



US Army Corps  
of Engineers  
Waterways Experiment  
Station

# Aquatic Plant Control Research Program

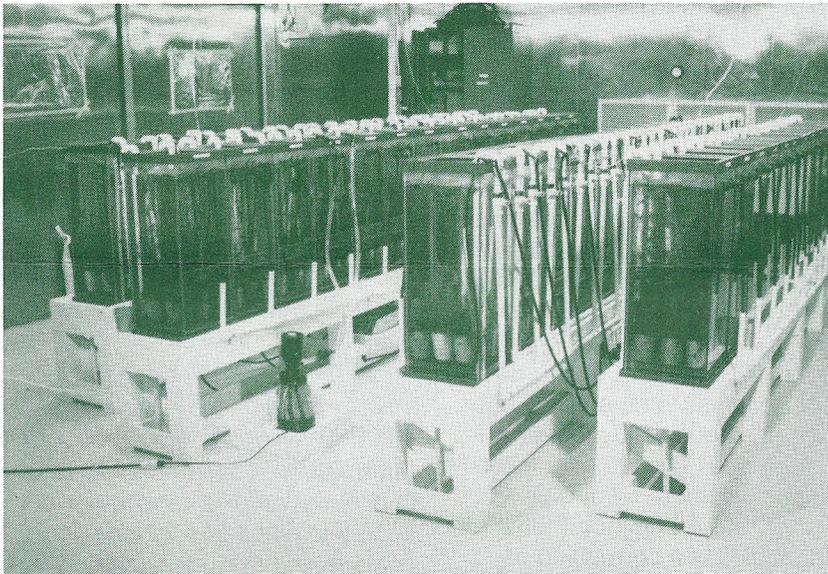
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## The improvement of aquatic herbicide delivery systems

by

Michael D. Netherland and Kurt D. Getsinger



Walk-in controlled-environment chamber at the US Army Engineer Waterways Experiment Station used for herbicide concentration/exposure time studies

**T**he key for successfully controlling submersed plants with chemicals in high water-exchange environments is in maintaining an adequate herbicide exposure period. Undoubtedly, the lack of adequate herbicide contact time has contributed to the widespread inconsistency of controlling submersed plants in flowing water. This situation has led researchers to study the possibility of control-

ling the release of herbicides in water to lengthen the contact time. One approach for extending contact time is to develop a controlled-release (CR) carrier or matrix.

A CR matrix is defined as a pesticide active ingredient (such as an insecticide, herbicide, or plant growth regulator) combined with an inert carrier (for example, polymer, lignin, or clay). Such a system is designed to deliver the

active ingredient to the target organism at a controlled rate for a specified period of time. In addition to providing long-term control, CR formulations could maintain herbicide concentrations in close proximity to the target plant, increase the efficacy and longevity of the herbicide by protecting it from environmental degradation, minimize herbicide residues available to the environment, and decrease application costs since less frequent applications would be required.

Van and Steward (1985), Dunn and others (1988), Steward and Nelson (1972), and other researchers have demonstrated that herbicide efficacy could be improved by increasing the length of time that a herbicide was released from various matrices. However, production problems, field application difficulties, inconsistent release profiles, and lack of optimal release rate information contributed to a general failure to promote further research in this area. New information generated from laboratory studies under the Aquatic Plant Control Research Program (APCRP) at the US Army Engineer Waterways Experiment Station (WES) and cooperative field studies by the WES; the US Army Engineer District, Jacksonville; and the University of Florida in flowing-water systems has



sparked a renewed interest in developing and applying CR technology.

A promising CR technology has emerged in the area of aquatic insect control. A gypsum-based product has been developed to release the mosquito larvicide Altosid (methoprene) for up to 60 days (Genereux and Genereux 1985). The ability of this matrix to release insecticides in an aquatic environment over an extended period of time and the approval of operational field use of this matrix by the US Environmental Protection Agency (EPA) is encouraging. Other matrices recently developed for CR pesticide use include protein colloids (Controlled Release Systems Research, Inc.), currently used to slowly release insecticides in aquatic environments, and starch encapsulation (Schreiber, White, and Shasha 1987), currently used to slowly release the herbicide trifluralin in row crops.

A need clearly exists to develop herbicide formulations and delivery techniques that will extend chemical contact time to improve the control of submersed vegetation in flowing-water systems. To address this need, a work unit entitled Herbicide Delivery Systems was initiated in 1989 under the APCRP. The objectives of this work unit are to develop and evaluate delivery systems that will maximize herbicide contact time against submersed macrophytes within a treatment area. This article gives background information

on the development of herbicide concentration/exposure time relationships—a prerequisite for developing CR formulations—and information on previously developed and evaluated aquatic CR matrices. The article also describes a research approach, outlining the development and evaluation of improved herbicide delivery systems.

### Herbicide concentration/ exposure time relationships

Information that defines the concentration/exposure time relationships and critical tissue burden levels of each herbicide against specific target plants is vital for the successful development of CR matrices. These relationships are being developed for hydrilla (*Hydrilla verticillata* Royle) and Eurasian watermilfoil (*Myriophyllum spicatum*) at WES using registered aquatic herbicides and herbicides currently labeled under Experimental Use Permits. Relationships have been developed for fluridone versus hydrilla (Hall, Westerdahl, and Stewart 1984, Van and Conant 1988), diquat versus hydrilla (Van and Conant 1988), 2,4-D versus milfoil (Green and Westerdahl 1990), endothall versus milfoil and hydrilla (Netherland, Green, and Getsinger 1991), and triclopyr versus milfoil (Netherland and Getsinger 1992). A common theme of these studies is a correlation between herbicide concentration and exposure time, and injury of the target species. These results enable the quantification of contact time required for different concentrations of

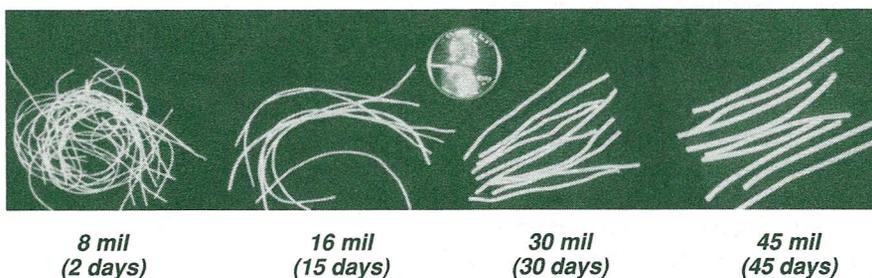
herbicides to achieve control of target plants.

In the field, liquid herbicides are often applied in flowing water or in large systems where rapid dispersion of the herbicide from the treatment area can occur. This rapid dispersion has been demonstrated in several large systems where submersed plant stands have been treated with liquid dye or dye in combination with herbicides (Fox, Haller, and Getsinger 1990, Getsinger, Green, and Westerdahl 1990). Off-target movement of herbicides can result in contact times that are insufficient to provide acceptable plant control.

Further, dye studies conducted in plant stands in static systems indicate that thermal stratification may prevent thorough mixing of herbicides through the water column in the treatment area (Getsinger, Haller, and Fox 1990). The warm, herbicide-laden upper layers of water can be isolated from the remainder of the water column and, thus, are susceptible to off-target movement under windy conditions. Spot treatments of small plant infestations along lake and reservoir shorelines are also subject to rapid dispersion and, therefore, short contact times.

Information from dye studies and concentration/exposure time work suggests that a lack of chemical contact time may be responsible for the failure of many of these types of herbicide treatments. The practice of increasing contact time has been used with 2,4-D and endothall granules, where spot treatments using these formulations help diminish rapid dispersion rates. While granular spot-treatment techniques may be superior to liquid applications, there is still a need for improving the herbicide release rate in granular formulations to maximize contact time.

Systemic herbicides, such as fluridone, 2,4-D, triclopyr, and bensulfuron methyl, require longer exposure periods than



Controlled-release fluridone fibers with herbicide release shown in days

contact herbicides, such as diquat or endothall. The advantage of using systemic herbicides is their ability to translocate throughout the plant, potentially providing complete kill of the target plant and thus long-term control. A properly designed delivery system could keep herbicides within treatment areas for extended periods, improving efficacy and allowing for less active ingredient to be used (that is, lower concentrations of active ingredient over longer exposure times).

In addition to herbicide concentration/exposure time relationships, internal tissue burden information for specific herbicides and target plants would be valuable for the development of CR matrices. Research by Haller and Sutton (1973), Van and Steward (1985), and Reinert and others (1985) has shown that with many aquatic herbicides, a relatively small proportion of the herbicide in the water is taken up by the plant. For example, Van and Steward found that the residue of fluridone in hydrilla tissue 21 days after treatment was equivalent to approximately 3 percent of the total amount of herbicide applied. The authors concluded that the slow uptake of fluridone by hydrilla suggests that the herbicide must remain in contact with the plant for a comparatively long time before fluridone is accumulated in the plant tissue at levels sufficient to achieve control. Haller and Sutton observed that approximately 6 percent of the total  $^{14}\text{C}$ -endothall incorporated was taken up by hydrilla after two days posttreatment under laboratory conditions. The authors suggested that the slow initial uptake of endothall might present a problem in controlling hydrilla in flowing water. Similarly, Reinert found that residues in milfoil accounted for only 1 to 5 percent of the endothall used during treatment in a mesocosm study.

Results of residue analyses of water sampled during concentration/



**Research ponds at the Lewisville Aquatic Ecosystem Research Facility, Lewisville, Texas**

exposure time experiments, conducted at the WES, also indicated that very little herbicide (2,4-D, fluridone, endothall, and triclopyr) was taken up by the plant tissue during the exposure period. Herbicide residue levels of water measured immediately after treatment showed little variance from herbicide residues measured following exposure times (0.25 to 21 days posttreatment). These studies showed that concentrations of herbicide within the plant are quite small when compared to treatment concentrations, and any improvement in increasing the level of herbicide within plant tissues should greatly improve efficacy.

### **Development of controlled-release matrices**

The concept of using CR systems to chemically control aquatic pests is not new. Early development of slow-release matrices for controlling aquatic insects and mollusks concentrated on the use of carrier materials such as cement briquettes (Evans and Fink 1960, Barnes and Webb 1968), rubber (Cardarelli, Senderling, and Wuerzer 1967, Shultz and Webb 1969), and plastics (Whitlaw and Evans 1968, Nelson and others 1970). Several types of CR herbicide matrices have also been developed and evaluated during the past two decades (Steward and Nelson

1972, Harris, Norris and Post 1973, Cardarelli and Raddick 1983, and Connick and others 1984).

In the late 1970s, a work unit was initiated in the APCRP to develop and evaluate CR herbicide formulations for controlling milfoil and hydrilla. Two CR formulations of 2,4-D were tested in the laboratory and the field. These formulations consisted of 2,4-D acid in Kraft lignin pellets (Westvaco, Inc.) and an acrylic polymer, glycidyl methacrylate (Poly GMA), plus 2,4-D impregnated in clay pellets (Wright State University).

Subsequent testing revealed that herbicide release rates from two generations of the Poly GMA formulation were relatively constant and provided a slow release of 2,4-D, under controlled static conditions for up to six months (Van and Steward 1982, 1983). Testing of the Kraft lignin formulation provided a 2,4-D release for approximately 2 months under static conditions, but high initial washout of the herbicide occurred by 21 days posttreatment (Van and Steward 1982). When field-tested against milfoil in Lake Seminole, Georgia, both the Poly (GMA) and Kraft lignin formulations failed to provide long-term 2,4-D concentrations in the water column, but these formulations did provide up to five months control of the target

plant (Hoepfel and Westerdahl 1982). Major problems associated with scale-up procedures to produce large quantities of the Poly (GMA) 2,4-D formulation limited the practical use of that CR matrix (Harris 1984). A third 2,4-D formulation, 14-ACE-B, a natural rubber elastomer combined with 2,4-D butoxyethanol ester (Creative Biology Laboratory, Inc.), was field-tested in Lake Seminole in 1983 (Getsinger and Westerdahl 1984). The 14-ACE-B formulation was ineffective in governing the slow release of 2,4-D, with most of the herbicide released into the water column during the first few days following treatment.

In the mid-1980s a fibrous CR system consisting of polycaprolactone (PCL) was developed at the Southern Research Institute, Birmingham, Alabama, for delivering the herbicides diquat and fluridone (Dunn and others 1988). Based on laboratory release rate profiles, it was determined that the fluridone/PCL CR matrix should be further evaluated in the field. Consequently, these fluridone fibers were applied to stands of

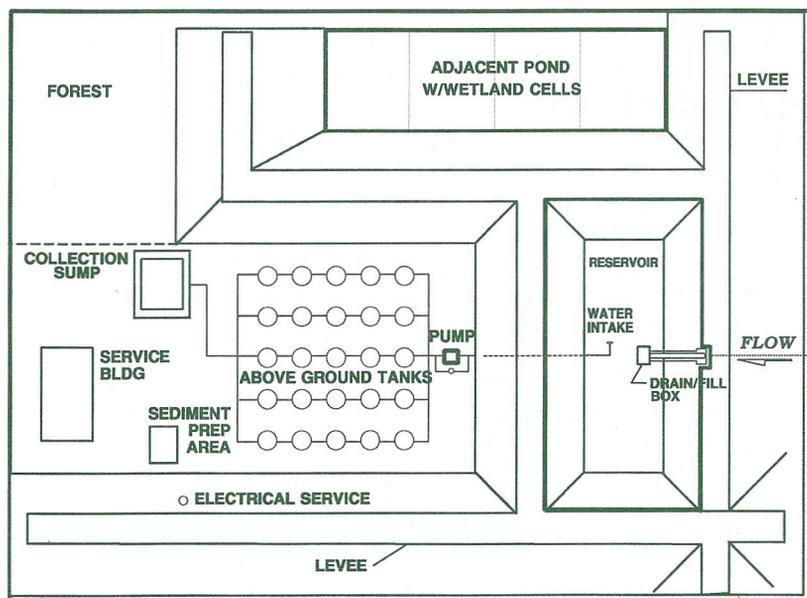
hydrilla (Toledo Bend Reservoir, Texas, and Lateral 28 drainage canal, Florida) and milfoil (Pend Oreille River and Winchester Wasteway, Washington), but resulted in poor to moderate control of target plants (Westerdahl, Hall, and Getsinger 1984).

As previously mentioned, materials such as cement, rubber, and plastics were used as inert components in the early development of CR matrices. These materials were generally nonbiodegradable and, therefore, were not considered good prospects for commercial use. Current development of CR matrices for aquatic insect control, however, have used matrices that will readily degrade in the environment. For example, a new technology using a gypsum-based ( $\text{CaSO}_4$ ) matrix has emerged as a leading CR technology in aquatic insect control. This product, incorporated with methoprene, has been successfully patented, licensed, and marketed by Zoecon Corporation as a mosquito larvicide. The gypsum matrix has EPA approval for use with the insecticides methoprene and

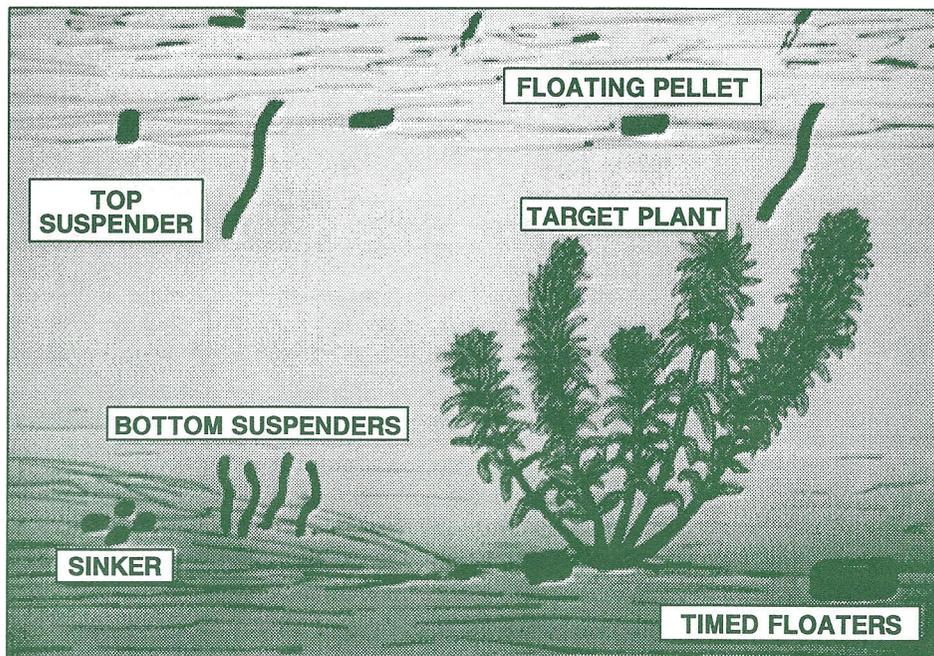
temephos, and release rates of these active ingredients from the matrix can range from 30 to 150 days. Another emerging CR technology in aquatic insect control (Controlled-Release Systems Research, Inc.) involves the use of a protein colloid matrix for the slow release of methoprene. This product is currently under review by the EPA for commercial use. The proven slow-release characteristics and environmental compatibility of these two matrices make them excellent candidates for CR testing with aquatic herbicides.

Evidence of slow herbicide uptake rates by target plants and relatively low concentrations of active ingredients found within tissues of dead or dying plants suggest that only a fraction of the herbicide applied to a treatment area is actually required to provide effective control. Although treatment areas have been overdosed (in a physiological sense, not in violation of product labels) using conventional application techniques and formulations (that is, applying phytotoxic concentrations of the active ingredient to the entire water volume), this overdosing has been required to produce acceptable efficacy. Developing systems designed to deliver herbicides to submersed plants in the most efficient manner (for example, a CR matrix) offers the potential of using less active ingredient to achieve greater efficacy. This process ultimately translates into lower treatment costs and better environmental compatibility.

A strong concern for the protection and conservation of natural resources, particularly wetlands, has recently developed. This public awareness has stimulated a demand for reduced loading of pesticides into the environment. Pesticide manufacturers are responding to this demand by developing new lines of chemistry with target-specific modes of action and low active ingredient dose



**Schematic of herbicide evaluation mesocosm facility under construction at the Lewisville Aquatic Ecosystem Research Facility**



requirements. The recent accumulation of herbicide concentration/exposure time and tissue burden information, advances in slow-release pesticide delivery systems, and the trend for reducing pesticide loading into the environment (via low-dose active ingredients) has manifested the need to develop improved herbicide delivery systems for controlling submersed aquatic plants.

### Research approach

Improved herbicide delivery systems will be developed and evaluated at the WES over the next several years, with initial efforts focusing on CR matrices. The approach described in the following paragraphs will be used to conduct the CR studies.

Environmentally compatible CR delivery systems will be identified and selected for evaluation. Emphasis will be placed on matrices that have shown the ability to slow-release pesticides (for example, insecticides and insect growth regulators) in an aquatic environment. Consideration will also be given to slow-release pesticide formulations currently used in agriculture and other plant science disciplines.

Once identified, matrices will be formulated with a variety of aquatic herbicides (for example, fluridone, triclopyr, bensulfuron methyl, 2,4-D, diquat, and endothall) to test for compatibility between the matrix and the herbicide active ingredient.

Successfully formulated herbicide matrices will be evaluated in the laboratory, under controlled-environment conditions, to determine whether controlled release is feasible with the matrix being tested. Release rates of the newly developed CR formulations will be compared with release rates of conventional granular carriers. Improvement in release rates of the new CR formulations must be demonstrated prior to further evaluation.

Once CR matrices have proven compatible with herbicides and are releasing at the designated rate, release rate profiles will be designed for each herbicide and target plant. Some systemic herbicides may require a matrix that releases over a period of 30 to 60 days, while contact-type herbicides may require release of the active ingredient over a period of 1 to 3 days. Information developed from

concentration/exposure time relationship and internal tissue burden studies at WES will be used to design release rate profiles for individual herbicides.

Matrices that release herbicides within design specifications will be further evaluated for efficacy against Eurasian watermilfoil and hydrilla in small-scale, controlled-environment studies at WES. Following these laboratory experiments, release rate profiles and efficacy determinations will be conducted using the most promising CR matrices in mesocosm and pond studies at the Lewisville Aquatic Ecosystem Research Facility in Lewisville, Texas. Herbicide residues will be determined in water, sediment and plant tissue during these studies. Determination of water residues will validate laboratory studies which have defined the contact time required for a given herbicide concentration to ensure optimal plant control. Sediment residues will identify the proportion of herbicide available for root uptake. The proposed plant tissue sampling will determine the threshold level (or tissue burden) of herbicide necessary to

ensure optimal plant control. In addition to defining use patterns for the CR matrices, large-scale studies will also aid in determining environmental effects on herbicide release rates.

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Dr. Kurt D. Getsinger is a research biologist in the Environmental Laboratory, US Army Engineer Waterways Experiment Station. He leads the Chemical Control Technology Team and his expertise includes management of nuisance aquatic vegetation using herbicides and plant growth regulators. Dr. Getsinger holds a Bachelor of Science degree in Biology from Campbell University, a Master of Science degree in Biology from East Carolina University, and a Ph.D. in Plant Physiology from Clemson.



Michael D. Netherland is a research biologist for the Chemical Control Technology Team in the Environmental Laboratory, US Army Engineer Waterways Experiment Station. His research focuses on the effects of herbicides and plant growth regulators on nuisance submersed aquatic vegetation. Michael has a Bachelor of Science degree in Biology from Cameron University and a Master of Science degree in Botany from Purdue.

To control submersed aquatic plants with chemicals in high water-exchange environments, adequate herbicide exposure periods are required. One approach for extending the exposure period is development of a controlled-release (CR) carrier or matrix. New information from the Aquatic Plant Control Research Program has sparked a new interest in developing and applying CR technology, following research in the 1970s and 1980s. This issue provides information on development of herbicide concentration / exposure time relationships and information on previously developed aquatic CR matrices. A research approach for developing and evaluating improved herbicide delivery systems is also described.



## AQUATIC PLANT CONTROL RESEARCH PROGRAM

This bulletin is published in accordance with AR 25-30 as one of the information dissemination functions of the Environmental Laboratory of the Waterways Experiment Station. It is principally intended to be a forum whereby information pertaining to and resulting from the Corps of Engineers' nationwide Aquatic Plant Control Research Program (APCRP) can be rapidly and widely disseminated to Corps District and Division offices and other Federal and State agencies, universities, research institutes, corporations, and individuals. Contributions are solicited, but should be relevant to the management of aquatic plants, which are, providing tools and techniques for the control of problem aquatic plant infestations in the Nation's waterways. These management methods must be effective, economical, and environmentally compatible. The contents of this bulletin are not to be used for advertising, publication, or promotional purposes. Citation of trade names does not constitute an official endorsement or approval of the use of such products. This bulletin will be issued on an irregular basis as dictated by the quantity and importance of information to be disseminated. Communications are welcomed and should be addressed to the Environmental Laboratory, ATTN: J.L. Decell, US Army Engineer Waterways Experiment Station, 3909 Halls Ferry Road, Vicksburg, MS 39180-6199, or call AC 601/634-3494.

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