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Persistence of Diquat, Endothall, and Fluridone In Ponds[1]

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ABSTRACT

Persistence of diquat, endothall and fluridone was measured in the water of irrigation supply ponds. Diquat concentrations decreased logarithmically in three ponds treated with 1 ppm diquat, and only trace concentrations were measured 35 hours after application. The concentration of endothall in ponds treated with Aquathol K decreased linearly with time after treatment. The initial concentration was 2 ppm and it is predicted using regression analysis that the concentration approached zero 26 days after treatment. Fluridone concentration decreased logarithmically with time after treatment and approached zero 64 and 69 days after treatment in two ponds treated with Sonar 4AS. In a pond treated with Sonar 5P no significant decrease in fluridone concentration was observed after 53 days.

Key words: herbicides, irrigation, Aquathol, Sonar, residue, North Carolina.

INTRODUCTION

Piedmont and coastal areas of the Southeastern United States rely heavily upon impounded surface waters for irrigation sources. North Carolina alone contains over 63,000 small ponds that are managed mainly for irrigating valuable golf course greens, ornamental plantings, and high cash crops such as tobacco. These impoundments often become so heavily overgrown with aquatic vegetation that pump intakes continuously become fouled and withdrawal of water for irrigation is severely restricted.

Most of these weed problems can be managed with aquatic herbicides, but groundskeepers and agricultural producers are often hesitant to use these compounds because of the potential for phytotoxic herbicide residues.

Although previous studies have shown that diquat (6,7-dihydrodipyrido[1,2-*a*:2′,1′-C]pyrazinediium ion), endothall (7-oxabicyclo[2,2,1]heptane-2,3-dicarboxylic acid), and fluridone (1-methyl-2-phenyl-5-[3-(trifluromethyl) phenyl]-3(IH)-pyridone) are not persistent in the aquatic environment (Calderbank, 1968; Hiltibran, 1962; West et al, 1983) data are variable among studies; and persistence can be affected by environmental parameters such as water chemistry and biota. It is therefore helpful to have additional data available from specific geographic regions to insure that herbicide residues will be adequately diminished when basing aquatic weed control recommendations for irrigation supplies on EPA labeled water-use restrictions.

The purpose of this study was to measure the persistence of diquat, endothall, and fluridone in typical irrigation source ponds, located mainly in the Piedmont plateau of North Carolina, to increase knowledge of their persistence in waters of this geographic region.

MATERIALS AND METHODS

Diquat[3,4], endothall[5] or fluridone[6] were applied as whole pond treatments in the Piedmont plateau, Coastal plain, and Sandhills regions of North Carolina and Virginia (USA) at various rates during May 1984 (Table 1). Diquat and endothall were applied to separate ponds in sufficient quantity for calculated final concentrations of 1 and 2 ppm, respectively. Fluridone was applied at a rate of 2.27 kg ai/ha to Apex Pond and 1.14 kg ai/ha to Virginia Pond and Whispering Pines Pond. All ponds had negligible water exchange. Water temperatures (surface) in all ponds was 19°C at the time of treatment. All liquid formulations were applied as surface sprays with no adjuvants. Pelleted fluridone was applied with a centrifugal applicator.

Water samples were collected from diquat treated ponds at 6, 12, 24, 36, 56, and 60 h, and 3, 4, 7, and 16 days after treatment (DAT); from endothall treated ponds at 6, 12, 18, 24, 30, 42 h, and 2, 3, 4, 7, and 21 DAT; and from fluridone treated ponds at various time intervals between 1 and 62 DAT. One-liter composite water column samples were collected by slowly lowering a weighted bottle between the surface and bottom, at three established sample stations in each pond. Aliquats of 333 ml from each sample station were then combined for a single composite sample from each pond at each sample time. All samples were stored frozen in opaque plastic bottles until analyses were conducted.

Analytica Laboratories[7] conducted diquat analyses by Method RM-5-5, Chevron Chemical Company[8]. This procedure has a detection limit of 3 ppb and a precision of 11 ppb at a concentration of 300 ppb.

Pennwalt Corporation[9] conducted endothall analyses

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[3]Use of trade names does not imply endorsement by North Carolina Agricultural Research Service of the products named or criticism of similar products not mentioned.

[4]Diquat Herbicide—H/A, Chevron Chemical Co., Ortho Ag. Chem. Div., San Francisco, CA.

[5]Aquathol K, Ag Chem Division, Pennwalt Corp., Philadelphia, PA.

[6]Sonar 5P, Sonar 4AS, Elanco Products Co., Indianapolis, IN.

[7]Analytica Laboratories, Inc., Sioux Falls, SD.

[8]Chevron Chemical Co., Ortho ag. Chem. Div., San Francisco, CA.

[9]Ag Chem Division, Pennwalt Corp., Residue Analysis Section, Tacoma, WA.

TABLE 1. DATES OF HERBICIDE APPLICATIONS AND LOCATIONS OF PONDS USED TO STUDY THE PERSISTENCE OF DIQUAT, ENDOTHALL AND FLURIDONE IN WATER.

Pond	Herbicide	Application Date	County, State	Geographic Region
Jenks	diquat (2L)	5/01/84	Wake, NC	Piedmont
Smith	diquat (2L)	5/01/85	Wake, NC	Piedmont
Rogers	diquat (2L)	5/01/84	Wake, NC	Piedmont
Apex	fluridone (4AS)	5/24/84	Wake, NC	Piedmont
Whispering Pines	fluridone (4AS)	5/09/84	Moore, NC	Sandhills
Virginia	fluridone (5P)	5/10/84	Southampton, VA	Coastal Plain
Mangum-A	endothall (3L)	5/20/84	Wake, NC	Piedmont
Mangum-B	endothall (3L)	5/20/84	Wake, NC	Piedmont

by gas chromatography. The samples were analyzed using two different detectors—an electrolytic conductivity detector and thermionic specific detector, both used for detection of N in the endothall derivative. Both provided sensitivity to 10.0 ppb at an efficiency of 85%. The values obtained by the different detectors compared favorably.

Fluridone analysis was conducted in our laboratory using method AM-AA-CA-Roll-CA-Roll-AC-755 Lilly Research Laboratories[10]. The method permits the determination of approximately 1 ppb of fluridone in water.

Herbicide dissipation rates were estimated as the slopes of best-fit regression equations. Slopes of regression lines among ponds, within herbicide treatments were compared using the Statistical Analysis System (SAS)[11].

RESULTS AND DISCUSSION

Diquat dissipated very quickly from all treated ponds (Figure 1). Statistical analysis showed that the rates of dissipation were significantly greater ($p = .05$) with increasing initial concentrations (although the instantaneous initial concentrations are not known, we assume that these are proportional to the first measured concentrations, i.e. 6 h). The regression equations, however, predict very similar lengths of time to reach zero concentration of diquat in these ponds. These were 33 h in Smith Pond and Rogers Pond and 30 h in Jenks Pond.

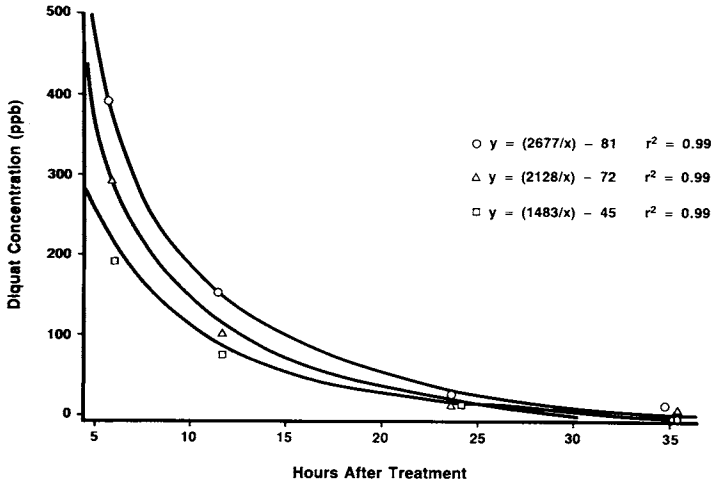

Figure 1. Dissipation of diquat from three ponds located in the Piedmont plateau of North Carolina.

The time required for diquat to dissipate below detection limits in other pond studies ranged from 8 days (detection limit = 0.1 ppm (Coats *et al.* 1964) to 38 days (Grezenda *et al.* 1966). The initial concentrations in these two studies were 0.4 ppm and 2.5 ppm, respectively, which may partially explain the contrasting observations. The initial concentrations in the ponds in our study (calculated) were approximately 1.0 ppm. Therefore, the rapid disappearance of diquat in this study cannot be attributed to to lower initial concentrations; but can probably be explained by rapid absorption by fairly dense infestations of filamentous algae (*Pithophora oedogoniana* [Mont.] Wittr.) and watermeal (*Wolffia columbiana Kars.*), rapid adsorption to the high clay particulate content that is characteristic of Piedmont impoundments and photolysis. Since rainfall did not occur during the sampling period, dissipation can be assumed dilution free. Diquat was not detected in any of the ponds between the 36 h sampling and the 16 day sampling suggesting that release back into the water from stressed or decaying vegetation did not occur.

The high rate at which diquat concentration declined in this study suggest the wide margin of safety for its use in irrigation supply water in Piedmont ponds.

Data from the two endothall treated ponds were pooled because regression analysis revealed no significant difference ($p = .01$) between the individual pond responses (Figure 2). Endothall concentration decreased linearly with time after treatment. It can be predicted by the regression equation that the concentration of endothall in the ponds was about 1.0 ppm 14 days after treatment, or the time at which the label would allow use of the water for irrigation. Since turfgrasses and crop plants such as squash, peppers and tomatoes have been irrigated with water containing 5.0-6.0 ppm endothall without phytotoxic effects[12], a wide margin of safety exists for using endothall for aquatics weed control in Piedmont ponds used as irrigation sources.

Endothall was non-persistent in water of ponds treated in this study. After 21 days the measured concentration of endothall in water of the ponds declined to less than 25% of the initial concentration; and it can be predicted by the regression equation that the endothall concentration

[10]Lilly Research Laboratories, Greenfield Laboratories, Greenfield, IN.

[11]SAS Institute Inc., Cary, NC.

[12]Pennwalt Corp. 1984. Review of the effects of endothall products on aquatic ecosystems.

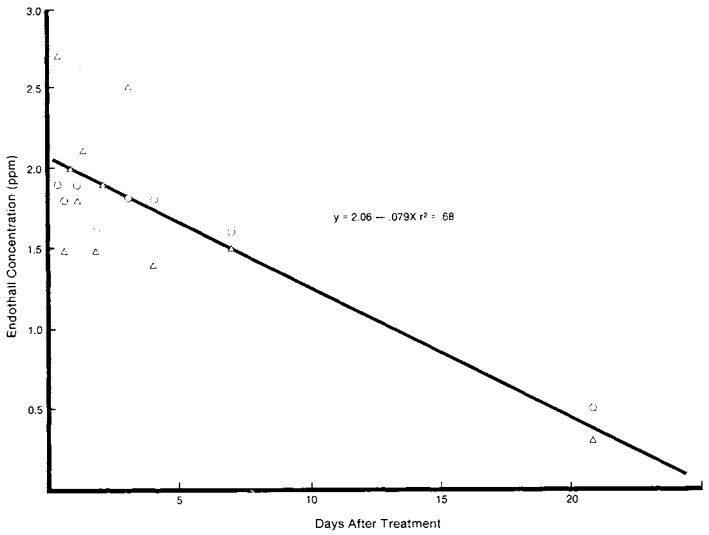

Figure 2. Dissipation of endothall from two ponds located in the Piedmont plateau of North Carolina.

would approach zero 26 DAT. When ponds were treated at similar initial concentrations in other studies, endothall declined to non-detectable concentrations in 25[13] to 36 days (Sikka, 1973). The rate at which endothall concentration declined in Piedmont ponds is therefore similar to its behavior in other geographic regions.

Fluridone was more persistent in water than either diquat or endothall. The two ponds that were treated with the liquid formulation of fluridone showed constant dissipation with very slight unexplained variability. Regression analysis indicated that greater than 90% of the variability of fluridone concentration could be explained by DAT; and the regression equations can be used to predict zero concentration of fluridone after 64 days in Apex Pond and 69 days in Whispering Pines Pond (Figure 3). These data agree with a previous report in which the time to reach no detectable residue levels (1.0-5.0 g/L) were between 2 months and 12 months (West *et al.* 1983).

Fluridone concentrations responded somewhat differently in the pond treated with the 5P formulation. Maximum fluridone concentration was not observed until 17 DAT (Table 2). This response was expected and reflects the time required for fluridone to dissociate from the clay carrier. After this, fluridone steadily decined in concentration until 51 DAT when an increase in fluridone concentration was observed. A similar increase in concentration is suggested in the Whispering Pines Pond between 24 and 36 DAT (Figure 3). Although the time sequence is somewhat different, both of these increases in concentration may reflect release of fluridone back into the water from stressed vegetation. Both Whispering Pines and Virginia Pond were heavily vegetated with proliferating spikerush (*Eleocharis baldwinii* (Torr.) Choap. whereas Apex Pond had little vegetation and a similar increase in concentration was not observed.

Regression analysis revealed no significant decrease (p = .05) in fluridone concentration over the 53-day sampling

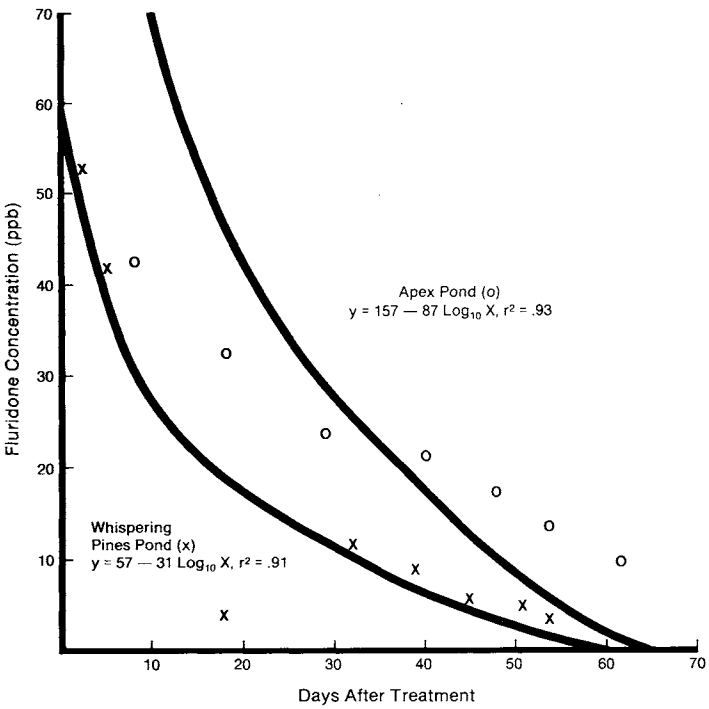

Figure 3. Dissipation of fluridone from two ponds located in the Sandhills region and Piedmont plateau of North Carolina. Values of 170 and 128 ppm for 1 and 3 days, respectively, in Whispering Pines Pond are included in the statistical analysis but are not included in the figure.

period in the 5P (Virginia Pond) treated pond compared to the declines observed in the 4AS treated ponds. Variability in fluridone persistence in enclosed ponds can be explained by environmental factors that effect photolysis of the compound (West *et al,* 1983). In Virginia Pond a major factor was probably the heavy shading over the pond and shading from spikerush in the water that reduced photolysis. Both Apex Pond and Whispering Pines Pond are relatively unprotected from sunlight.

Light rainfall occurred several times during the sampling period of the fluridone treated ponds, and one heavy precipitation event (c.a. 6 in) occurred. However, since these events could not be detected in the data, and regression equations were "good fit" (r^2 = 0.92 and 0.91), dilution effects were probably negligible.

TABLE 2. CONCENTRATIONS OF FLURIDONE IN THE WATER OF A VIRGINIA (USA) POND AFTER APPLICATION OF SONAR 5P. CHANGES IN CONCENTRATION ARE INSIGNIFICANT WITH RESPECT TO DAYS AFTER TREATMENT (p = .05).

Days after Treatment	Fluridone Concentration (ppb)
1	8.1
3	9.4
17	44.4
23	37.7
31	26.9
38	22.9
44	20.9
51	28.9
53	28.9

[13]Pennwalt Corp. Unpublished data.

CONCLUSION

A 14-day waiting period is required before diquat or endothall (greater than 0.50 ppm and less than 4.25 ppm) treated water can be used for irrigation. Concentrations of these herbicides declined to below detectable limits or below concentrations known to be phytotoxic to crops, within 14 days after application to irrigation source ponds located mainly in the Piedmont plateau of North Carolina. Recommendations can, therefore, be made with confidence for using the compounds for aquatic weed control in this geographic region.

At the time of writing this manuscript, registration of fluridone as an aquatic herbicide is under consideration by the EPA, and water use restrictions will not be known until the herbicide label is available. Based upon the moderate persistence of fluridone observed in this study we recommend caution when applying fluridone to enclosed irrigation source ponds until more information is available pertaining to phytotoxicity of crops to low concentrations of fluridone in irrigation water.

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