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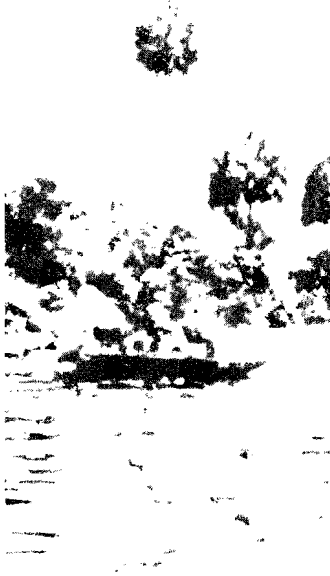
EVALUATION OF ENDOTHALL/ADJUVANT
MIXTURES IN FLOWING WATER

by

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<p>Four endothall/adjuvant formulations (inverts Asgrow 403 and I'vod and polymers Nalquatic and Poly Control) were evaluated for endothall release profiles and control of Eurasian watermilfoil (<i>Myriophyllum spicatum</i> L.) at flow velocities of 1.5 and 3 cm/sec. Endothall release profiles from the adjuvant formulations were compared with release profiles from conventional, liquid endothall formulations.</p> <p>All of the endothall/adjuvant formulations released endothall for longer periods than did the conventional endothall formulation at the 1.5-cm/sec flow velocity. The polymer formulations released endothall for longer periods (72 to 84 min posttreatment) than the invert formulations (36 to 48 min posttreatment). At the 3-cm/sec flow velocity, endothall residues were below detection (0.01 mg/l) by 12 min posttreatment when using all of the</p> <p style="text-align: right;">(Continued)</p>					
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endothall formulations. In comparison, endothall release profiles were 12 to 132 min shorter than 2,4-dichlorophenoxyacetic acid profiles obtained from a previous study using identical adjuvants and flow velocities.

The extended release profiles of the polymers Nalquatic and Poly Control make these adjuvants the most promising of those tested for use with endothall in stands of Eurasian watermilfoil where flow velocities are <3 cm/sec.

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PREFACE

This study was conducted by personnel of the US Army Engineer Waterways Experiment Station (WES) as part of the US Army Corps of Engineers Aquatic Plant Control Research Program (APCRP). Funds were provided by the Office, Chief of Engineers (OCE, DAEN-CW), under Department of the Army Appropriation No. 96X3122 Construction General, and by the US Army Engineer District, Baltimore, under Document No. E868480397, August 1984. Mr. E. Carl Brown, OCE, was Technical Monitor.

This study is being conducted in three phases. Phase I evaluated the release characteristics of hydrophobic herbicide/adjuvant mixtures. Phase II (reported herein) evaluated the release characteristics of hydrophilic herbicide/adjuvant mixtures. Phase III will evaluate the potential role of controlled-release herbicide formulations.

The principal investigators for this work were Drs. Kurt D. Getsinger and Howard E. Westerdahl of the Aquatic Processes and Effects Group (APEG), Ecosystem Research and Simulation Division (ERSD), Environmental Laboratory (EL). They were assisted by Dr. Troy Stewart, Mr. Ed Wilkerson, and Mmes. Dawn Meeks and Nancy Craft of the APEG.

The work was initiated in May 1985 under the general supervision of Dr. John Harrison, Chief, EL, and Mr. Donald L. Robey, Chief, ERSD, and under the direct supervision of Dr. Thomas L. Hart, Chief, APEG. Mr. J. Lewis Decell was the Program Manager for the APCRP.

The Tennessee Valley Authority (TVA) Aquatic Research Laboratory (ARL), under the direct supervision of Mr. Billy G. Isom, provided facilities and technical assistance under a cooperative agreement with WES. Technical expertise and assistance were provided by Messrs. Dan Haraway, Jeff Longacre, Jay Griffith, and Greg Harrison and Ms. Rachel Hean of ARL. Personnel of the Fisheries and Ecology Branch, TVA, under the direct supervision of Mr. A. Leon Bates, assisted in field collection of plant material. The Laboratory Branch, TVA, provided results of endothall residue analysis under a cooperative agreement with WES. Chemicals used in this study were provided by The Asgrow Florida Company, JLB International Chemical, Nalco Chemical Company, and Pennwalt Corporation.

Technical reviews of this report were provided by Dr. Kien Luu and Mr. Reed Green, APEG, and Dr. Bill Zattau, Environmental Resources Division,

EL. The report was edited by Ms. Jessica S. Ruff of the WES Information Products Division.

Director of WES was COL Dwayne G. Lee, CE. Technical Director was Dr. Robert W. Whalin.

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EVALUATION OF ENDOTHALL/ADJUVANT MIXTURES IN FLOWING WATER

PART I: INTRODUCTION

Background

1. The development of herbicide formulations and application techniques to control nuisance submersed plants in flowing water is a major challenge for aquatic weed scientists and managers. The herbicide concentration and contact time needed to control submersed plants are not easily obtained in flowing water because the water column is continuously moving, while herbicides released into the water are transported downstream. One area of research and development in flowing-water environments has focused on the use of herbicide/adjuvant combinations. Adjuvants, such as inverting oils and polymers, are blended with liquid herbicides to create viscous formulations that sink and cling to submersed vegetation. In general, inverting oils consist of hydrophilic, water-soluble, polar "heads" and lipophilic, water-insoluble, nonpolar "tails." Commercially available inverting oils are produced by blending various proportions of oils and selected emulsifiers, while polymers are produced by blending together selected polymeric materials and emulsifiers. The specific types and proportions of compounds used in the adjuvant manufacturing process are proprietary information and differ with each manufacturer.

2. In theory, the adjuvant holds the herbicide in the vicinity of the target plant, increasing herbicide contact time. Therefore, a herbicide/adjuvant formulation should be more efficacious than a simple, liquid herbicide formulation, particularly in flowing-water environments. Previous work (Silver, Mansell, and Illingworth 1974) suggests that herbicides, when combined with adjuvants, are released into the aqueous phase and subsequently taken up uniformly by the target plants. The hydrophilicity of a herbicide may determine its rate of release from the adjuvant mixture, following placement of the mixture in a polar medium, such as water. For example, endothall (7-oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid), a hydrophilic herbicide, would be released more rapidly from an adjuvant than 2,4-D (2,4-dichlorophenoxyacetic acid), a hydrophobic herbicide.

Purpose

3. The purpose of this three-phase study is to determine which adjuvant, conventional, and controlled-release herbicide formulations can be used effectively for the control of submersed plants in flowing-water environments. Phase I (Getsinger and Westerdahl 1986) evaluated the release characteristics of hydrophobic herbicide/adjuvant mixtures, using the dimethylamine formulation of 2,4-D. Phase II, reported in this document, evaluated the release characteristics of a hydrophilic herbicide, using the dipotassium salt of endothall. Phase III will evaluate the potential role of controlled-release herbicide formulations.

Objectives

4. The objectives of Phase II were to:
- a. Determine the release profiles of endothall from selected endothall/adjuvant mixtures in flowing water.
 - b. Compare these results with release profiles from conventional, liquid endothall formulations.
 - c. Recommend the best adjuvants for use with endothall in flowing water.

PART II: MATERIALS AND METHODS

Flume System

5. Herbicide/adjuvant experiments were conducted in two outdoor hydraulic channels (~110 m long × 4 m wide × 2 m deep) located at the Tennessee Valley Authority Aquatic Research Laboratory (TVA/ARL), Brown's Ferry, Ala. The channels were modified to contain a series of subchannels (~7 m long × 1 m wide × 1 m deep) that were used for duplicate treatments of each formulation (Figure 1). Apical shoots (~15 cm long) of Eurasian watermilfoil (*Myriophyllum spicatum* L.), collected in nearby Lake Wheeler, were planted, several centimeters deep, in the bottom of the channels in a mud sediment, capped with 5 cm of washed sand. Shoots were bundled in groups of three and planted 5 to 10 cm apart to produce stands ~3 m long × 0.8 m wide, consisting of >1,000 shoots each. Plant stands were allowed to grow for 4 weeks to a height of ~70 cm.

Flow Velocity Measurements

6. Flow velocities were measured with a Model 201 Marsh-McBirney Flowmeter (accuracy ±2 percent). The sensor was attached to a platform that enabled measurements to be taken on a horizontal plane. Water depth was held at 70 cm, and a constant incoming flow velocity of 1.5 or 3 cm/sec was maintained during all experimental runs. Flow velocities were characterized in each channel by taking measurements (5 cm subsurface, middepth, and 5 cm above the bottom) at the upstream edge, center, and downstream edge of the plant stands.

Mixtures and Application Techniques

7. The chemicals used in this study are listed in Table 1. Endothall, a contact herbicide, is registered for use in slow-moving water and is effective in controlling Eurasian watermilfoil. Adjuvants used in this study include inverting oils and polymers compatible with endothall and commonly used for aquatic plant control. Commercially available inverting oils consist of various proportions of oils and selected emulsifiers. When inverting oils

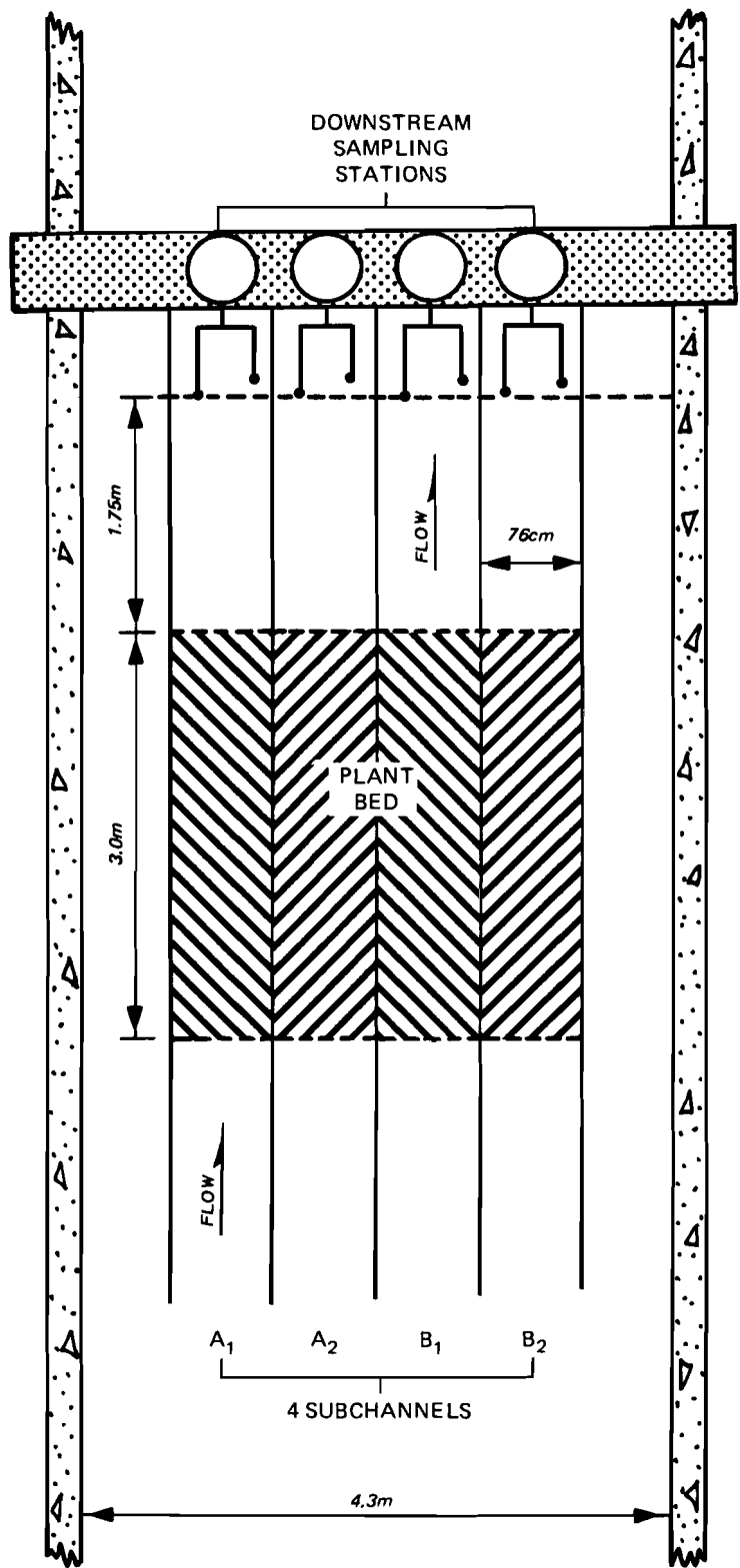


Figure 1. Overhead view of flume channels

Table 1
Chemical Compounds Used in Flowing Water Study

Compound	Active Ingredient	Manufacturer
Aquathol K	Dipotassium salt of endothall (40.3%)	Agchem Division- Pennwalt Corporation
Asgrow 403 invert emulsifier	Water-in-oil emulsifiers and selected solvents 100%	Asgrow Florida Company
I'vod inverting oil	d'Limonene [d-1,8(9)-p- menthadiene] 50%, plus selected emulsifiers 50%	JLB International Chemical, Inc.
Nalquatic	Polycarboxylate polymer 30%	Nalco Chemical Company
Poly Control	Polyacrylamide copolymer 30%	JLB International Chemical, Inc.

are mixed with water in an appropriate fashion, an invert is formed. An invert consists of a dispersion of water droplets in oil (e.g., water surrounded by oil or a water-in-oil emulsion) and is designed to function as a sticking and drift control agent. Polymers are anionic or nonionic compounds that are blended with selected emulsifiers and designed to function as sinking, sticking, confinement, and drift control agents. The exact chemical structures and proportions of components used in the production of inverting oils and polymers are considered proprietary information and are, therefore, unavailable for publication.

8. Inverting oils were blended with water and endothall to form a thick, mayonnaiselike invert material using a 7:1 (water-to-inverting oil) ratio. Polymers were blended with water and endothall, using a 2.5-percent polymer, to form a thick, mucuslike material. All herbicide formulations were prepared to provide an endothall treatment rate of 5 mg active ingredient (ai)/ℓ. These formulations were transferred to a spray system consisting of a pressurized paint pot, hose, and spray wand with a multiple-hole, fan-type nozzle. These mixtures were injected below the water surface, throughout the plant stands, at 1.36 atmospheric (20 psi).

Sample Collection and Residue Analysis

9. Water samples were collected in the center of each flume channel, 175 cm downstream from the plant stands, using an ISCO Model 2100 automatic water sampler adapted to sample a water column depth of 10 to 60 cm. Discrete, 100-ml samples were collected every 2 min posttreatment and composited to give a 600-ml sample, representing a 12-min interval. This procedure was continued for 3 hr posttreatment. Water samples were also collected 5 m upstream from each plant stand every hour during experimental runs to monitor possible herbicide contamination.

10. Samples were analyzed for endothall residues by the Analytical Method for the Determination of Endothall, No. 180.293 (Pesticide Analytical Manual II) (US Food and Drug Administration 1973). Endothall recovery from samples was 84 percent, based on percent recovery of spiked water samples. Endothall analyses were performed by Industrial Laboratories Company, Denver, Colo., under contract to the TVA Laboratory Branch, Chattanooga, Tenn.

11. Residue data reported in this study represent the mean of two composited samples (one from each channel) for each specific time interval.

PART III: RESULTS

Adjuvant Performance

12. The inverting oils, Asgrow 403 and I'vod, formed thick, mayonnaise-like mixtures when blended with water and endothall at the recommended proportions. Following application, the invert formulations adhered readily to the leaves and stems of the target plants. The amount of invert "flakes" that floated to the surface was minimal due to the subsurface application technique. Invert "flakes" were observed on the plants up to 30 hr posttreatment.

13. The thick, mucuslike polymer formulations sank rapidly and adhered to the target plants in the form of long strings or large droplets. Remnants of the polymer formulation were visible on the plants for only 4 to 6 hr posttreatment.

Endothall Release Rates

14. Endothall residues from all of the formulations tested were measurable above the detection limit (0.1 mg/l) up to 84 min posttreatment when herbicide mixtures were applied at flow velocities of 1.5 cm/sec. Endothall concentrations fell below detection, however, by 24 min posttreatment when using the conventional, liquid formulation of Aquathol K (Figure 2). The use of invert formulations resulted in endothall release profiles of 36 min posttreatment with Asgrow 403 (Figure 3) and 48 min posttreatment with I'vod (Figure 4). The highest endothall concentration (0.045 mg/l at 24 min posttreatment) was measured using the invert formulation Asgrow 403.

15. The longest herbicide release profiles found in this study were achieved with the polymer formulations (Figures 5 and 6), as endothall concentrations fell below detection at 72 min with Nalquatic and at 84 min with Poly Control. The highest endothall concentration measured using the polymer formulations was 0.035 mg/l, with Nalquatic at 24 min posttreatment.

16. In contrast, endothall residues from all of the formulations tested were below detection at 12 min posttreatment when herbicide mixtures were applied at flow velocities of 3 cm/sec.

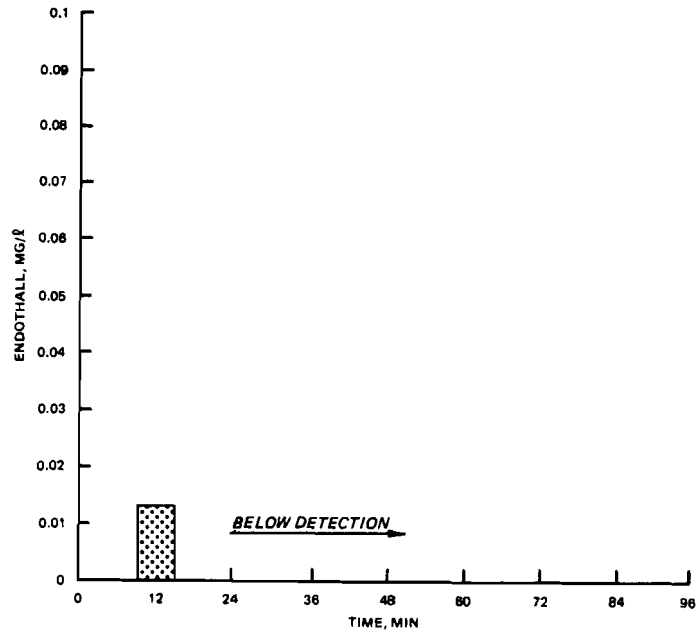


Figure 2. Effect of time on endothall residues 1.75 m downstream of plant stands using a conventional, liquid formulation of Aquathol K at a flow velocity of 1.5 cm/sec

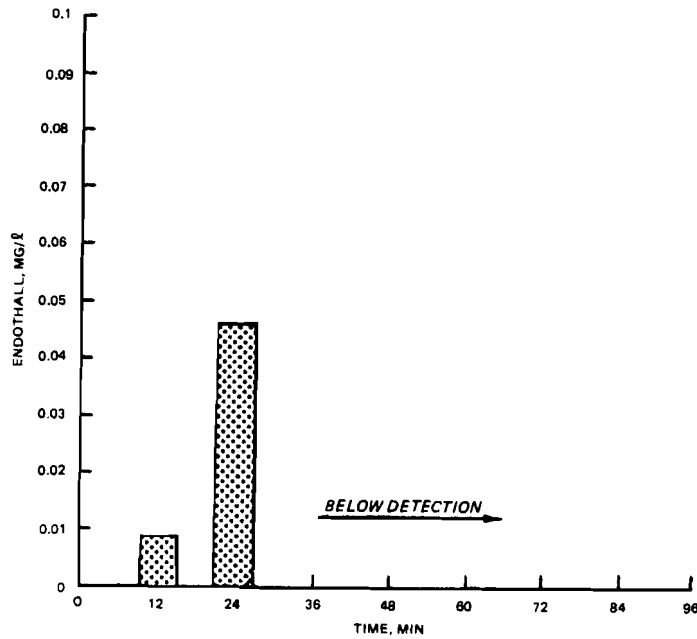


Figure 3. Effect of time on endothall residues 1.75 m downstream of plant stands using a formulation of Asgrow 403/endothall at a flow velocity of 1.5 cm/sec

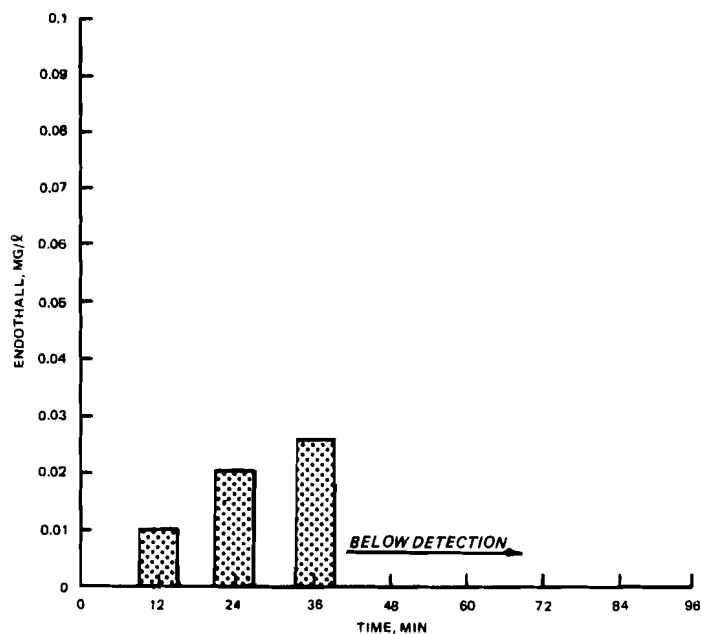


Figure 4. Effect of time on endothall residues 1.75 m downstream of plant stands using a formulation of I'vod/endothall at a flow velocity of 1.5 cm/sec

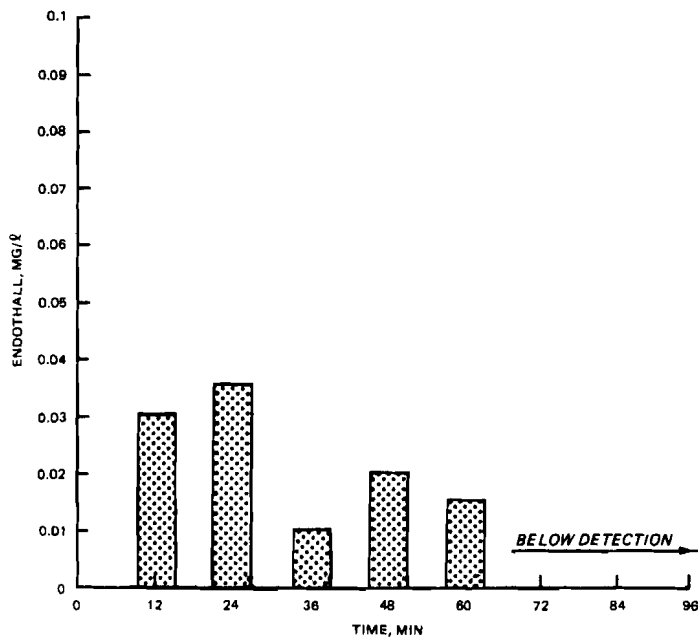


Figure 5. Effect of time on endothall residues 1.75 m downstream of plant stands using a formulation of Nalquatic/endothall at a flow velocity of 1.5 cm/sec

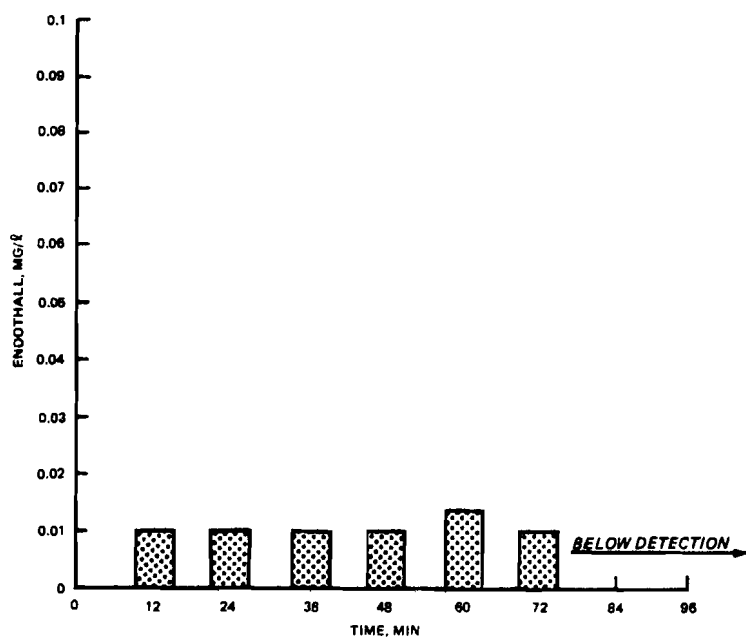


Figure 6. Effect of time on endothall residues 1.75 m downstream of plant stands using a formulation of Poly Control/endothall at a flow velocity of 1.5 cm/sec

17. Since the experiments were terminated at posttreatment 3 hr, plant efficacy was not evaluated. Water samples collected 5 m upstream from each plant stand during experimental runs showed no herbicide contamination.

PART IV: DISCUSSION

Sample Collection

18. The ISCO water sampler is designed to remove water from the flow stream at approximately 30 cm/sec to prevent particles from settling out in the sampling line. This results in an effective withdrawal zone larger than the intake orifice at flow velocities <30 cm/sec. Consequently, theoretical values of herbicide concentration downstream (Getsinger and Westerdahl 1986) will be less than the measured concentration at low water velocities. As streamflow velocity increases (approximating 30 cm/sec), the herbicide concentration in the water samples obtained by the ISCO sampler should approximate theoretical estimates, assuming the herbicide is conservative. State-of-the-art sampling equipment and techniques preclude isokinetic water sampling for herbicide analysis from open-channel, flowing water (<30 cm/sec) environments. As a result, sampling the water column 175 cm downstream of the treated plants was necessary since the sampling equipment would disrupt the hydraulics of the plant stand and sample herbicide/adjuvant "floc" on the plants within the treatment area. This condition would result in higher than actual herbicide concentrations being measured in the water column.

19. The calculated herbicide transport time from the treatment area to the sampling point was 2 and 4 min at the tested flow velocities of 3 and 1.5 cm/sec, respectively (Getsinger and Westerdahl 1986). Consequently, herbicide concentrations in the water samples approximated the concentrations in the aqueous phase within the treatment area with a 2- to 4-min delay. Sampling water at 2-min intervals posttreatment for 180 min and compositing to represent 12-min periods per sample was selected to show the average herbicide residue concentration in the water over this time period.

20. Changes in herbicide release from the adjuvant and other factors affecting herbicide residue levels in water, e.g., uptake and adsorption, occurring within the 2- to 4-min period would be reflected in the water sample. However, it must be realized that the sampling technique and compositing of samples made it impossible to infer the dynamics of the herbicide levels within the 12-min interval. Due to limitations of the sampling technique, determining a mass balance for the herbicide was inappropriate in assessing herbicide concentration per unit time.

21. For most aquatic herbicides a contact time of >12 min is necessary for the control of submersed species. Therefore, a 12-min composite sample was considered reasonable for comparing relative changes in herbicide concentration over time. Cost constraints associated with herbicide residue analysis in water samples further warranted the compositing of samples.

Endothall Residues

22. In theory, adjuvants that release herbicides for extended time periods should be more efficacious than adjuvants that release herbicides for short time periods, provided that a lethal dose of herbicide is maintained for the duration of the exposure period. Therefore, efficacy is obtained by some combination of herbicide concentration and exposure time. Although the tested polymer formulations released endothall for >60 min posttreatment, the concentrations were very low (no higher than 0.04 mg/l and the exposure times were short (<84 min).

23. Price (1969) reported that an endothall concentration of 3 mg/l was required, for a 3-hr period, to control several *Potamogeton* species in irrigation canals. Using an exposure time of 3 hr in a static assay, endothall (5 mg/l) reduced elodea (*Elodea canadensis* L.) biomass by only 26 percent (Bowmer and Smith 1984). These same studies showed that injection of endothall in flowing water channels (5 to 10 mg/l, for 2 to 3 hr) resulted in poor control of elodea. Van and Conant (in preparation) reported that, in laboratory experiments, a 6- to 12-hr contact time with a concentration of 5 mg/l endothall was required to achieve a 73- to 80-percent reduction of plant biomass in young hydrilla (*Hydrilla verticillata* (L.F.) Royle) shoots. When the endothall concentration was reduced to 1 mg/l, a contact time of 48 to 96 hr was required to achieve a 72- to 82-percent reduction in hydrilla shoots.

24. Results of an endothall concentration/exposure time study conducted on Eurasian watermilfoil at Pat Mayse Lake, Texas, showed that an endothall concentration of 0.6 mg/l would provide 95-percent control of Eurasian watermilfoil, if that concentration could be maintained for at least 8 hr.* The concentration/exposure time requirement reported by Rodgers far exceeds the endothall dosage and contact time determined in this study.

* Personal Communication, 1987, J. H. Rodgers, Institute of Applied Sciences, North Texas State University, Denton, Tex.

25. Since a direct determination of efficacy was not obtained in this study, results from a future endothall concentration/exposure time study on Eurasian watermilfoil will be needed to correlate plant death with endothall dosage and contact time. Efficacy determinations can only be inferred with this type of comparison. The relatively small size of the submersed plant stands (3 m in length) used in the herbicide/adjuvant flowing-water studies limits the direct determination of efficacy. Some riverine studies have shown that control of submersed species occurred >20 m downstream from the treated area, rather than in the actual zone of herbicide application.* Future flowing-water studies should be conducted on larger plant stands (20 to 30 m in length) to directly assess efficacy.

26. Phase I of the herbicide/adjuvant flowing-water studies evaluated the release profiles of 2,4-D from adjuvant formulations (Getsinger and Westerdahl 1986). The experimental design of the 2,4-D study (including target plants, adjuvants, application, and sampling techniques) was identical to the experimental design of the endothall study. A comparison of herbicide residues from the 2,4-D and endothall studies showed that the release profiles of 2,4-D were considerably longer than the release profiles of endothall, for all of the adjuvants tested at 1.5- and 3-cm/sec flow velocities. For example, at a flow velocity of 1.5 cm/sec, the release profile of 2,4-D was 180 min for both I'vod and Poly Control, compared to an endothall release profile of 48 min for I'vod and 84 min for Poly Control. This relatively rapid release of endothall from the adjuvant mixtures, compared with 2,4-D, is most likely due to its degree of hydrophilicity.

27. Based on results from the endothall/adjuvant evaluations, Nalquatic and Poly Control show the greatest potential as effective adjuvants for the release of endothall in flowing water, where velocities within the plant stands are <3 cm/sec. Studies have shown that velocities <3 cm/sec within submersed plant stands in field situations are not unusual (Wenninger 1986; Getsinger, in preparation).

28. Finally, results from Phases I and II of the herbicide/adjuvant evaluations show that inverts and polymers, when properly mixed with compatible herbicides and correctly applied to submersed plant stands, will

* Personal Communication, 1986, J. Clayton, Ministry of Agriculture and Fisheries, Ruakura Soil and Plant Research Station, Hamilton, New Zealand.

aid in the placement of herbicides on target vegetation (i.e., the formulations will sink and adhere to plants). However, this advantage in formulation placement must be tempered with evidence that adjuvant formulations may not provide the necessary herbicide contact time to control submersed aquatic plants in flowing-water environments.

PART V: CONCLUSIONS AND RECOMMENDATIONS

Conclusions

29. Results of Phase II of the herbicide/adjuvant studies show that at flow velocities of <3 cm/sec within submersed plant stands, the use of selected endothall/adjuvant formulations will provide longer herbicide release profiles compared with a conventional, liquid endothall formulation. However, none of the endothall/adjuvant formulations may provide a sufficient herbicide exposure time for acceptable control of Eurasian watermilfoil in flowing water.

30. At flow velocities of ≥ 3 cm/sec, there was no apparent difference between the endothall release profiles of the adjuvant formulations and the conventional formulation. Of the adjuvants tested, the herbicide release profiles from the polymers Nalquatic and Poly Control make them the most promising adjuvants for use with endothall or other hydrophilic herbicides in flowing water to control submersed plants. In addition, results from this study suggest that hydrophilic herbicides (e.g., endothall) are released more rapidly from adjuvants in flowing water systems than are hydrophobic herbicides (e.g., 2,4-D).

Recommendations

31. The recommendations suggested by the Phase II work include the following:

- a. None of the adjuvants tested are recommended at the present time for use with endothall to control Eurasian watermilfoil in flowing water at velocities >3 cm/sec within the plant stands.
- b. A better understanding of flow velocities within plant stands is necessary before selecting the appropriate herbicide formulation or herbicide/adjuvant combination.
- c. The most promising adjuvants for use in flowing water, as identified in Phases I and II, are I'vod and Poly Control. These adjuvants should be evaluated in experimental systems that more accurately simulate field conditions, e.g., flow velocities >3 cm/sec (up to 30 cm/sec) and plant stands >20 m in length.
- d. Techniques should be developed to apply herbicides in the immediate vicinity of plant stands in flowing water, taking

advantage of lower flow velocities found within the stands that maximize herbicide contact time.

- e. More information is needed on the chemical characteristics of adjuvants for a better understanding of the behavior of herbicide/adjuvant mixtures in flowing water.
- f. Herbicide concentration/exposure time studies should be continued to identify the most probable effective combinations at various flow velocities.

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