevents accumulation of plastid pigments of susceptive grass, resulting in foliage devoid of pigmentation (Duke, 1985).

In recent years, many researches on application of clomazone in rice for weed control were performed to evaluate the influence of clomazone on rice (Webster, 1998; 1999; Bollich, 2000; Pratley, 2002; Taylor, 2002). It was reported that clomazone at rates below 1.7 kg/hm^2 did not reduce grain yield of rice, even when clomazone causes early-season bleaching. Furthermore disulfoton use has the ability of reducing bleaching of rice follage caused by clomazone and rice was more injured by clomazone on silt loam soil than on silty clay soil (Jordan, 1998). In another study, clomazone was proved to enhance the grain yield of rice comparing to other non-treated with clomazone, which showed the great potential of clomazone use in rice (Webster, 1999; Bollich, 2000).

Recent concerns over the contamination of groundwater supplies by agricultural pesticides has led to renewed interest in the fate of herbicides including dissipation, adsorption and leaching process. Clomazone is not labeled for use in rice in the U.S., but is used extensively in rice production in South America (Jordan, 1998a). Effect of barnyard grass control with clomazone equals or exceeds that with residual herbicides currently registered for use in rice (Hatfield, 1996; Jordan, 1996). Furthermore, clomazone was reported to be effective to control some resistant weeds and appeared to be promising in rice (Baldwin, 1995; Jordan, 1998b). Risk assessment especially, on the environment after clomazone application in paddy rice was not confirmed so far. In formal study, toxicity tests proved that the EC_{50} of algae (*Selenastrum capricornutum*), macrophytes (*Lemna valdiviana*), and cladocera (*Daphnia similes*) for clomazone ranged from 14.5 to 32.2 mg/L and clomazone had no effect on any parameters of tested first-instar larvae at concentrations up to 0.288 mg/L ($p > 0.05$) (Jonsson, 1998c). As human beings are the final acceptors in a food chain, accumulating effects of pollutants could not be

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avoided. Some pollutants at certain concentration may be harmless for lower organisms but poisoning response would occur if they were accumulated to certain concentration in the bodies of higher organisms, especially for human beings. Though clomazone is low toxic, clomazone toxicity should not yet be ignored. Associated with human health, tolerance for clomazone was provided as follows: winter pumpkin 0.1 mg/kg, sweet potato 0.05 mg/kg, peppers 0.05 mg/kg, watermelon 0.1 mg/kg, cottonseed 0.05 mg/kg. The permitted amount of daily intake (ADI) for one person is 0.043 mg/kg, which is at a low concentration level. As studies on sorption and degradation of clomazone in soils were performed before, clomazone degradation was affected by soil moisture, temperature, microorganisms and pH etc. (Loux, 1989; Li, 2004), and sorption was influenced by organic matter, clay content and cation-exchange content (Mervosh, 1995).

To our knowledge, no studies under controlled laboratory conditions on clomazone dissipation, sorption and leaching ability in paddy soils have been published in reviewed literature based on the specialties of paddy soils. The objectives of this study were to determine the behavior of clomazone, compare the difference of dissipation, sorption and leaching in various paddy soils and carry out assessment of environmental risk.

1 Materials and methods

1.1 Soil preparation

Moist soils were collected in October 1999 from the top 10 cm. Those fields had never received clomazone application before. Soil was air dried and sieved through a 2 mm screen, thoroughly homogenized, and stored at - 20℃ in thin polyethylene bags until use. The characteristics of different paddy soils about several fields located in Changsha County of Hunan Province are shown in Table 1, in which rice had just been harvested. All soil samples were collected on the same date.

Table 1 Basic chemical properties for the tested soils

Soil type	Parent materials	pH	*CEC*, cmol/kg	Organic carbon content, %	Clay content, g/kg
SE-1 Yellow clayey paddy soil	Weathering materials of slate and shale	4.9	11.4	1.85	724.1
SE-2 Reddish yellow paddy soil	Red earth of the quaternary	5.4	16.0	1.22	613.0
SE-3 Purple sandy paddy soil	Weathering materials of purple sandstone	5.9	15.8	2.06	696.1
SE-4 Alluvial sandy paddy soil	Alluvial deposit of rivers	5.0	7.2	1.24	587.2

1.2 Chemicals

Technical-grade clomazone (99.97% purity) was obtained from FMC Corp. All other inorganic reagents used were of HPLC reagent grade.

1.3 Clomazone extraction procedures

Soil samples (25 g dry wt. basis) were placed into 250 ml low-density polyethylene screw-top bottle. Soil samples were immersed in 70 ml acetone for one night. All samples were shaken on an agitator for 1 h and filtered through pressure-relief method. Soil extracts were filtered through two filter papers, retaining the filtrate. The residue was rinsed three times with approximately 10 ml acetone each time and then filtered through a membrane. The combined filtrates were transferred to 250 ml tap funnel and diluted in 100 ml of distilled water. With 10 g sodium chloride and 50 ml petroleum benzin added in, the tap funnel was shaken for several minutes, then placed quietly and consequently, aqueous phase was separated and organic phase was remained. The aqueous phase was extracted twice by equal 30 ml petroleum benzin. The organic extracts were combined and evaporated to 2 ml by rotoevaporation. The concentrated organic phase of each sample was dried under nitrogen, and the residue was dissolved in 1 ml methanol then filtered through a 0.2 μm syringe filter. These extracted samples were stored in lighttight vials at - 20℃ until HPLC analysis.

1.4 Sample analyses

Concentrations of clomazone in soil extracts were determined using reverse-phase high performance liquid chromatography (HPLC). The analytical column was packed with 4 μm C_{18} media and the length was 25 centimeters. A guard column was used to filter contaminated matter and thus protect the analytical column. All solvents were HPLC grade and all injection volumes were 20 μl. Analytical parameters for clomazone included absorbance detecting wavelength of 210 nm and a mobile phase flow rate of 0.8 ml/min. The mobile phase was methanol and water (75 : 25 by volume). Clomazone retention time was 2.429 min. All recoveries were ≥ 90% (data not shown). Clomazone concentrations were quantified using an external standard technique, which was accomplished by comparison of integrated curve areas with a standard curve derived from representative standards. All extracts were within the linear range of constructed calibration curves.

1.5 Dissipation

For residue study, each soil sample (25 g dry soil) from four paddy fields was put into triangular flask. Soil moisture was adjusted to the water content at a tension of 857 kPa by distilled water and then incubated in the dark at 25℃ for four days in advance. Then, 0.55 mg of clomazone was applied to the soil surface and these samples were reincubated in the dark at 25℃. Sampling times were 0, 3, 5, 7, 15, and 30 d after clomazone application. Each treatment was replicated three times and all flasks in the experimental run were covered with a single layer of laboratory film, maintaining humidity and preventing evaporation.

The most commonly used kinetic model for describing pesticide degradation in soils is the first-order equation. Clomazone concentration data was also fit to the first-order degradation formula described by the following linear equation: $C_t = C_0 e^{-kt}$, where C_t is the concentration at time t. C_0 is the initial concentration, k is the first-order

rate constant. Half lives(DT_{50}) of clomazone were calculated through such an equation: $DT_{50} = \ln 2/k$, where DT_{50} is the half-life of clomazone, k is the first-order degradation kinetic coefficient.

1.6 Adsorption

Clomazone adsorption kinetics was examined using a batch equilibration method. A solution/soil ratio of 10:1 with an initial solution concentration of 2.5 mg/kg was prepared for adsorption equilibrium period determination experiment. The kinetics study examined the rate of clomazone adsorption over time and established an equilibration period for later K_d determination. In the kinetics experiment, clomazone adsorption was measured in soil samples at 0, 2, 4, 8, 12, 16, 20, 24, 28, 32, 36, 40, 44, 48 h after clomazone addition. It proved that the complete equilibration period was 24 h, which was similar to described(Loux, 1989) and used in subsequent experiments examining clomazone adsorption. Adsorption isotherms were determined using standard clomazone (99.7% purity). Appropriate volumes of clomazone stock solutions were diluted with 0.01 mol/L $CaCl_2$ to give final solution concentrations of 0.50, 1.00, 2.50, 5.00, and 10.0 mg/L. All samples in glass triangular flask with lids covered were equilibrated on shakers at 23℃ for 24 h. Sorbent and solution phase were separated by centrifugation at 2300 r/min for 15 min. Final concentrations of clomazone in the solution phase should be extracted by petroleum benzin as mentioned above. The amount of clomazone adsorbed by the soil was calculated according to the difference between initial and final solution phase concentrations.

Adsorption of clomazone in paddy soils gave an approximate fit to the Freundlich adsorption isotherm equation $C_s = K_d \cdot C_{eq} 1/n$, which can be changed into $\ln C_s = \ln K_d + (1/n)\ln C_{eq}$, where C_s is the adsorbed amount and C_{eq} is the equilibrium concentration, and K_d as the distribution adsorption coefficient is a measure of the strength of sorption, which can be reflected by the slope of the line equation. The values of K_d may be different for various kinds of soils. The higher slope of the equation, the higher clomazone adsorption degree. The constant $1/n$ reflects the degree of nonlinearlity between solution concentration and absorbed amount. Correlations were made between the constant K_d values and pH, clay content, *CEC* and organic carbon content. Organic carbon constant was determined by the equation $K_{oc} = K_d/$ organic carbon, where K_d is the adsorption coefficient, K_{oc} is the organic carbon constant.

1.7 Translocation

Soils of approximate 200 g with 10% of water content was packed into the stainless steel lysimeters (with the diameter of 3.46 cm and length of 32 cm) which could be taken apart into eight segments with 4 cm for each. Soil amount and density of each column should be noted to see if it was filled uniformly and tightly during the course. After finishing the process, a layer of glass filament was covered on the surface and then a layer of filter paper was added with some sandstone pressed on it. After application of clomazone with 0.1004 mg, which is equal to 1.07 kg/hm^2 and below to recommended level 1.7 kg/hm^2, onto the soil surface of lysimeters, simulated rainfall of 200 mm began to fall at the correct speed, which equals to 188 ml distilled water to get through the lysimeters uniformly over 24 h. Leachate was collected for determining the clomazone content. Each treatment was replicated three times. With the leaching process completed, the columns were taken apart and each segment with 4 cm long was used for determining soil water content, clomazone content of soil and leached liquid.

2 Results and discussion

2.1 Dissipation

Clomazone dissipation was fit to the first-order kinetic equation (R^2 = 0.989—0.999). In different paddy soil derived from the different parent soil, the degradation speed of clomazone differed greatly. The half-lives for total clomazone degradation in paddy soils ranged from 5.7 to 22.0 d(Fig.1). In reddish yellow paddy soil, the greatest speed occurred with DT_{50} of 5.7 days. While in purple sandypaddy soil, DT_{50} of clomazone reach 22.0 d. In previous study, the degradation of clomazone is dependent on microorganisms (Mervosh, 1995). This fact was proved in different paddy soils(Li, 2004). Although biodegradation rates in soils often become slower than the rate predicted by first-order kinetics thus underestimating the half-life of the clomazone disappearance. Therefore, the actual DT_{50} of clomazone in four different paddy soils was greater than that determined by first-order kinetic model. This experiment showed that DT_{50} values of clomazone were somewhat different from most earlier studies and seemed more rapid, comparing to the DT_{50} range of 28—84 d described (Mervosh, 1995; Gallaher, 1996; Kirksey, 1996), but similar to the results described (Cumming, 2002). According to previous research, Clomazone degradation was affected by many environmental factors such as temperature, moisture, pH and properties of soils (Mervosh, 1995). In this study, the main factor resulting difference of clomazone disappearance in paddy soils was the various paddy soil types. Especially, soil organic carbon content was correlated with degradation rate constant K at the 1% level(data not shown). No obvious correlations were presented between K and other properties like pH, *CEC* content and clay content. This means clomazone dissipation rate in soils was quite different due to various soil properties. The order of clomazone dissipation rate was reddish yellow paddy soil > alluvial sandy paddy soil > yellow clayey paddy soil > purple sandy paddy soil.

2.2 Adsorption

The results about the clomazone adsorption experiments in paddy soils are shown in Table 2. The related coefficient (R^2) of equations describing adsorption isotherms ranged from 0.93 to 0.99 for clomazone. The value of $1/n$ was from 0.67 to 0.80, which indicated the nonlinerity adsorption.

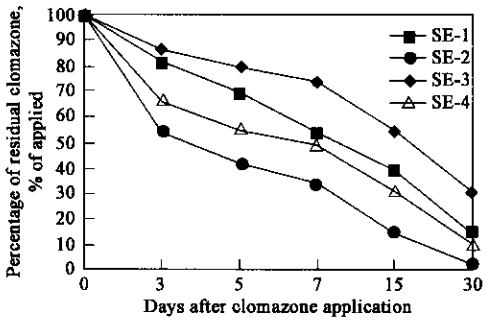

Fig.1 Degradation kinetic curves of clomazone in different paddy soils

The R^2 value for the regression of K_d on K_{oc} was only 0.62, which indicated clomazone sorption was significantly correlated with organic carbon in such paddy soils. When linear regression of K_d on clay content was performed, clay content played a most important role for the adsorption of clomazone with R^2 value 0.67. Meanwhile, positive correlation($R^2 = 0.66$) was found between K_d and *CEC*. In our work, clomazone sorption was weakly correlated ($R^2 = 0.32$) with pH values. K_{oc} values for the paddy soils studied ranged from 354 to 767. The mean K_{oc} value for all samples was 554. Soil with the greatest K_{oc} values just contained the lowest organic carbon content, which was similar to previous results(Loux, 1989). It can be ensured that purple sandy paddy soil had the greatest adsorption degree for clomazone due to the highest organic carbon content and high clay content, then yellow clayey paddy soil, and then reddish yellow paddy soil. Alluvial sandy·paddy soil had the least adsorption strength for clomazone because of lowest clay content and organic carbon. It can be concluded that with the characteristics of the herbicide itself involved, sorption of clomazone was controlled by chemical and physical properties of soils. But how the mechanism of clomazone adsorption works was not very certain.

Table 2 Distribution coefficients (K_d), $1/n$ values and K_{oc} values for clomazone adsorption in tested paddy soils

Samples	K_d	R^2	$1/n$	K_{oc}
SE-1	10.19	0.96	0.73	550
SE-2	9.36	0.93	0.80	767
SE-3	11.25	0.99	0.67	546
SE-4	6.10	0.99	0.70	354

2.3 Translocation

It showed that clomazone was readily to be leached into lower surface water for its high water solubility in this studied paddy soil. There was about 2.6%—4.2% of applied clomazone leached out of 20 cm cultivated soil layer. Alluvial sandy paddy soil column showed the greatest clomazone leaching loss(4.2%) from its column, then reddish yellow paddy soil (3.6%), and then yellow clayey paddy soil (3.10%). The purple sandy paddy soil had the least clomazone leaching loss(2.6%) from its column. Then, the order of clomazone leaching capacity was alluvial sandy paddy soil > reddish yellow paddy soil > yellow clayey paddy soil > purple sandy paddy soil. The distribution of clomazone in four paddy soils is shown in Fig.2. For yellow clayey paddy soil, clomazone concentration gradually increased with the depth of soil column increasing, except for the depth of 16—20 cm. But for reddish yellow paddy soil, clomazone content reached summit in the depth of 8—12 cm. Clomazone was mainly concentrated in the depth of 0—12 cm for the purple sandy paddy soil. There was the least total content of clomazone remaining in the alluvial soil column over 24 h with 200 mm rainfall, and the greatest clomazone loss occurred with the summit concentration in the depth of 12—16 cm.

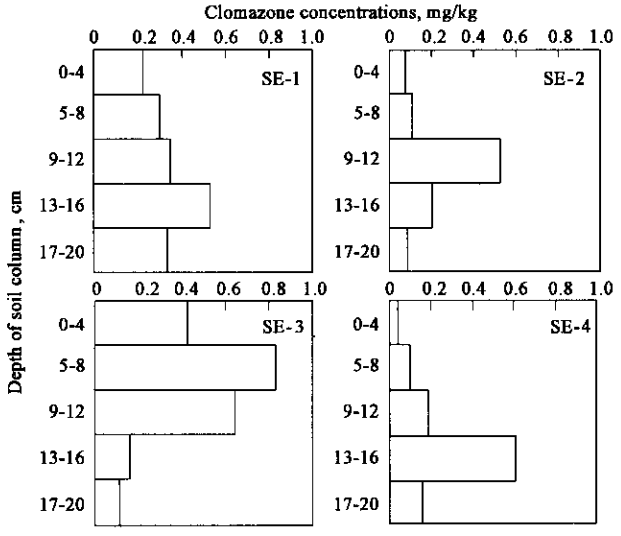

Fig.2 Vertical distribution of clomazone in four soil columns

Regression results showed that adsorption constant K_d was significantly correlated($R = -0.96$) at 5% level with clomazone leaching loss from different soil columns. This means that sorption of herbicides to soils had a significant influence on the fate of herbicides leaching below the soil surface. Leachability of clomazone in soils was a complicated process and influenced by many factors such as adsorption, degradation, plant cover and the interactions among these ingredients. Any factor that could influence sorption and degradation etc. would affect the translocation of clomazone. Comparing the results, it can be observed that main factors controlling leaching process of clomazone were soil properties including pH, organic carbon content and clay content etc., which also influenced the sorption and degradation trait of clomazone. Simple regression results manifested that these factors such as pH, *CEC*, organic carbon, clay, and degradation rate constant had been negatively correlated with the percentage of clomazone loss from soil columns(Table 3).

Table 3 Correlation coefficient (R) of percentage of lost clomazone through leaching with other factors

Parameters	Percentage of clomazone leaching loss from soil columns/ % of applied
Adsorption, K_d	-0.96*
pH	-0.603
Cation-exchange capacity	-0.679
Clay, %	-0.87
Organic-carbon, %	-0.92
Degradation, K	-0.565

Notes: * Correlation significance denoted at the 0.05 level

It suggested that the risk of clomazone application to paddy fields during heavy rainfall resulting in underground water contamination should be possible due to the mobility of

clomazone, especially for those fields with high groundwater level and sandy land with low organic carbon, low clay content, low cation-exchange content and no good cover of plant. Addition of organic fertilizer and lime into the paddy soils may be efficient for alleviating the tension of underground water pollution.

3 Conclusions

Clomazone degradation varied greatly among four kinds of paddy soils. The dissipation half-lives of clomazone in these soils ranged from 5.7 to 22.0 d. In reddish yellow paddy soil clomazone degraded the most rapidly, then in alluvial sandy paddy soil and then in yellow clayey paddy soil. Clomazone in purple sandy paddy soil degraded most slowly with 22.0 days of half-life.

Clomazone sorption was significantly correlated with organic carbon and clay content in the tested paddy soils. For the relative low organic matter and clay content, clomazone was adsorbed weakly by alluvial sandy paddy soil. The order for adsorption affinity in tested soils was purple sandy paddy soil > yellow clayey paddy soil > reddish yellow paddy soil > alluvial sandy paddy soil. Positive correlation was also found between apparent adsorption coefficient K_d and cation exchange content.

Under the simulated rainfall of 200 mm over 24 h leaching through unsaturated soil lysimeters, different clomazone leaching loss was found and 2.6%—4.2% of applied clomazone leached out of 20 cm cultivated soil layer for four different soils, which meant clomazone was readily to be leached into lower surface soil and there was some risk of polluting the underground water by the application of clomazone.

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