FIELD EFFICACY AND AVAILABILITY, MOVEMENT, AND PERSISTENCE OF ICIA-0051 HERBICIDE IN SOILS

by

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(ABSTRACT)

Fields studies conducted in 1987 and 1988 determined the weed control efficacy of ICIA-0051 and SC-0774 in conventional and no-till systems of corn (*Zea mays* L.) culture. Results of the preemergence and postemergence applications of ICIA-0051, across all treatments after 8 weeks, showed 85% control or better of triazine-resistant smooth pigweed (*Amaranthus hybridus* L.), while fall panicum (*Panicum dichotomiflorum* Michx.) control ranged from 43 to 87%. Giant ragweed (*Ambrosia trifida* L.) control ranged from 30 to 95%, while control of ivyleaf morningglory (*Ipomoea hederacea* (L.) Jacq.) was below 75% in the preemergence treatments and ranged from 89 to 99% in the postemergence treatments. In general, the addition of atrazine to the pre- and postemergence treatments of ICIA-0051 improved weed control. SC-0774 treatments gave 85% or better control of fall panicum, but inadequate broadleaf weed control (75% or worse).

Soil mobility studies using soil thin-layer chromatography and soil leaching columns indicated that the movement of ICIA-0051 was highly negatively correlated with the organic/humic matter fraction. Although the mobility

patterns were similar, ICIA-0051 was more mobile than SC-0774, which was more mobile than atrazine. A comparison of ICIA-0051 across soils indicated that the order of mobility was Appling loamy sand (Rf = 6.4) > Davidson clay (Rf = 5.6) > Bojac sandy loam (Rf = 5.0) = Frederick silt loam (Rf = 4.9) > Hyde silty clay loam (Rf = 1.1). Other soil properties such as the clay content and pH were not strongly correlated with ICIA-0051 movement.

Results of the adsorption/desorption studies indicated that the organic/humic matter fraction was primarily responsible for the binding and retention of ICIA-0051 across the five soils investigated. Based on the K constants derived from the Freundlich equation, the order of adsorption was Hyde > Frederick > Davidson = Bojac > Appling. The desorption results indicated that ICIA-0051 was not tightly bound to the soil particles, with losses between 20 and 50% of the amount adsorbed after two desorptions.

Results of the greenhouse persistence study, using mustard (Brassica kaber L.) as a bioassay species, indicated that ICIA-0051 was more biologically available than atrazine. Similar to the adsorption and leaching results, the persistence of ICIA-0051 was highly positively correlated with the soils' organic matter. Regardless of the rate used, crop injury decreased over time, although the highest rate (1 ppm of ICIA-0051) showed significant crop injury even after 6 months in several soils in the greenhouse studies.

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I. LITERATURE REVIEW

Soil and Herbicide Properties that Influence Chemical Availability, Movement, and Persistence

Once a herbicide is applied to the plant or soil, various processes can occur that will determine the fate of the herbicide. Herbicide losses are often associated with soil or herbicidal properties and various environmental degradation processes. Some specific degradation processes that occur are biological degradation (involving a living organism), chemical decomposition (involving a chemical process), and photodecomposition (degradation by radiant sunlight) (48). Generally the fate of a herbicide involves the interaction of several of these processes, making the herbicide less active, or in some cases, more active (1, 14). These processes and their interactions ultimately affect the availability, movement, and persistence of the herbicide in the environment.

The above degradation processes are related to various transfer reactions which determine the movement of the herbicide in the environment (1, 29, 48). One of these processes involves the absorption and/or exudation of the herbicide by either plants or animals (1, 34). Herbicides can penetrate the hydrophilic and hydrophobic tissues of organisms and then undergo degradation (1, 12, 34). After the herbicide enters the plant or animal, the parent compound or metabolite can be exuded back into the environment (34). The second process involves the retention of the herbicide by the plant vegetation on or in the harvested product (1, 34, 48). Another process is soil adsorption/desorption to/from soil colloids or organic matter (2, 14, 15, 23, 38, 47). This basically involves the uptake of the herbicide from the liquid phase to the soil surface. The soil colloidal properties and other soil properties, such as pH, greatly influence the herbicide's adsorption or release.

Volatilization may also affect dissipation of the herbicide; in this process, the herbicide is transferred from the liquid or solid phase to a vapor phase (1, 34, 48). Here the potential for herbicide loss is correlated with its vapor pressure, soil properties, and other environmental conditions.

Herbicide loss can also be caused by runoff, whereby the herbicide moves horizontally along with and due to runoff losses of water and/or soil sediments (45, 48). Runoff losses are influenced by such factors as rainfall amounts and intensity, soil texture, herbicide properties, and type of herbicide application.

Finally, the herbicide may move in several directions through the soil profile. Vertical and lateral herbicide movement is usually caused by the percolation of water through the soil profile and is often referred to as leaching. Herbicide movement is related to soil properties such as pore space, and various herbicide properties such as organic matter and clay content (5). Herbicides can be carried laterally by the underground aquifers, or occasionally, they move upward in the soil profile by capillary action.

This dissertation deals with understanding the various interactions between the soil and a herbicide, which thereby determines its fate and usage for weed control in the environment. To understand these soil and herbicide interactions it is essential to study the availability, mobility, and persistence of a herbicide in various soil types. However, due to the various complex properties of the soil and herbicides, understanding their relationships one to another is often a very difficult process. Nevertheless, when the various constituents of the soil and herbicides are broken down by their key components, it is easier to understand their interactions.

There are numerous properties of the soil that go beyond the focus of this review; however, the key soil factors that influence the fate of the herbicide in the soil are: soil colloidal properties such as the clay type or content and organic matter, pH, moisture, and temperature (1, 5, 34).

The soil colloids play one of the most important roles in determining the herbicide's fate in the environment. Of the soil colloids, organic matter generally has a greater influence on herbicide adsorption than does clay (3, 15, 34, 46). It has been estimated that a 1% increase in the soil organic matter content (e.g. 2 to 3%) imparts a five- to ten-fold increase in herbicide adsorption versus a similar 1% increase in the clay content (e.g. 30 to 31%) (34). However, depending on the soil texture, both the clay and organic fractions must be considered. Both the organic and clay fractions have a high reaction potential created by their high surface to volume ratio and net negative soil charge (15, 34).

Both the clay and the organic matter fractions of the soil influence the negative charge of the soil (5, 15). This net negative charge on the soil plays a significant role in the herbicide's availability, movement, and persistence. The soil's negative charge is caused by the decomposition of organic matter and the isomorphic substitution of the clays (5). The soil charge within the clay fraction is associated with SiOH and AlOH groups, while carboxyl (COOH) and hydroxyl (OH) groups are associated with the organic fraction (5).

The soil's reaction or pH is controlled by the hydrogen ions adsorbed on soil particles that disassociate into the soil solution (5, 15). The two primary cations responsible for the soil acidity are ionic hydrogen and aluminum. Soil acidity is created by the reaction of Al^{3+} or aluminum hydroxy ions with water, thus giving off hydrogen ions (5, 15). Excess hydrogen concentration in the soil solution can also be caused by the decomposition of organic matter, exchange of other cations such as Ca^{2+} on the clay micelle, fertilizers additions, root exchange, or dissolved CO_2 which forms carbonic acid (5).

The organic fraction has a higher cation exchange capacity (CEC) than the clay fraction (1, 5, 34). The CEC and base saturation, which is the relative proportion of the adsorbed cations on the colloidal complex, are good indicators of the soil's ability to exchange ions and thus influence the pH of the soil. Lowering the percent base saturation makes the soil more acidic (5). The CEC for organic matter typically ranges from 100 to 300 meq/100 g; whereas the CEC of kaolinite clay, typical of southeastern soils, ranges from 3 to 15 meq/100 g; and montmorillonite, typical of midwestern soils, ranges from 80 to 150 meq/100 g (5, 34). Vermiculite clay has the highest CEC of the clays at 100 to 150 meq/100 g, and illite clay has a CEC of 10 to 40 meq/100 g.

The soil pH plays an important role in the herbicide's fate. Soil pH determines the charge of the herbicide and whether the herbicide is ionized or not. The ionization of a basic or acidic herbicide determines its activity and persistence (34).

Soil moisture influences the herbicide's fate by competing with the adsorption sites on the soil colloids (34). Herbicide losses generally increase with increasing soil moisture. Some herbicides due to their high water solubility are more sensitive to soil moisture levels (34).

The soil temperature influences the soil microbe population and volatilization of the herbicides (1, 34). Compounds with high vapor pressures are more likely to volatilize. Soil temperature affects the environment for soil microbes. Soil microbes are responsible for herbicide degradation through various chemical reactions such as oxidation, reduction, and hydrolysis (1, 34). These reactions also occur in the soil solution as well.

Several key properties of herbicides that affect their behavior in the soil, listed generally in descending order of their significance, are: 1) ionization, 2) water solubility, 3) volatility, 4) functional groups present, 5) molecular size, and 6) stability (46, 47). The actual importance of each of these herbicide properties varies depending on the specific herbicide or class.

Herbicide ionization is a pH-dependent reaction which determines the overall charge of the molecule (34). The charge associated with the herbicide is either positive, negative, or no charge (nonionic). The degree of ionization of a herbicide depends on the pKa of the compound (4). The higher the pKa, the stronger the base, and likewise the lower the pKa, the stronger the acid[1]. When the pH equals the pKa for that particular herbicide, 50% of the molecules are in the ionic form and 50% in the nonionic form.

The cationic herbicides such as paraquat (1,1'-dimethyl-4,4'-bipyridinium ion) are strong bases and are completely ionized in aqueous solutions (34, 48). Because of their strong net positive charge, they are bound tightly to clay particles and soil organic matter and thus have no soil activity or mobility (46).

The basic herbicides, which include the triazines such as atrazine [6-chloro-N-ethyl-N'-(1-methylethyl)-1,3,5-triazine-2,4-diamine], are pH-dependent and become ionized under low pH conditions and are thus bound to soil colloids (38, 46). This binding usually increases the herbicide's persistence and potential for crop injury, depending on the availability of the herbicide for plant uptake (34, 46). Under neutral or alkaline soil conditions, the basic herbicides are nonionized and tend to be more mobile in the soil (34, 38). Although more of the herbicide is available for weed uptake under neutral or alkaline conditions, this also increases the potential for groundwater contamination.

The acidic herbicides, which include the phenoxy compounds such as 2,4-D [(2,4-dichlorophenoxy)acetic acid], are also pH-dependent and are ionized to their anionic form under high pH conditions (34). These anionic molecules are repelled by the net negative charge of the soil and tend to be more mobile and less persistent. Under neutral or acid conditions these herbicides are in their nonionized form and their persistence and mobility are dependent on other properties of the herbicide (34).

The nonionic herbicides, which include the phenylureas such as diuron [*N*'-(3,4-dichlorophenyl)-*N*,*N*-dimethylurea] and the dinitroanilines such as trifluralin [2,6-dinitro-*N*,*N*-dipropyl-4-(trifluoromethyl)benzenamine] are not pH-dependent and do not develop a net overall charge. Their mobility is more dependent on other chemical properties (4).

The second important property that influences a herbicide's fate in the environment is the compound's water solubility. The herbicide's affinity for a soil particle is determined by its hydrophilic and hydrophobic properties (1, 5, 34). Polar groups on the herbicide molecule form hydrogen bonds with water, increasing its water solubility (5, 34). Herbicides are classified by their water solubility as follows: high water solubility, 1000 ppm or greater (e.g. phenoxy compounds), moderate solubility, 100 to 1000 ppm (e.g. many of the acetanilides), and low solubility, 100 ppm or less (e.g. most triazines and dinitroanilines) (47). In general, compounds which are readily ionized or have polar functional groups are more water soluble than nonionized compounds with limited polar groups. Certain functional groups of the herbicide, such as alkyl or nitro groups, generally decrease water solubility (4). The influence of halogen groups such as Cl^- on the molecule's water solubility varies, depending on the herbicide. Generally, increasing halogenation of acids (e.g. 2,4-D) increases their water solubility, while increasing halogenation of basic (e.g. triazines) and nonionic (e.g. phenylureas) herbicides decreases their water solubility (4).

The herbicide's potential to volatilize is another important factor determining its fate in the soil. Herbicides are also classified according to their vapor pressures as follows: high potential-greater than 10^{-4} mm Hg (e.g. thiocarbamates), moderate potential-10^{-5} to 10^{-4} mm Hg (e.g. acetanilides), low potential-less than 10^{-5} mm Hg (triazines) (34). Herbicides often need to be soil-incorporated, due to their potential to volatilize from the soil surface (e.g. thiocarbamates) or photodecompose (e.g. dinitroanilines) (1, 34). Generally, ionic compounds are less volatile than nonionic compounds (4).

The fourth factor that influences the herbicide's fate in the environment is the functional groups associated with the molecule. Certain functional groups such as phosphates and arsenates can complex with clay minerals and make the herbicide immobile (4). Glyphosate [N-phosphonomethyl) glycine] is an example of a herbicide that has phosphate groups which complex with the soil. Other functional groups such as nitro, amino, hydroxyl, and carboxyl groups bind to humic or organic matter, thus regulating the herbicide's movement in the soil (5). The soil humus, is a portion of the organic matter that is more decomposed and stable, and is generally considered to be the organic fraction of the soil associated with herbicide binding (5).

The fifth factor determining the fate of a herbicide in the soil is its molecular size. As a general rule, when all other factors are equal, increasing the molecular size decreases the diffusion and movement of the compound in the soil (4). Assuming the compounds are dissolved in the soil solution, smaller molecules diffuse more rapidly and percolate easier throughout the small pores than larger molecules.

The final property of a herbicide that determines its fate in the soil is the compound's chemical stability (1, 12, 34). Since chemicals are subjected to various soil degradation processes, their persistence or fate is determined by their resistance to these processes. The soil half-life values for most herbicides have been calculated and are a good indication of the compounds' soil stability. Soil half-life is correlated with the various herbicide properties previously mentioned, such as water solubility and ionization. Examples of the soil longevity of some commonly used herbicides for corn (*Zea mays* L.) are: alachlor [2-chloro-*N*-(2,6-diethylphenyl)-*N*-(methoxymethyl) acetamide], 2-4 months; atrazine, 6-12 months; and 2,4-D, 1 month (34).

Techniques Used in Soil/Herbicide Interaction Studies

Various research techniques have been used to determine the availability, mobility, and persistence of herbicides in soils. Some of these techniques include soil column leaching and bioassay analysis, soil thin-layer chromatography, adsorption/desorption analysis, and/or recropping persistence studies (13, 18, 19, 22, 23, 38, 45, 46, 49, 51). All of these techniques are well established and widely accepted, and were used in the laboratory and greenhouse sections of this dissertation.

Soil column leaching studies and soil thin-layer chromatography analysis are used to determine the relative mobility of an experimental herbicide in a soil. With soil leaching columns, water is used as a leachate for the herbicide, and the herbicide's downward movement is influenced by the flow rate (saturated or unsaturated) and total amount of water applied (45). After the soil leaching columns have been treated, they are split vertically or the soil removed from the various soil sections and a sensitive bioassay species is planted to detect potential herbicide levels. The properties of the soil and herbicide, as discussed earlier, are important factors in the herbicide's mobility.

Soil thin-layer chromatography requires the use of radiolabeled compounds and occurs under saturated flow as the herbicide moves up the soil plate (13, 49). The general procedure involves applying the herbicide to the bottom of the soil plates, and then using water as carrier to move the herbicide. After the plates are dry, autoradiography is used to detect the herbicide movement in the soil and Rf values can be calculated.

Work published by Helling (13) and others (49, 51) indicates that the soil thin-layer techniques are an excellent method for determining herbicide mobility, although this technique has some limitations. These limitations include the frequent lack of available radiolabeled isotopes and the difficulty in working with heavy textured soils caused by the soil cracking on the chromatography plates.

Researchers have shown numerous influences of, and between, the soil type and pH which affect the relative mobility of the herbicide (13, 15, 22, 25, 51). Depending on the soil type, the Rf values for atrazine movement have ranged from 3.5 to 8.9 (1 to 10 scale) (13).

Both the soil leaching columns and the soil thin-layer techniques allow the researcher to quantify the relative movement of a herbicide in a given soil. Given the resources (radiolabeled compounds) the soil thin-layer technique is usually a less time consuming process, however results obtained from the use of the two approaches are often comparable (51). One advantage of the soil column leaching technique over soil thin-layer chromatography is that, by using a standard curve, one can better quantify the actual herbicide concentration throughout the soil profile.

Soil adsorption/desorption analysis measures the amount of the herbicide in the soil solution. Radiolabeled herbicides are used, and through liquid scintillation quantitation, the amount of the herbicide binding to the soil is determined

(49, 51). Desorption extractions are used to determine the amount and rate of herbicide release. Using the Freundlich equation, the data collected are used to construct adsorption isotherms and make correlations between soil properties and herbicide availability (49). Most herbicides follow an L-type isotherm, which implies an initial high affinity between the soil and solute, followed by a lesser affinity as the concentration increases (46).

One approach to measuring herbicide persistence is to use various rates of a herbicide in a soil and measure its effects on a sensitive crop species over time. By recropping these pots over time, the loss of the compound can be monitored, as well as its potential to carry over to other sensitive crop species. This technique can be employed to measure herbicide loss due to degradation and other modes of dissipation over time.

Previous Work with ICIA-0051 and Other Similar Compounds

Corn remains one of the primary acreage (U.S., 71 million acres in 1981) and cash crops produced in the U.S (34). Although many economic problems face producers, one of the major concerns is the adequate control of weeds. To combat this problem, many effective herbicides have been developed over the years, although several weed species are still not adequately controlled. Problem weeds still not adequately controlled in corn included triazine-resistant smooth pigweed

(Amaranthus hybridus L.) and johnsongrass (Sorghum halepense L. Pers.).

Due to ICI Americas' purchase of Stauffer Chemical Company, ICI has obtained several new experimental compounds which they are developing for weed control in several crops. Based on initial field testing by company and university researchers, this new class of chemistry appears to have excellent activity on a number of problem weeds in corn. Although many chemical analogs have been tested, the most promising compound is ICIA(SC)-0051 (chemistry not released). Currently the major research emphasis is on field testing involving weed efficacy studies and crop tolerance; consequently, little is known about the properties of these materials in soil. For this reason, the major emphasis of this dissertation has involved investigating the soil factors which influence the availability, movement, and persistence of ICIA-0051 in the soil. This research was divided into three major aspects of study: field, greenhouse, and laboratory work with ICIA-0051.

Because ICIA-0051 has been tested for only the past four years at the university level, few publications exist, although several abstracts of field studies can be found (7-11, 16, 17, 20-28, 30-33, 35-37, 43, 44). Most of the reports are on ICIA-0051, SC-0774, and SC-0735, which apparently have similar structures (although the chemistry has not been disclosed), show similar crop selectivity, and control similar spectrums of weeds. Corn, sorghum (Sorghum bicolor (L.) Moench.), and rice (Oryza sativa L.) have been found to exhibit tolerance to ICIA-0051, SC-0774 and SC-0735 (39-41).

ICIA-0051, a selective herbicide in corn, shows good to excellent activity on a broad range of annual broadleaf weeds and some grass species (9-11, 16, 24-28, 30-33, 36, 43). The compound can be applied either as a preemergence surface application or as an over-the-top postemergence treatment (39). Although ICIA-0051 alone controls a fairly large spectrum of weeds, the addition of a triazine such as atrazine will significantly increase the weed control spectrum, particularly for postemergence applications (9, 24, 25, 36, 39).

Several researchers have shown that ICIA-0051 has good to excellent activity on redroot pigweed (Amaranthus retroflexus L.), common lambsquarters (Chenopodium album L.), and common ragweed (Ambrosia artemisiifolia L.) (8, 10, 11, 21, 24-26, 32, 33, 36). Field work done by Foy and Witt (10, 11) in 1985 and 1986, prior to this dissertation project, indicated that ICIA-0051 also gave adequate control of annual grasses such as large crabgrass [Digitaria sanguinalis (L.) Scop.]. Triazine-resistant lines of smooth pigweed and common lambsquarters exist in Virginia and elsewhere in the U.S. and abroad (42). Within the studies reported, ICIA-0051 provided adequate control of several triazine resistant species (24, 33, 50). Weeds shown to have some tolerance to ICIA-0051 include giant foxtail (<u>Setaria</u> <u>faberi</u> Herrm.), johnsongrass, and Texas panicum (<u>Panicum</u> <u>texanum</u> Buckl.) (21, 24, 33, 50).

Field use rates for ICIA-0051 as a preemergence treatment range from 0.28 to 2.2 kg ai/ha, while postemergence rates vary from 0.28 to 1.12 kg ai/ha (39). For postemergence applications of ICIA-0051, the addition of Tween-20 [oxysorbic(20 POE)polyoxyethylene sorbitan monolaurate] is recommended as an adjuvant (39). Improved weed performance has been noted when ICIA-0051 has been tank-mixed with either atrazine, cyanazine [2-{[4-chloro-6-(ethylamino)-1,3,5-triazin-2-yl)amino}-2-methylpropanenitrile], alachlor, or metolachlor [2-chloro-<u>N</u>-(2-ethyl-6-methylphenyl)-<u>N</u>-(2-methoxy-1-methylethy)acetamide] (16, 21, 26, 27, 36, 39, 43).

SC-0774 can be applied preemergence for control of a wide range of annual grasses and some broadleaf weeds (40). As with ICIA-0051, the addition of a triazine such as atrazine or cyanazine will improve weed control (17, 25, 26, 32, 37, 40). Research has shown good to excellent control of large crabgrass, broadleaf signalgrass [<u>Brachiaria</u> <u>platyphylla</u> (Griseb.)Nash], and common lambsquarters (8, 17, 21, 22, 24-27, 37). Some tolerant weeds include Texas panicum, junglerice [<u>Echinochloa</u> <u>colonum</u> (L.) Link], prickly sida (<u>Sida</u> <u>spinosa</u> L.), sicklepod (<u>Cassia</u> <u>obtusifolia</u> L.), and spurred anoda [<u>Anoda</u> <u>cristata</u> (L.) Schlecht.] (24).

Because of the problems with crop injury to corn with SC-

0774 in many field studies, crop safeners such as R-29148 have been investigated (6, 25, 32). R-29148 (chemistry undisclosed) is an experimental crop safener similar to dichlormid (formerly R-25788) [2,2-dichloro-N,N-di-2-propenylacetamide]. Recent reports within ICI Americas indicate that further development will be limited due to the lack of consistent broadleaf weed control with SC-0774.

Evans and Gunnell (8) noted reduced activity of both ICIA-0051 and SC-0774 due to the lack of moisture at one of their test sites. In general, SC-0774 is considered to give better grass control, while ICIA-0051 is considered to give better broadleaf control.

SC-0735 is being developed primarily as a rice herbicide although corn does exhibit tolerance (41). SC-0735 is similar to SC-0774, in that it is more effective for the control of annual and perennial grasses than broadleaf weeds.

All three of the ICI compounds show bleaching symptoms indicating that they may be inhibitors of carotenoid biosynthesis. Although the exact mode of action has not been worked out, other carotenoid inhibitors exhibit similar symptoms (29, 39-41). Nandihalli and Bhowmik (30-31) working with both ICIA-0051 and SC-0774, concluded that these compounds interfere with carotenoid biosynthesis and also possibly directly with chlorophyll synthesis. Mayonado (22) reported that ICIA-0051 inhibits carotenoid formation by blocking the desaturation of phytoene to phytofluene. These results were similar to those obtained with norflurazon [4-chloro-5-(methylamino)-2-(3-(trifluoromethyl)phenyl)-3(2H)-pyridazinone] (29). Inhibition of the carotenoid synthesis pathway, which results in the accumulation of phytoene and phytofluene, would result in the loss of carotenoids (29). Since the carotenoids protect the plant's organelles from photodamage, the inhibition of carotenoid synthesis leads to chlorophyll degradation in the light (29). Consequently, this absence of chlorophyll gives the plant a bleached appearance after its emergence or following postemergence applications of these compounds.

Field and laboratory soil studies with ICIA-0051 and SC-0774 have shown that both herbicides were more mobile in high pH soils, while under low pH conditions, these herbicides cause more crop injury to sensitive species (22).

The research reported in this dissertation covers the results on the field efficacy of ICIA-0051 and SC-0774 under conventional and no-tillage systems with different weed species not reported by other researchers (e.g. triazine-resistant pigweed). Also, a major portion of this dissertation addresses the key soil properties such as organic matter, clay content, CEC, and pH, and their relative importance and interactions in influencing herbicide mobility through the use of soil leaching columns and soil thin-layer chromatography. Also, the adsorption/ desorption behavior of ICIA-0051 was investigated and related to the above-mentioned soil properties. Finally a herbicide persistence study was conducted to measure the loss of biological activity over time, and ICIA-0051's potential for herbicide carryover to sensitive crops.

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II. FIELD EFFICACY STUDIES WITH ICIA-0051 AND SC-0774, ALONE AND IN COMBINATION WITH OTHER HERBICIDES, IN CONVENTIONAL AND NO-TILLAGE CORN (ZEA MAYS)

Abstract. Field studies were conducted in 1987 and 1988 to determine the weed control efficacy of ICIA-0051 and SC-0774 (chemistry not released) in no-till and conventional corn (*Zea mays* L.). Treatments consisted of both pre- and postemergence applications of ICIA-0051 alone and in combination with atrazine (6-chloro-*N*-ethyl-*N*'-(1-methylethyl)-1,3,5-triazine-2,4-diamine). SC-0774 treatments were applied preemergence, alone and in combination with atrazine. The weed species present at the no-till site consisted of fall panicum (*Panicum dichotomiflorum* Michx.) and triazine-resistant smooth pigweed (*Amaranthus hybridus* L.). The weeds present at the conventional tillage site were giant ragweed (*Ambrosia trifida* L.) and ivyleaf morningglory [*Ipomoea hederacea* (L.) Jacq.]. Results of the preemergence weed control evaluations taken 8 weeks after application indicated that ICIA-0051 across all treatments gave 85% or better control of smooth pigweed, while fall panicum control ranged from 43 to 87%. Giant ragweed control ranged from 30 to 95%, while control of ivyleaf morningglory was below 75%. Generally, the addition of atrazine to ICIA-0051 improved the control of most weed species. The postemergence treatments of ICIA-0051 applied at half the preemergence rate, gave

similar results to the preemergence treatments, except for ivyleaf morningglory control where the postemergence treatments were significantly better. SC-0774 applied preemergence at 8 weeks gave 85% or better control of fall panicum, but inadequate control of the broadleaf weed species (75% or worse). The numerically highest corn yields in the conventional plots were obtained with the higher rates of ICIA-0051 (1.7 kg ai/ha) alone or in combination with atrazine applied either pre- or postemergence. The numerically highest yields in the no-till corn were obtained with the preemergence applications of the ICIA-0051 alone (1.7 kg ai/ha rate only), with all ICIA-0051 plus atrazine combinations, or with the SC-0774 treatments.

INTRODUCTION

Corn is the primary acreage (U.S. 71 million acres in 1981) and cash crops produced in the U. S. and abroad. Although many economic problems face producers, one of the major concerns is the adequate control of weeds. To combat this problem, many effective herbicides have been developed over the years, although several weed species such as smooth pigweed and johnsongrass (Sorghum halepense L. Pers.) are still not adequately controlled.

Another problem associated with corn production, is the shift in the weed spectrum, caused by changing cultural practices from conventional tillage to no-tillage systems (4). Weed populations often shift and\or weeds may become resistant to certain herbicides. A classic example of weed resistance in corn production is smooth pigweed resistance to triazine herbicides such as atrazine (11). Based on initial field testing by company and university researchers, this class of compounds appears to provide adequate weed control of many common and difficult-to-control weed species (5-10). Previous field work done in 1985 and 1986 had shown promise for controlling annual broadleaf weed species such as smooth pigweed and annual grasses such as fall panicum with both ICIA-0051 and SC-0774 (2, 3).

The objective of this research was to investigate the effectiveness of ICIA-0051 and SC-0774, alone and in combination with atrazine, for weed control in conventional and no-till cropping systems. Treatments of ICIA-0051 and SC-0774 were applied both preemergence and postemergence to determine the best application method for a given weed species.

MATERIALS AND METHODS

General procedures. Weed species present at the conventional tillage site consisted of triazine-resistant smooth pigweed and fall panicum, while the no-tillage site contained giant ragweed and ivyleaf morningglory. Various rates and combinations of ICIA-0051 and SC-0774 were tested in field studies conducted in 1987 and 1988 to determine the spectrum of weed control and the most effective application time.

The experimental design at each location was a randomized complete block with four replications. Each of the treatments consisted of four-row plots 7.6 m long and 3.8 m wide with a row spacing of 97 cm. Plots were sprayed with a CO_2-pressurized backpack sprayer delivering 216 L/ha at 234 kPa. All treatments were sprayed at 4.8 km/h and water was used as the carrier. The sprayer boom consisted of four 11002 flat fan nozzles set 51 cm apart. The boom height during herbicide application was 46 cm above the ground preemergence and 46 cm above the weed canopy postemergence.

Preemergence treatments were sprayed approximately two days after planting, whereas postemergence treatments were sprayed based on the height and number of weeds present. The height of the weeds at the time of the postemergence applications were fall panicum 5 to 15 cm, smooth pigweed 8 cm, giant ragweed 8 to 12 cm and ivyleaf morningglory 8 cm. All postemergence treatments of ICIA-0051 received a nonionic surfactant Tween 20 [oxysorbic(20 POE)polyoxyethylene sorbitan monolaurate] applied at 0.5% v/v, while the postemergence treatment of atrazine received a crop oil concentrate applied at 38 L/ha. Both the conventional and no-till treatments were rated at 4, 6, and 8 weeks after application, although only the 4 and 8 week rating will be presented. Ratings were made for weed control and the crop monitored for crop injury, particularly after the postemergence applications. Weed ratings were based on a comparison to the control plots, using a scale of 0 to 100%, with 0 = no weed control and 100 = complete kill. Crop yields were obtained by collecting ears from the two middle rows over a 6.1-m section of the plot.

No-tillage. The no-till locations for both 1987 and 1988, were established at the Kipps research farm at Blacksburg, VA. The soil type at this location was a Groseclose loam (Clayey, mixed, mesic Typic Hapludults). The soil pH at this site was 6.5 and the organic matter content was 2.8% by weight. A broadcast application of paraquat (1,1'-dimethyl-4,4'-bipyridinium ion) was applied to all plots prior to planting to kill the rye (<u>Secale cereale</u> L.) cover crop. The no-till corn was planted on May 11, 1987 and May 12, 1988, using 'Pioneer 3233' and 'Hytest 686' corn varieties, respectively. Preemergence herbicide treatments were applied on May 13 in both 1987 and 1988. The postemergence treatments were applied on May 27, 1987 and June 10, 1988.

Weeds present at the time of the postemergence applications were fall panicum (approximately 5 cm tall) and smooth pigweed (6 cm) for both years. In 1988, the rye cover was mowed one day prior to the postemergence applications at a height 20 cm, due to the poor control by paraquat. This mowing did not affect the smaller weeds present at that time.

Conventional tillage. The conventional locations for both 1987 and 1988 were established at the Whitethorne-Kentland research farm near Blacksburg, VA. The soil type at this location was a Ross loam (fine-loamy, mixed, mesic Cumulic Hapludolls). The soil pH at this site was 5.9 and the organic matter content was 2.8% by weight. Plots were disked and planted on June 2, 1987 and June 6, 1988, using 'Southern States 727' and 'Southern States 737' corn varieties, respectively. Preemergence treatments were applied on June 5, 1987 and June 10, 1988. The postemergence treatments were applied on June 19, 1987 and June 24, 1988. Weeds present at the time of the postemergence applications were ivyleaf morningglory and giant ragweed, both ranging from 5 to 10 cm tall. In 1988, late season johnsongrass pressure affected yields in two replications.

Statistical analysis. Since different weed species were present at the conventional and no-tillage sites, the data from the two tillage systems were not combined. However, a statistical procedure was used to check for homogeneity between years within either the conventional or no-tillage plots over years. The homogeneity test used was the Snedecor and Cochran test for equality between two variances. Based on the test results, the weed ratings for the 4- and 8-week periods and the yield data were combined over years. The conventional or no-till experiments, combined over the two-year period, were subjected to an analysis of variance, and means were separated by using the Waller-Duncan t-test (K-ratio = 100).

RESULTS AND DISCUSSION

General results. There were no significant differences between years for either the conventional or no-till plots, so the data were combined over years. Weed control for both ICIA-0051 and SC-0774 in both sites varied depending on the weed species present. In general, ICIA-0051 was more effective against broadleaf species while SC-0774 was more effective against grass species, although several ICIA-0051 treatments give adequate control of both broadleaf weeds and grasses. Less rainfall was received in 1987 than in 1988; nevertheless, corn yields for the two years were comparable (Table 1). Also, the dry weather in 1987 did not appear to have a significant effect on weed growth or herbicide performance relative to 1988.

No-tillage. The preemergence application of ICIA-0051 alone, at the 1.1 and 1.7 kg ai/ha rates, gave adequate control (85% or better) of smooth pigweed, at both the early and late season ratings (Table 2 and 3). Those results were similar to the postemergence application rates of ICIA-0051 alone at 0.6 and 1.1 kg ai/ha. The high rates of ICIA-0051 used for both the preemergence (1.7 kg/ha) and postemergence treatments (1.12 kg /ha), gave significantly better weed control than the middle rates of ICIA-0051 (Table 2 and 3). ICIA-0051 at all of these rates was significantly more effective than atrazine applied either preemergence or postemergence. The primary reason for the poor performance of atrazine was because this site was infested with triazine-resistant pigweed.

Similar smooth pigweed control was obtained using the preemergence application of ICIA-0051 either alone, at 1.7 kg ai/ha, or in combination with atrazine, and these treatments were significantly better than the lower rates of ICIA-0051 alone and in combination with atrazine (Table 2 and 3). Adding metolachlor (2-chloro-_N_-(2-ethyl-6-methylphenyl)-_N_-(2-methoxy-1-methylethyl)acetamide) at 2.2 kg ai/ha to ICIA-0051 also significantly improved pigweed control compared to the same rate of ICIA-0051 or metolachlor alone, and the control was comparable to the ICIA-0051 plus atrazine combinations. The standard treatment of atrazine plus metolachlor gave poor control of smooth pigweed (48%) at 8 weeks, and was significantly less effective than all of the treatments with ICIA-0051 applied alone or in combination (Table 3). The addition of atrazine to the postemergence applications of ICIA-0051, however, did not significantly increase weed control (Table 3).

The preemergence application of SC-0774, alone or in combination with atrazine, gave only moderate smooth pigweed control, with a range from 66 to 81% at the 8-week evaluation (Table 3). SC-0774, alone or in combination with atrazine, provided lower smooth pigweed control than did ICIA-0051 at 1.7 kg/ha applied preemergence or 0.6 or 1.1 kg/ha applied postemergence.

These results agree with those of other researchers who have reported good to excellent control of both smooth and redroot pigweed (Amaranthus retroflexus L.) with ICIA-0051 alone and in combination with atrazine (6, 7, 9, 10).

For fall panicum, ICIA-0051 applied preemergence at 1.7 kg/ha alone or in combination with either atrazine or metolachlor gave adequate control at 8 weeks, i.e. 83, 84, and 87%, respectively (Table 3). The postemergence applications of ICIA-0051 alone or in combination did not give acceptable control (less than 60%) at 8 weeks. The atrazine treatments alone (pre- or postemergence), metolachlor alone (pre), or the two in combination (pre), also gave unacceptable control of fall panicum, and these treatments were generally less effective than the treatments applied preemergence.

The preemergence applications of SC-0774 alone or in combination with atrazine gave the best early and late season control of fall panicum (> 92%). SC-0774 applied preemergence was significantly better than all postemergence treatments. These results agree with those of other researchers who have shown that SC-0774 provides good to excellent control of several grass species such as large crabgrass [Digitaria sanguinalis (L.) Scop.] and broadleaf signalgrass [Brachiaria platyphylla (Griseb.) Nash] (1-3, 5, 7, 8).

Yield results from the combined no-till sites indicated that the numerically best preemergence treatments were ICIA-0051 alone at the high rate, or all combinations of ICIA-0051 plus atrazine or metolachlor (Table 3). These yields were comparable to the SC-0774 treatments alone or in combination with atrazine. All postemergence treatments were significantly less effective than the above-mentioned treatments. Based on visual observations, fall panicum, due to its heavy infestation, was more competitive and exerted a greater impact on yield in both years than did smooth pigweed. Generally, the treatments that gave moderate control of smooth pigweed and excellent control of fall panicum resulted in the highest yields.

Conventional tillage. The preemergence application of ICIA-0051 at the 1.7 kg ai/ha rate, gave both early and late season control (80%) of giant ragweed, and this was significantly better than the two lower rates used (Table 4 and 5). The addition of atrazine to ICIA-0051 preemergence significantly improved control at all rates of ICIA-0051 examined, although only the 1.1 and 1.7 kg ai/ha rate of ICIA-0051 plus atrazine gave adequate control. The addition of metolachlor to ICIA-0051 improved giant ragweed control compared to the same rate of ICIA-0051 alone, although control was only 75% with the combination at the 8-week rating (Table 5). Atrazine or metolachlor or the combination of the two applied preemergence resulted in poor giant ragweed control (< 50% at 8 weeks).

SC-0774 alone or in combination with atrazine did not provide adequate control of giant ragweed (Table 4 and 5). These results are similar to the work of other researchers who have shown that SC-0774 is ineffective on several broadleaf weeds such as sicklepod (*Cassia obtusifolia* L.) and spurred anoda [*Anoda cristata* (L.) Schlecht.] (9, 12).

The postemergence applications gave better control of giant ragweed than did the preemergence treatments, with the exception of atrazine postemergence and ICIA-0051 plus atrazine both at the 1.7 kg/ha rate preemergence (Tables 4 and 5). The postemergence treatments of ICIA-0051 were significantly better than the same rate applied preemergence. Similarly, the ICIA-0051 plus atrazine postemergence applications were better than the same treatments applied preemergence. The addition of atrazine to ICIA-0051, applied postemergence, did not significantly improve giant ragweed control at either the 4- or 8-weeks rating.

For the control of ivyleaf morningglory, only ICIA-0051 at 1.1 kg/ha plus atrazine postemergence gave greater than 90% control at 8 weeks after treatment (Table 4 and 5). None of the preemergence treatments with ICIA-0051 or SC-0774 gave adequate control of ivyleaf morningglory (> 75%). Initially, at the 4-weeks rating, all of the postemergence applications of ICIA-0051 did provide acceptable control; however, the two lower rates gave unacceptable (80%) results because of late season morningglory pressure (Table 4).

Yield data for the conventional sites indicated that the numerically best preemergence treatments were ICIA-0051 at 1.1 and 1.7 kg ai/ha alone or in combination with atrazine. The high rate of ICIA-0051 alone or in combination with atrazine was significantly better than the standard treatments of either atrazine, metolachlor, or the combination of the two. Corn yields for ICIA-0051 applied preemergence, at 1.7 kg/ha either alone or in combination with atrazine or metolachlor, or ICIA-0051 at 1.1 kg/ha postemergence either alone or in combination with atrazine were similar. The numerically best postemergence treatments were all combinations of ICIA-0051 or the high rate of ICIA-0051 alone.

With the weeds investigated in these experiments, ICIA-0051 appears to have potential for improved control of triazine-resistant pigweed, fall panicum, and giant ragweed. Depending on the weed species present, the higher rates of ICIA-0051 may be required for adequate season-long control. These results, similar to others reported (7), showed that the addition of atrazine to ICIA-0051 either pre- or postemergence did improve weed control efficacy on some of the species investigated. ICIA-0051, alone or in combination with atrazine or metolachlor, gave inadequate control of ivyleaf morningglory.

Although SC-0774 provided excellent control of fall panicum and appeared to have some activity against smooth pigweed, its performance overall was not as effective as most ICIA-0051 treatments. SC-0774 was particularly ineffective in the conventional corn tests where broadleaf weeds such as giant ragweed and ivyleaf morningglory were present. Although some researchers have reported crop injury with SC-0774 applied preemergence in field experiments, no noticeable crop injury was observed in the no-till and conventional tests during either 1987 or 1988 (6, 8). Further research with both of these herbicides is warranted, including investigating the potential of other tank-mix combinations.

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Table 1. Rainfall by month in Montgomery County, Virginia.

Year	May	June	July	August	September
	(cm)				
1987	4.3	3.3	7.9	4.2	4.1
1988	3.2	8.2	14.2	14.2	10.5

Table 2. Control of fall panicum and smooth pigweed by ICIA-0051 and SC-0774, 4 weeks after preemergence applications and 2 weeks after postemergence applications, in no-till planted field corn in 1987 and 1988.

Treatment	Rate	Time of application	Weed control[a] PANDI[b]	AMACH
	(kg ai/ha)		(%)	
1. ICIA-0051	0.6	Pre	58 h	71 kl
2. ICIA-0051	1.1	Pre	80 d	88 d-f
3. ICIA-0051	1.7	Pre	89 b	95 a-c
4. Atrazine	1.7	Pre	51 hi	0 m
5. Metolachlor	2.2	Pre	82 cd	73 jk
6. ICIA-0051 + atrazine	0.6 1.7	Pre	81 d	94 a-d
7. ICIA-0051 + atrazine	1.1 1.7	Pre	88 bc	99 a
8. ICIA-0051 + atrazine	1.7 1.7	Pre	98 a	99 a
9. ICIA-0051 + metolachlor	1.1 2.2	Pre	98 a	98 ab
10. Atrazine + metolachlor	1.7 2.2	Pre	76 d-f	75 i-k
11. SC-0774	0.6	Pre	96 a	80 g-i
12. SC-0774	1.1	Pre	98 a	92 b-d
13. SC-0774 + atrazine	0.6 1.7	Pre	94 ab	78 h-j
14. SC-0774 + atrazine	1.1 1.7	Pre	100 a	92 b-d
15. ICIA-0051	0.3	Post	46 i	66 l
16. ICIA-0051	0.6	Post	68 g	83 f-h
17. ICIA-0051	1.1	Post	78 de	90 d-e
18. Atrazine	1.1	Post	38 j	0 m
19. ICIA-0051 + atrazine	0.3 1.7	Post	71 f-g	85 e-g
20. ICIA-0051 + atrazine	0.6 1.7	Post	73 e-g	88 d-f
21. ICIA-0051 + atrazine	1.1 1.7	Post	78 de	99 a
22. Untreated	0.0	-	8 k	5 m

[a] Means within the same column followed by the same letter are not significantly different at the 0.05 level as determined by Waller-Duncan t-test (K-ratio = 100).

[b] Letters are a WSSA-approved computer code from Composite List of Weeds, Weed Sci. 32, Suppl. 2. Available from WSSA, 309 West Clark Street, Champaign, IL 61820.

Table 3. Control of fall panicum and smooth pigweed by ICIA-0051 and SC-0774, 8 weeks after preemergence applications and 6 weeks after postemergence applications, and yield in no-till planted field corn in 1987 and 1988.

Treatment	Rate	Time of application	Weed control[a] PANDI[b]	AMACH	Corn yield
	(kg ai/ha)		(%)	(%)	(kg/ha)
1. ICIA-0051	0.6	Pre	43 jk	78 d	5175 fg
2. ICIA-0051	1.1	Pre	66 ef	85 c	6945 c-e
3. ICIA-0051	1.7	Pre	83 d	100 a	7669 a-d
4. Atrazine	1.7	Pre	33 l	0 h	2174 h
5. Metolachlor	2.2	Pre	58 f-h	69 e	5742 e-g
6. ICIA-0051	0.6	Pre	57 f-h	81 cd	6993 b-e
+ atrazine	1.7				
7. ICIA-0051	1.1	Pre	69 e	93 b	8542 a
+ atrazine	1.7				
8. ICIA-0051	1.7	Pre	84 cd	98 ab	8676 a
+ atrazine	1.7				
9. ICIA-0051	1.1	Pre	87 a-d	99 a	8099 a-c
+ metolachlor	2.2				
10. Atrazine	1.7	Pre	46 i-k	48 f	6493 d-f
+ metolachlor	2.2				
11. SC-0774	0.6	Pre	92 a-c	66 e	8012 a-c
12. SC-0774	1.1	Pre	95 a	81 cd	8339 ab
13. SC-0774	0.6	Pre	86 b-d	68 e	8715 a
+ atrazine	1.7				
14. SC-0774	1.1	Pre	94 ab	84 c	8792 a
+ atrazine	1.7				
15. ICIA-0051	0.3	Post	50 h-j	86 c	5099 g
16. ICIA-0051	0.6	Post	55 gh	98 ab	5377 f-g
17. ICIA-0051	1.1	Post	62 e-g	100 a	5685 e-g
18. Atrazine	1.1	Post	40 kl	14 g	3453 h
19. ICIA-0051	0.3	Post	56 gh	86 c	4896 g
+ atrazine	1.7				
20. ICIA-0051	0.6	Post	58 f-h	98 ab	5973 e-g
+ atrazine	1.7				
21. ICIA-0051	1.1	Post	57 f-h	100 a	6147 e-g
+ atrazine	1.7				
22. Untreated	0.0	-	0 m	18 g	3021 h

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[a] Means within the same column followed by the same letter are not significantly different at the 0.05 level as determined by Waller-Duncan t-test (K-ratio = 100).

[b] Letters are a WSSA-approved computer code from Composite List of Weeds, Weed Sci. 32, Suppl. 2. Available from WSSA, 309 West Clark Street, Champaign, IL 61820.

Table 4. Control of giant ragweed and ivyleaf morningglory by ICIA-0051 and SC-0774, 4 weeks after preemergence applications and 2 weeks after postemergence applications, in conventionally planted field corn in 1987 and 1988.

Treatment	Rate	Time of application	Weed control[a]	
			AMBTR[b]	IPOCC
	(kg ai/ha)		(%)	
1. ICIA-0051	0.6	Pre	63 f	48 h
2. ICIA-0051	1.1	Pre	80 d	66 fg
3. ICIA-0051	1.7	Pre	89 bc	73 ef
4. Atrazine	1.7	Pre	89 bc	79 de
5. Metolachlor	2.2	Pre	49 g	48 h
6. ICIA-0051 + atrazine	0.6 1.7	Pre	87 cd	81 c-e
7. ICIA-0051 + atrazine	1.1 1.7	Pre	95 ab	93 ab
8. ICIA-0051 + atrazine	1.7 1.7	Pre	98 a	88 bc
9. ICIA-0051 + metolachlor	1.1 2.2	Pre	89 bc	86 b-d
10. Atrazine + metolachlor	1.7 2.2	Pre	48 g	74 ef
11. SC-0774	1.1	Pre	71 e	63 g
12. SC-0774 + atrazine	1.1 1.7	Pre	84 cd	86 b-d
13. ICIA-0051	0.3	Post	99 a	88 bc
14. ICIA-0051	0.6	Post	100 a	92 ab
15. ICIA-0051	1.1	Post	100 a	97 a
16. Atrazine	1.1	Post	98 a	96 a
17. ICIA-0051 + atrazine	0.3 1.7	Post	100 a	98 a
18. ICIA-0051 + atrazine	0.6 1.7	Post	100 a	99 a
19. ICIA-0051 + atrazine	1.1 1.7	Post	100 a	97 a
20. Untreated	0.0	-	23 h	16 i

[a] Means within the same column followed by the same letter are not significantly different at the 0.05 level as determined by Waller-Duncan t-test (K-ratio = 100).

[b] Letters are a WSSA-approved computer code from Composite List of Weeds, Weed Sci. 32, Suppl. 2. Available from WSSA, 309 West Clark Street, Champaign, IL 61820.

Table 5. Control of giant ragweed and ivyleaf morningglory by ICIA-0051 and SC-0774, 8 weeks after preemergence applications and 6 weeks after postemergence applications, and yield in conventionally planted field corn in 1987 and 1988.

Treatment	Rate	Time of application	Weed control[a] AMBTR[b]	Weed control[a] IPOCC	Corn yield
	(kg ai/ha)		(%)	(%)	(kg/ha)
1. ICIA-0051	0.6	Pre	30 k	22 h	4921 d-f
2. ICIA-0051	1.1	Pre	61 hi	35 g	5725 a-e
3. ICIA-0051	1.7	Pre	80 de	55 e	6441 a-c
4. Atrazine	1.7	Pre	51 j	40 fg	4859 ef
5. Metolachlor	2.2	Pre	4 l	4 i	2238 h
6. ICIA-0051 + atrazine	0.6 1.7	Pre	57 ij	42 fg	4752 ef
7. ICIA-0051 + atrazine	1.1 1.7	Pre	87 cd	74 c	5712 a-e
8. ICIA-0051 + atrazine	1.7 1.7	Pre	92 a-c	74 c	7057 a
9. ICIA-0051 + metolachlor	1.1 2.2	Pre	75 ef	43 fg	4735 ef
10. Atrazine + metolachlor	1.7 2.2	Pre	34 k	35 g	2952 gh
11. SC-0774	1.1	Pre	30 k	5 i	3733 fg
12. SC-0774 + atrazine	1.1 1.7	Pre	65 gh	46 f	5405 b-e
13. ICIA-0051	0.3	Post	89 bc	64 d	5299 d-e
14. ICIA-0051	0.6	Post	93 a-c	78 bc	4955 d-f
15. ICIA-0051	1.1	Post	96 ab	84 ab	5773 a-e
16. Atrazine	1.1	Post	71 fg	65 d	4820 ef
17. ICIA-0051 + atrazine	0.3 1.7	Post	93 a-c	81 bc	6390 a-d
18. ICIA-0051 + atrazine	0.6 1.7	Post	96 ab	84 ab	6863 ab
19. ICIA-0051 + atrazine	1.1 1.7	Post	99 a	92 a	5390 b-e
20. Untreated	0.0	-	8 l	8 i	1813 h

[a] Means within the same column followed by the same letter are not significantly different at the 0.05 level as determined by Waller-Duncan t-test (K-ratio = 100).

[b] Letters are a WSSA-approved computer code from Composite List of Weeds, Weed Sci. 32, Suppl. 2. Available from WSSA, 309 West Clark Street, Champaign, IL 61820.

III. SOIL MOBILITY OF ICIA-0051 AS INFLUENCED BY VARIOUS SOIL PROPERTIES

Abstract. Soil thin-layer chromatography and soil leaching column techniques were used to determine the effect of various soil properties on the mobility of ICIA-0051 in five soils. Atrazine [6-chloro-N-ethyl-N'-(1-methylethyl)-1,3,5-triazine-2,4-diamine] was used as reference standard in both studies and SC-0774 was included in the soil thin-layer chromatography work. The soil series selected, which represented predominant corn soils, were Appling loamy sand, Bojac sandy loam, Davidson clay, Frederick silt loam, and Hyde silty clay loam. Each of these soils was analyzed for various key soil properties such as percent organic matter, clay content, pH and other properties. Within each soil, the order of mobility from most mobile to least was ICIA-0051, SC-0774, and atrazine in all soils except Hyde. A comparison of ICIA-0051 across all soils, indicated that the order of mobility was: Appling > Davidson > Frederick = Bojac > Hyde. Both SC-0774 and atrazine had the same relative order of movement across all soils. The correlation of determination indicated that the organic/humic matter soil fraction was highly negatively correlated ($r^2 = 0.96$) with the movement of ICIA-0051. Other soil properties such as pH ($r^2 = 0.29$), and clay content ($r^2 = 0.16$) were not strongly correlated with herbicide movement. Results of the soil

column leaching study for ICIA-0051 and atrazine were similar to those obtained in the soil thin-layer study.

INTRODUCTION

Because of the concern for potential groundwater contamination or reduced weed control caused by the excessive herbicide movement in soil, more information is needed concerning the various herbicide and soil interactions. The availability and persistence of a herbicide in the soil is strongly influenced by its mobility. This mobility, however, in influenced by many environmental, herbicidal, and soil factors. Several researchers have investigated the various soil and herbicide interactions that affect mobility through the use of soil leaching columns or soil thin-layer chromatography (4, 5, 11, 12). According to Wu (12), results from these studies are often comparable, and both methods are good research tools to measure herbicide mobility in the soil.

Some of the important properties of the soil which influence the herbicide's mobility are the percent organic matter, clay content, and pH. Likewise, certain properties of the herbicide such as the ionization, water solubility, and molecular functional groups also influence the herbicide's mobility in the soil.

Two new herbicides, ICIA-0051 and SC-0774, have been tested over the last four years for weed control in corn (*Zea mays* L.), although little is known about their soil activity. Mayonado (7) reported that increasing the soil pH in a sandy loam soil would increase the mobility of ICIA-0051 and SC-0774. However, no work has been conducted on the effect of other soil properties on the mobility of ICIA-0051 and SC-0774.

The objective of this research was to determine the soil mobility of ICIA-0051 in various soils having a wide range in soil properties such as percent organic matter, clay content, and pH. The techniques used in this study were soil thin-layer chromatography and soil column leaching. Atrazine was included in both studies as a reference standard and SC-0774 was included in the soil thin-layer study.

MATERIALS AND METHODS

Soil selection and analysis. Five soil series were selected from across the state of Virginia. These soils were Appling loamy sand (clayey, kaolinitic, thermic Typic Hapludult), Bojac sandy loam (coarse-loamy, siliceous, thermic Typic Hapludult), Davidson clay (clayey, kaolinitic, thermic Rhodic Paleudult), Frederick silt loam (clayey, mixed, mesic Typic Paleudult), and Hyde silty clay loam (fine-silty, mixed, thermic Typic Umbraquult). The soils selected represented predominant corn soils in the state and also a wide range in organic matter, clay content, pH, and other soil characteristics (Table 1). The sand, silt, and clay content, and the percent organic matter were analyzed at the Virginia Polytechnic Institute and State University soil testing laboratory. The sand, silt, and clay analysis was similar to the methods described by Day (3), and the organic matter analysis used the Walkley (9) procedure. All other soil properties such as pH, CEC, and humic matter were analyzed at the North Carolina State soil testing laboratory.

Soil thin-layer chromatography. The chromatographic technique used for this procedure was similar to that described by Helling 1971 (4). The five soils described above were collected and initially sieved through a 2-mm mesh screen, followed by sieving through a 707-micron mesh screen.

The soil chromatographic plates were prepared by making a soil slurry and applying the soil uniformly to a plate 20 cm X 20 cm, at a thickness of 1 mm. Work done by Helling (5), had shown that varying the soil thickness from 250 micron to 1 mm had no significant effect on herbicide movement.

After each soil was applied to the plates, it was allowed to dry slowly for 4 days to prevent cracking. Using a 10-uL syringe the following amounts of each radiolabeled herbicide were applied to each soil, 1.5 cm from the bottom of the plate, as follows: ICIA-0051, 4 uL or 0.071 uCi (sp. act. 28.2 mCi/mmole), SC-0774 5 uL or 0.072 uCi (sp. act. 23.0 mCi/mmole), atrazine, 8 uL or 0.078 uCi (sp. act. 16 mCi/mmole). Plates were then placed in chromatography tanks, with distilled water used as the solvent. The water was allowed to move up the plates 11.5 cm, and then the plates were removed from the chromatography tanks and dried for 4 days. An autoradiography technique (1, 2) was used involving exposure of the plates to X-ray film for 5 days, after which the film was developed. The Rf value for each herbicide within each soil was calculated, by measuring the point of greatest herbicide movement. Each herbicide treatment was replicated four times within a given soil and the experiment was repeated.

Soil column leaching. The method used for the preparation of the soil columns was similar to that described by Weber 1986 (10). The columns were made of polyvinyl chloride pipe which was 35 cm long, and had an internal diameter of 9.7 cm. The columns were split vertically and beads of silicone sealer were applied to the insides of the columns at 5-cm increments. These silicone beads prevented the movement of water and/or chemicals down the wall inside the columns.

The two vertical halves were joined together with a silicone sealer, and a 5-cm cap with a 7-mm drain hole in the middle was fitted to the bottom of the column. A nylon mesh screen, the same diameter as the column, was placed in the bottom, and coarse quartz sand was added to fill the bottom cap. Each column was then uniformly packed by volume with one of the five soils. The amount of soil added to each column was: Appling loamy sand (3,251 g/column), Bojac sandy loam (3,048 g/column), Davidson clay (2,502 g/column), Frederick silt loam (2,627 g/column), and Hyde silty clay loam (2,127 g/column). Each column was saturated with water over a two-day period, and then allowed to drain for 24 h before herbicide application.

The herbicides applied to each soil were ICIA-0051 at 1.12 and 2.24 kg/ha, and atrazine at 1.68 kg/ha. An untreated check was also used for each soil type. Water was applied in 50-ml increments every 30 min for 4 h over a two-day period, and this was equivalent to a total amount of 10 cm (800 ml) of water. The columns were allowed to drain overnight, and were then split vertically. Preliminary work with mustard (Brassica kaber L.), showed this plant to be a sensitive bioassay species for both atrazine and ICIA-0051, therefore it was planted in each of the columns at 5-cm depth increments. Mustard fresh shoots were harvested and dry weights recorded 3 weeks after planting. Each soil was replicated four times and the experiment was repeated.

Statistical analysis. In both the soil thin-layer chromatography and soil leaching studies, the repeated experiments were tested for homogeneity using the Snedecor and Cochran test for equality between two variances. Since the experiments were homogeneous, the data for repeated runs were combined. The data from both experiments were then subjected to an analysis of variance and the means separated by using the Waller-Duncan t-test (K-ratio = 100). A Duncan's multiple range test at the 0.05 significance level was used in the soil column leaching study, where means for each herbicide were compared across all soils.

RESULTS AND DISCUSSION

Soil thin-layer chromatography. ICIA-0051 had the greatest movement of the herbicides investigated, in all of the soils except Hyde (Table 2). In general, the order of movement for the three herbicides in the Appling, Bojac, Davidson, and Frederick soil series was ICIA-0051 more mobile than SC-0774, followed by atrazine. In the Hyde soil the order of movement was atrazine, followed by ICIA-0051, and then SC-0774. Even though herbicide movement was limited in the Hyde soil, the greater movement of atrazine relative to ICIA-0051 and SC-0774 might have been due to the high organic matter of this soil. Atrazine appeared to be less affected by the soil's high organic matter, due to less adsorption to this fraction (10).

The Rf value for ICIA-0051 in the Appling soil was 6.4 (scale: 0 to 10 cm) and this was significantly greater than the Rf value of 5.9 for SC-0774 or 5.4 for atrazine (Table 2). The Rf values for ICIA-0051 in the Davidson, Frederick, and Bojac soils were 5.6, 5.0, and 4.9, respectively. In each of these soils, ICIA-0051 moved significantly farther than did SC-0774 or atrazine. The Rf values for atrazine, ICIA-0051, and SC-0774 for the Hyde soil were 1.4, 1.1, and 0.7, respectively.

A comparison of ICIA-0051 across all soils indicated that the relative order of movement for ICIA-0051 was: Appling >

Davidson > Frederick = Bojac > Hyde (Figure 1). Similarly, SC-0774 compared across all soils had the same order of movement, although it did not move as far as ICIA-0051 in any particular soil (Figure 2). The order of movement for the standard treatment of atrazine across all soils was different from either ICIA-0051 or SC-0774. The relative order of movement for atrazine was: Appling > Bojac = Davidson > Frederick > Hyde (Figure 3). The Rf values reported for atrazine (3.6 and 5.4) are comparable to those reported by other researchers for sandy soils which, depending on the soil series, ranged from 3.5 to 8.9 (5). The lower numbers for atrazine in the Davidson, Frederick, and Hyde soils might be due to the relatively high organic and/or clay fractions in these soils.

Correlation analysis for the soil thin-layer chromatography, indicated that the Rf value for ICIA-0051 in each soil was strongly influenced (negative correlation) by the organic/humic matter fractions (Table 3). The r^2 (correlation of determination) values for the organic matter and humic matter were both 0.96. Other research by Wilson (10) has shown that the organic/humic matter is also highly positively correlated with the availability and binding of ICIA-0051 to these fractions in soil. Consequently, it appears that this binding is responsible for the decreased mobility of ICIA-0051 in soil. This would explain the poor mobility of ICIA-0051 in the Hyde soil (11.7% organic matter).

Other correlation analyses indicated that the soil pH (Table 1) was not as strongly correlated ($r^2 = 0.29$) with herbicide mobility as was the soil organic fraction (Table 4). Mayonado (6) reported that increasing the soil pH in a sandy loam soil did increase the mobility of both ICIA-0051 and SC-0774. Technical data would support this since both ICIA-0051 and SC-0774 are more water soluble under high pH conditions (7, 8). However, across a variety of soils investigated in this study, which ranged in pH from 4.5 to 6.7, the influence of pH was not the most important soil factor affecting soil mobility (Table 1). The CEC and base saturation, which are related to soil pH, were also poorly correlated ($r^2 = 0.26$ and 0.06, respectively) with herbicide mobility in the soil.

The clay content (Table 1) of the soil also had a poor correlation ($r^2 = 0.16$) with the mobility of ICIA-0051 in the soil (Table 4). The clay content range in these soils varied from a high of 48% to a low of 5%.

Soil column leaching. Results from the leaching study were similar to those observed for the soil thin-layer chromatography study. Soil mobility of ICIA-0051 across all soils, at the 2.24 kg/ha rate, indicated that the order of mobility was: Appling > Davidson = Bojac > Frederick = Hyde (Figure 4). The lower rate of ICIA-0051, 1.12 kg/ha, gave similar results. Although movement in the Appling soil was not significantly greater than in the Davidson soil, ICIA-0051 was significantly more mobile than in the Bojac soil (Figure 5).

Although it is difficult to compare the mobility of atrazine to ICIA-0051, a dose response curve indicated that both herbicides gave a similar sensitivity to the bioassay species at an equal soil concentration. Visual and dry weight data indicated that ICIA-0051 was more mobile than atrazine across all soils investigated. The order of mobility of atrazine across soils was Appling = Davidson > Bojac > Frederick > Hyde (Figure 6).

Comparing the mobility within a column by sections, indicated that the sandy soils (Appling and Bojac) and Davidson clay allowed a greater herbicide leaching throughout the column than did the Frederick or Hyde soil (Tables 4 and 5). In comparison to the check, 50% or more injury was observed in all six column sections of the Appling loamy sand. The upper sections did have significantly more injury than the lower sections. Results from the Bojac sandy loam and Davidson clay showed a similar trend to that of the Appling soil, although each section showed less overall injury by depth. Crop injury in the Frederick and Hyde soils was limited to the upper two sections (0 to 5 and 6 to 10 cm) of the soil column. Comparing atrazine mobility by soil depth to ICIA-0051 showed similar results, although in general atrazine was less mobile (Table 6).

In summary, both studies indicated that increasing the organic/humic matter in the soil content would decrease the mobility of these herbicides. Results also indicated that in most soils ICIA-0051 was more mobile than SC-0774, followed by atrazine.

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Table 1. Physical and chemical characteristics of the five soils studied.

Soil		Particle size distribution			Soil
Series	Texture	Sand	Silt	Clay	pH
			(%)		
Appling	loamy sand	78.5	16.5	5.0	5.1
Bojac	sandy loam	66.8	28.2	5.0	4.8
Davidson	clay	13.1	39.2	47.7	6.7
Frederick	silt loam	12.7	67.2	20.1	5.9
Hyde	silty clay loam	7.2	55.7	37.1	4.5

Table 1. Physical and chemical characteristics of the five soils studied (cont'd.).

Soil series	Organic matter by volume	Humic matter	CEC	Bulk density	Base saturation
	(%)	(g/100 cm^3)	(meq/100cm^3)	(g/cm^3)	(% of CEC)
Appling	1.2	0.2	2.7	1.48	55
Bojac	2.1	0.5	4.8	1.21	58
Davidson	1.9	0.2	10.2	1.11	96
Frederick	2.5	0.7	5.5	1.05	78
Hyde	11.7	3.7	9.0	0.62	60

Table 2. Comparison of Rf values of ICIA-0051, SC-0774, and atrazine in five soils using soil thin-layer chromatography.

Herbicide treatment	Soil series[a]				
	Appling	Bojac	Davidson	Frederick	Hyde
	(Rf value)				
ICIA-0051	6.4 a	4.9 a	5.6 a	5.0 a	1.1 b
SC-0774	5.9 b	4.2 b	4.9 b	3.7 b	0.7 c
Atrazine	5.4 c	3.6 c	4.1 c	3.7 b	1.4 a

[a] Means within the same column followed by the same letter are not significantly different at the 0.05 level as determined by Waller-Duncan t-test (K-ratio = 100).

Table 3. The correlation of determination for the mobility of ICIA-0051 and SC-0774 in five selected soils using soil thin-layer chromatography.

Soil property	Correlation	
	ICIA-0051	SC-0774
	———— (r^2) ————	
Organic matter (%)	- 0.96	- 0.90
Humic matter	- 0.96	- 0.91
Bulk density	- 0.87	- 0.92
pH	- 0.29	- 0.22
CEC	- 0.26	- 0.25
Clay content	- 0.16	- 0.17
Base saturation	- 0.06	- 0.03

Table 4. Movement of ICIA-0051, applied at 1.12 kg/ha, within each soil in the soil column leaching study as determined by shoot weight reduction of mustard.

Column section	Soil series[a]				
	Appling	Bojac	Davidson	Frederick	Hyde
(cm)	(% of check weight)				
0 - 5	26 bc	33 bc	37 bc	33 c	43 c
6 - 10	10 c	15 c	28 bc	61 b	75 b
11 - 15	15 c	15 c	24 c	94 a	90 a
16 - 20	35 ab	40 a-c	28 bc	98 a	91 a
21 - 25	40 ab	69 ab	42 ab	107 a	95 a
26 - 30	48 a	75 a	55 a	107 a	95 a

[a] Means within the same column followed by the same letter are not significantly different at the 0.05 level as determined by Waller-Duncan t-test (K-ratio = 100).

Table 5. Movement of ICIA-0051, applied at 2.24 kg/ka, within each soil in the soil column leaching study as determined by the shoot weight reduction of mustard.

Column section	Soil series[a]				
	Appling	Bojac	Davidson	Frederick	Hyde
(cm)	(% of check weight)				
0 - 5	7 b	10 c	32 ab	28 d	37 d
6 - 10	13 b	13 c	25 b	48 c	76 c
11 - 15	16 b	20 c	26 b	79 b	87 bc
16 - 20	17 b	36 bc	30 b	101 a	95 ab
21 - 25	43 a	65 ab	37 ab	102 a	98 ab
26 - 30	45 a	75 a	48 a	105 a	101 a

[a] Means within the same column followed by the same letter are not significantly different at the 0.05 level as determined by Waller-Duncan t-test (K-ratio = 100).

Table 6. Movement of atrazine, applied at 1.68 kg/ha, within each soil in the soil column leaching study, as determined by the shoot weight reduction of mustard.

Column section	Soil series[a]				
	Appling	Bojac	Davidson	Frederick	Hyde
(cm)	(% of check weight)				
0 - 5	12 c	9 c	30 d	24 d	49 c
6 - 10	20 c	15 c	30 d	65 c	81 b
11 - 15	34 b	58 b	28 d	84 b	97 a
16 - 20	73 a	94 a	43 c	94 ab	100 a
21 - 25	70 a	100 a	60 b	98 a	103 a
26 - 30	60 a	111 a	89 a	99 a	104 a

[a] Means within the same column followed by the same letter are not significantly different at the 0.05 level as determined by Waller-Duncan t-test (K-ratio = 100).

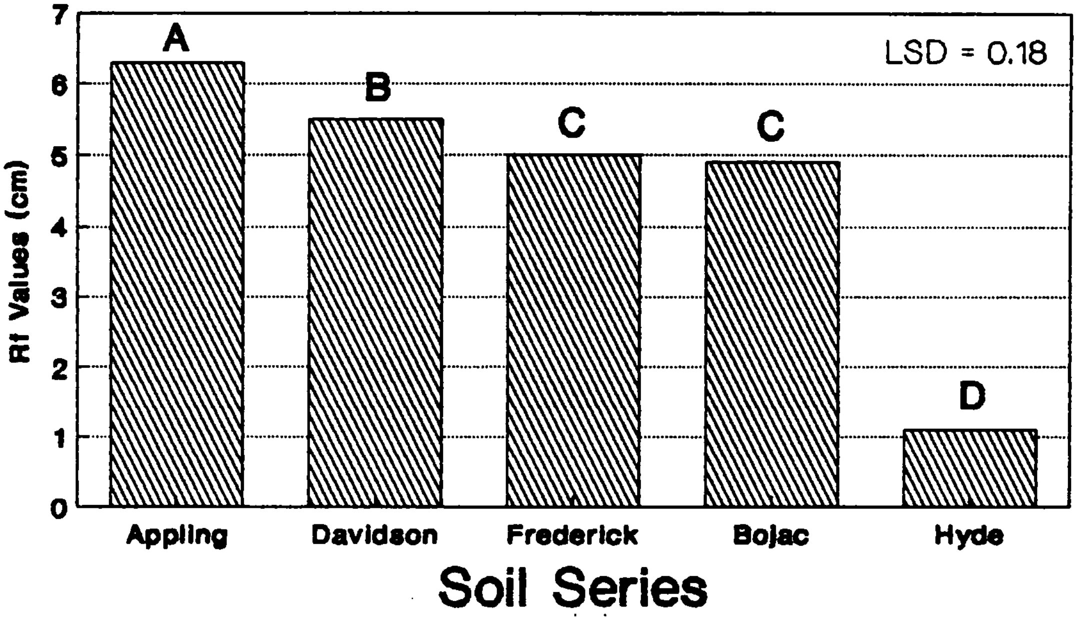

Figure 1. Mobility of ICIA-0051 in five soils using soil thin-layer chromatography.

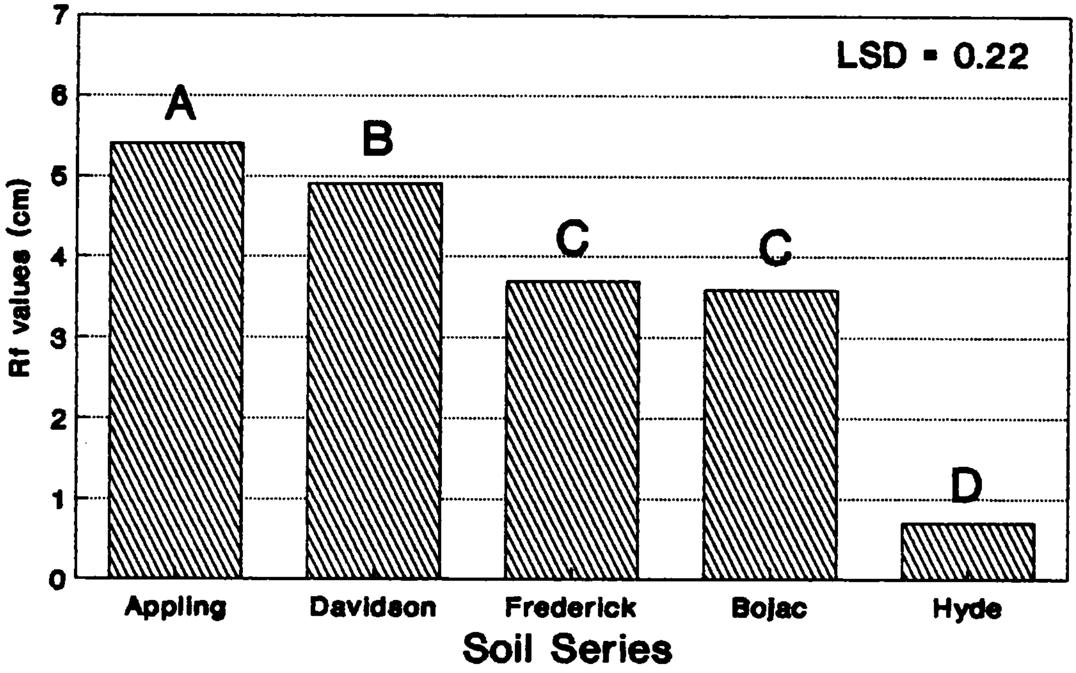

Figure 2. Mobility of SC-0774 in five soils using soil thin-layer chromatography.

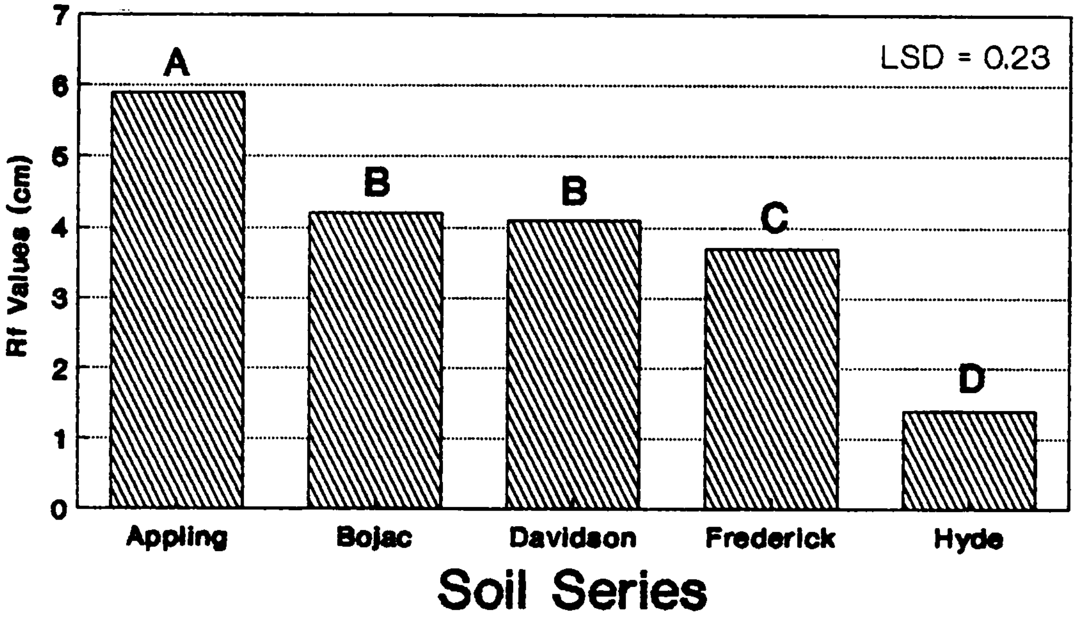

Figure 3. Mobility of atrazine in five soils using soil thin-layer chromatography.

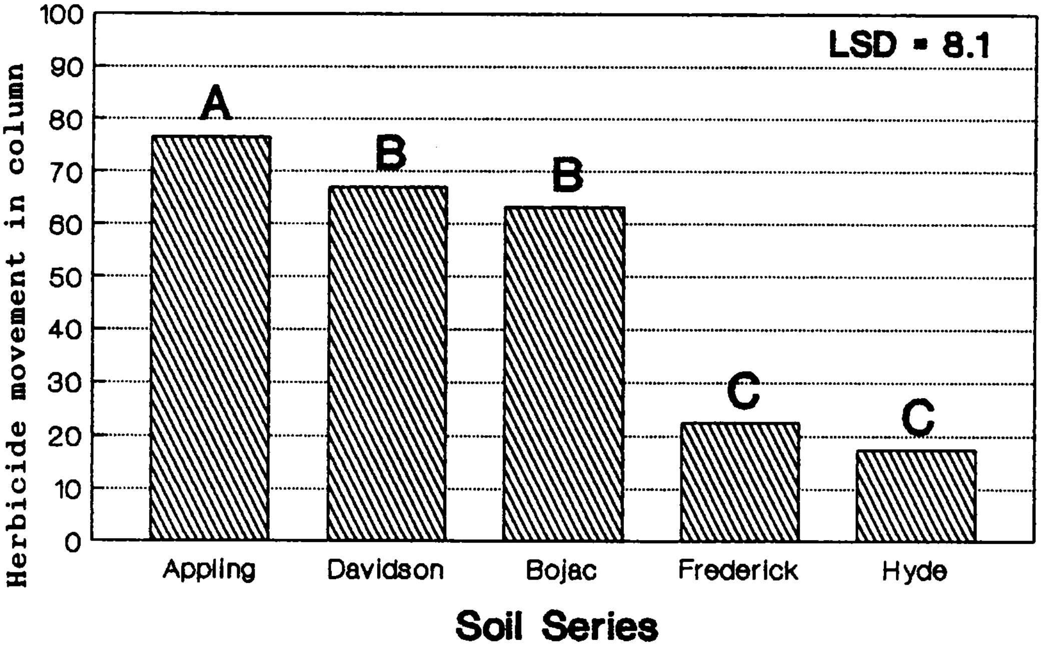

Figure 4. Comparison of the mobility of ICIA-0051 at 2.24 kg/ha in five soils, using a soil column leaching technique.

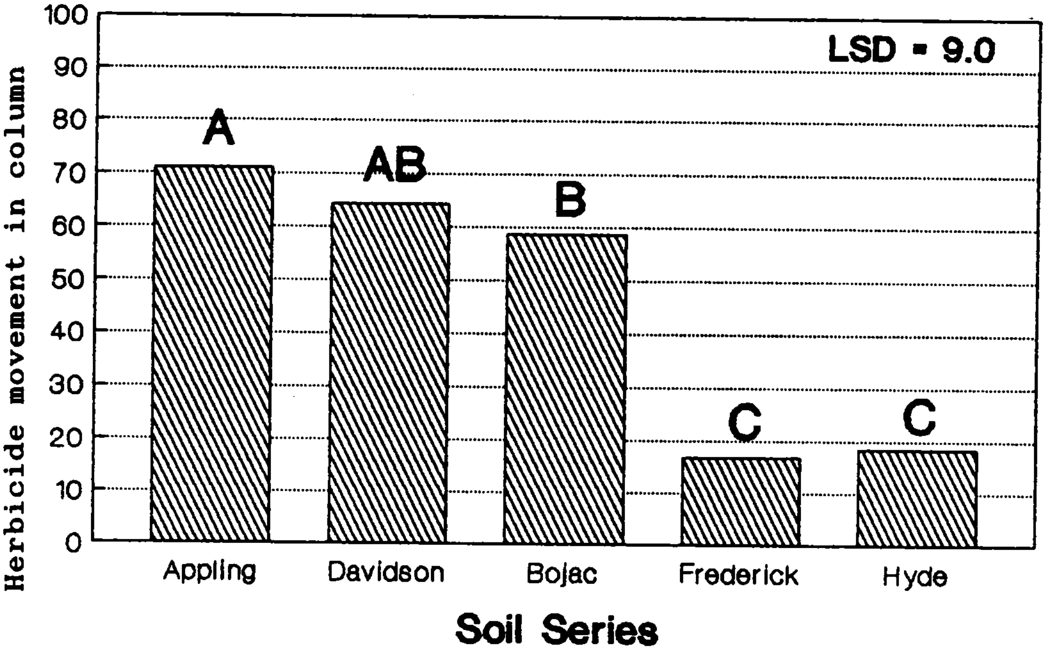

Figure 5. Comparison of the mobility of ICIA-0051 at 1.12 kg/ha in five soils, using a soil column leaching technique.

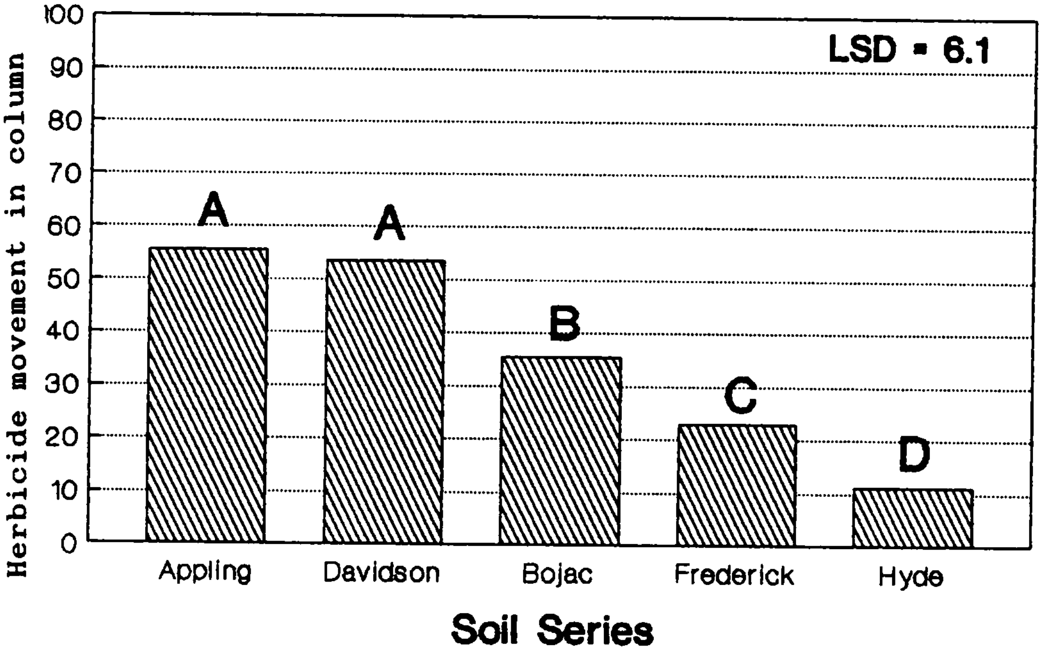

Figure 6. Comparison of the mobility of atrazine at 1.68 kg/ha in five soils, using a soil column leaching technique.

IV. INFLUENCE OF VARIOUS SOIL PROPERTIES ON THE ADSORPTION AND DESORPTION OF ICIA-0051 IN FIVE SOILS

Abstract. An adsorption and desorption study was conducted on ICIA-0051 to determine the influence of various soil properties on the herbicide's availability. Results indicated that the organic matter and/or humic matter fraction was highly positively correlated (r_2 = 0.99 and 0.98, respectively) with the adsorption of ICIA-0051. The clay content and other soil factors were less correlated with adsorption. The Freundlich equation was used and K and 1/n constants were calculated for each of the five soils. K values ranged from 0.63 (Appling, sandy loam) to 7.03 (Hyde silty clay loam). The Freundlich equation appeared to describe adequately the adsorption of ICIA-0051 by the various soils investigated. Based on the K constants, the general order for adsorption for each soil was Hyde (silty clay loam) (K = 7.04) > Frederick (silt loam) (K = 1.02) > Davidson (clay) (K = 0.98) = Bojac (loamy sand) (K = 0.98) > Appling (sandy loam) (K = 0.63). Results of the two desorptions indicated that across all soils, 25 to 50% of the amount adsorbed was removed by the two desorptions. The Appling, Bojac, and Davidson soils adsorbed and also retained less herbicide after two desorptions (0.47 ug/g, 0.69 ug/g, and 0.40 ug/g, respectively) than did the two higher organic matter soils (Frederick 0.73 ug/g and Hyde 4.14 ug/g). The

organic matter also appeared to be positively correlated with retention of ICIA-0051 in the soil.

INTRODUCTION

The availability and persistence of a herbicide is influenced by several properties of soil. Recent concern about herbicide carryover to sensitive rotation crops and potential groundwater contamination has increased the need to understand better the interactions between herbicides and the soil environment. Several researchers have demonstrated that the soil organic matter fraction, as well as the percent and type of clay in the soil, strongly influences the availability of herbicides (3, 4, 6, 9). The organic matter in particular has been positively correlated with absorbance of the triazine and phenylurea herbicides (3, 9). However, other soil factors such as soil pH, cation exchange capacity, and various environmental factors, such as the amount and intensity of rainfall, can also influence a herbicide's fate in the soil. Understanding which soil properties are more important is difficult, because of the various interactions between the herbicide and soil which affect or determine herbicide availability in the soil.

Although ICIA-0051 has been field-tested for many years by industry and various universities, little is known about its interactions in the soil, which determine its availability. Soil leaching and field work has indicated that increasing the soil pH increases the mobility of ICIA-0051 in sandy loam soils (5). Wilson (10) demonstrated that the organic matter was negatively correlated with the mobility of ICIA-0051 and that the clay content, CEC, pH, and other soil properties were less correlated with mobility. Since adsorption and desorption of a compound influence herbicide retention and availability, research to investigate these properties, using ICIA-0051 in various soil types was conducted.

The objective of this study was to investigate the effect of organic matter, clay content, and various other soil properties on the availability of ICIA-0051. An adsorption/desorption experiment was conducted with ICIA-0051 on five different corn soils.

MATERIALS AND METHODS

Soil selection and analysis. Five predominant Virginia corn soils were selected which represented a wide range of organic matter, humic matter, clay content, pH, cation exchange capacity (CEC), and other soil properties. The soil series selected were Appling loamy sand (clayey, kaolinitic, thermic Typic Hapludult), Bojac sandy loam (coarse-loamy, siliceous, thermic Typic Hapludult), Davidson clay (clayey, kaolinitic, thermic Rhodic Paleudult), Frederick silt loam (clayey, mixed, mesic Typic Paleudult), and Hyde silty clay loam (fine-silty, mixed, thermic Typic Umbraquult). The organic content and humic matter, respectively, in these soils was as follows Hyde 11.7%, 3.7 g/100 cm^3; Frederick 2.5%, 0.7 g/100cm^3; Bojac 2.1%, 0.5 g/100cm^3; Davidson 1.9%, 0.2 g/100cm^3; and Appling 1.2%, 0.2 g/cm^3. The pH ranged from 4.5 to 6.7, while the clay content ranged from 5 to 48%. Soils were analyzed at the Virginia Polytechnic Institute and State University soil testing laboratory, and confirmed at the North Carolina State University soils testing laboratory. The sand, silt, and clay analysis was similar to the methods described by Day (2), and the organic matter analysis used the Walkley (7) procedure.

Adsorption/desorption procedure. The adsorption and desorption study conducted was similar to that described by other researchers (4, 6, 8). Formulated ICIA-0051 and ^{14}C-labeled ICIA-0051 (sp. act. 28.2 mCi/mmole) were combined to obtain an initial herbicide concentration of 1, 4, 8, and 12 ppmw. Each soil was air-dried and sieved through a 2-mm mesh screen, followed by sieving through a 707-micron screen. Adsorption isotherms were determined by placing 1 g of soil and 10 ml of herbicide containing solution at each concentration into 15-ml scintillation vials. Each treatment within a soil was replicated three times. The samples were shaken on a rotator at 24 to 26 C for 24 h. Preliminary work had indicated that herbicide solutions reached equilibrium with the soil within 6 to 12 h, so the 24-h period was selected to ensure equilibrium of all samples. Samples were then centrifuged at 2500 rpm for 15 min.

A 0.5-ml aliquot was taken from each vial and added to another 15-ml scintillation vial containing 12 ml of Ecolume[1] cocktail. The samples were counted with a Beckman LS-255[2] scintillation counter, until a 1.0% counting error was obtained. Quenching was checked by using the external channel ratio method and only the Davidson soil exhibited any quenching. A quenching curve was used to adjust for quenching in the Davidson soil, using a method described by

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[1] Ecolume. 1989. ICN Biomedicals, Inc. Radiochemicals Division. Irvine, CA.

[2] Beckman LS-255. 1974. Beckman Instruments, Inc. Irvine, CA.

Corbin (1). All samples were adjusted for the counting efficiency of the scintillation counter.

The resulting radioactivity in solution from the 0.5-ml aliquots were compared to 0.5-ml aliquots from the concentration standards. Differences between the amounts of ^{14}C-ICIA-0051 in the standard solutions and the supernatant of the samples were considered to be the amounts adsorbed.

Desorption was determined by using the same samples used for adsorption. A known quantity of the supernatant liquid was removed from each vial and replaced with herbicide-free water. The same procedure was followed for the desorption run whereby the vials were centrifuged and 0.5 ml was taken from each vial. Two desorption runs were conducted.

The adsorption data were described by the Freundlich equation $X = KC^{1/n}$, where X = adsorbed amount (ug/g), K and n = constants for a given soil, C = equilibrium concentration (ug/ml). The logarithmic form of the above equation was fitted by the method of least squares to the set of experimental data. The K and n constants were calculated, and linear regression analysis was performed to determine the degree of fit between the observed data and the Freundlich constants.

RESULTS AND DISCUSSION

Based on the K values, the greatest adsorption occurred in the Hyde silty clay loam soil, followed by the Frederick silt loam, Davidson clay, Bojac sandy loam, and the Appling loamy sand (Table 1). Correlation analysis indicated that the organic matter and/or humic matter content (r^2 = 0.99 and 0.98, respectively) strongly influenced the adsorption of ICIA-0051 in the soil (Table 2). The bulk densities of the soils were also positively correlated (0.87) with the adsorption of ICIA-0051. Since the organic and clay fractions help determine the soil's bulk density, this observation would support the high correlations obtained between the herbicide adsorption and the organic fractions.

The correlations for the Freundlich K constant of each soil with either the soil's clay content or the pH were low, 0.19 and 0.29, respectively. These correlations would indicate that the organic fraction of the soil and, more specifically, the more reactive and stable humic matter fraction, is possibly the primary factor responsible for the adsorption of ICIA-0051 across the soils investigated. This is in agreement with the findings of other researchers, who have demonstrated that the organic matter fraction is the primary adsorbant for several classes of herbicides such as triazines, phenylureas, and acetanilides (metolachlor) (3, 6, 9).

Although the structure of ICIA-0051 is unknown, its high affinity for organic matter and the relatively low affinity for clay is similar to other weak acids such as the phenoxy compounds (3). Other work by Wilson (10), demonstrated that increasing the organic matter was also negatively correlated with decreasing the mobility of ICIA-0051 in the soil. However, the pH and clay content were not strongly correlated with the mobility of ICIA-0051.

The 1/n values, which are the values for the slope of the line, were less than 1 for all of the soils examined (Table 1). This would indicate decreased adsorption of the herbicide as the adsorptive sites on the soil became occupied. The adsorption isotherms of ICIA-0051, derived by plotting the equilibrium concentration by the adsorbed amount of herbicide, indicated that these five soils give an L-type isotherm (Figure 1). This L-type isotherm is common for most herbicides in a given soil, and indicates a moderately high affinity between the soil and the herbicide. The Hyde soil, which was high in organic matter (11.7%), indicated a very high affinity for the herbicide. Based on the K values for the 1 ug/ml concentration, the percent adsorbed from the total amount in solution varied significantly by soil type. As a percent of the original amount in solution, the Appling soil adsorbed approximately 5%, while the Bojac, Davidson, and Frederick soils adsorbed approximately 10%, and the Hyde soil 70%. Similarly, the 1/n values also followed the same order of increase from the Appling (0.86) to the Hyde (0.96) soils.

Results of the first desorption indicated that, across all soils, from 20 to 50% of the previously adsorbed herbicide was lost from the soil particles (Table 3). The greatest loss, as a percentage of the amount originally adsorbed, occurred in the Davidson clay soil. This would indicate weak binding of ICIA-0051 to clay particles. The sandy soils such as Appling and Bojac, and also the higher organic matter soils, such as Frederick and Hyde, generally lost approximately 25% of the initially adsorbed herbicide. As a percent of the original amount adsorbed, all soils in the second desorption run had less herbicide loss (Table 3). The average losses for the second desorption ranged from 6% (Bojac) to 20% (Hyde). Regardless of the desorption run, the Hyde soil retained a larger total amount of herbicide per gram of soil, compared to all other soils. The general order of retention after two desorptions was: Hyde > Frederick > Davidson = Bojac > Appling. This supports the theory that the organic matter fraction and not the other soil properties such as pH and clay content is the primary soil property involved in the retention of ICIA-0051.

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Table 1. Freundlich constants of ICIA-0051 in the five soils studied.

Soil			
Series	Texture	K	1/n
Appling	loamy sand	0.63	0.86
Bojac	sandy loam	0.98	0.89
Davidson	clay	0.98	0.92
Frederick	silt loam	1.02	0.91
Hyde	silty clay loam	7.04	0.96

Table 2. Correlation coefficients for the adsorption of ICIA-0051 in five selected soils.

Soil property	Correlation
	(r^2)
Organic matter (%)	0.99
Humic matter (%)	0.98
Bulk density	0.77
pH	0.29
CEC	0.24
Clay content	0.19
Base saturation	0.07

Table 3. Amount of ICIA-0051 bound to the soil particles after the initial adsorption and two desorptions.

Soil type	Adsorption/ desorption	Initial ICIA-0051 concentration, ug/ml 1	4	8	12
		(ug/g)			
Appling loamy sand	A1	0.63	2.09	3.78	5.36
	D1	0.54	1.69	3.12	4.78
	D2	0.47	1.46	2.66	4.09
Bojac sandy loam	A1	0.98	3.35	6.21	8.91
	D1	0.69	2.58	4.34	6.08
	D2	0.62	2.13	3.60	4.98
Davidson clay	A1	0.98	3.50	6.60	9.59
	D1	0.49	2.26	3.55	5.79
	D2	0.40	1.90	2.75	4.46
Frederick silt loam	A1	1.02	3.60	6.77	9.80
	D1	0.87	2.95	5.68	8.26
	D2	0.73	2.30	4.61	6.83
Hyde silty clay loam	A1	7.04	26.64	51.82	76.48
	D1	5.70	21.01	40.89	60.71
	D2	4.14	15.04	29.74	44.65

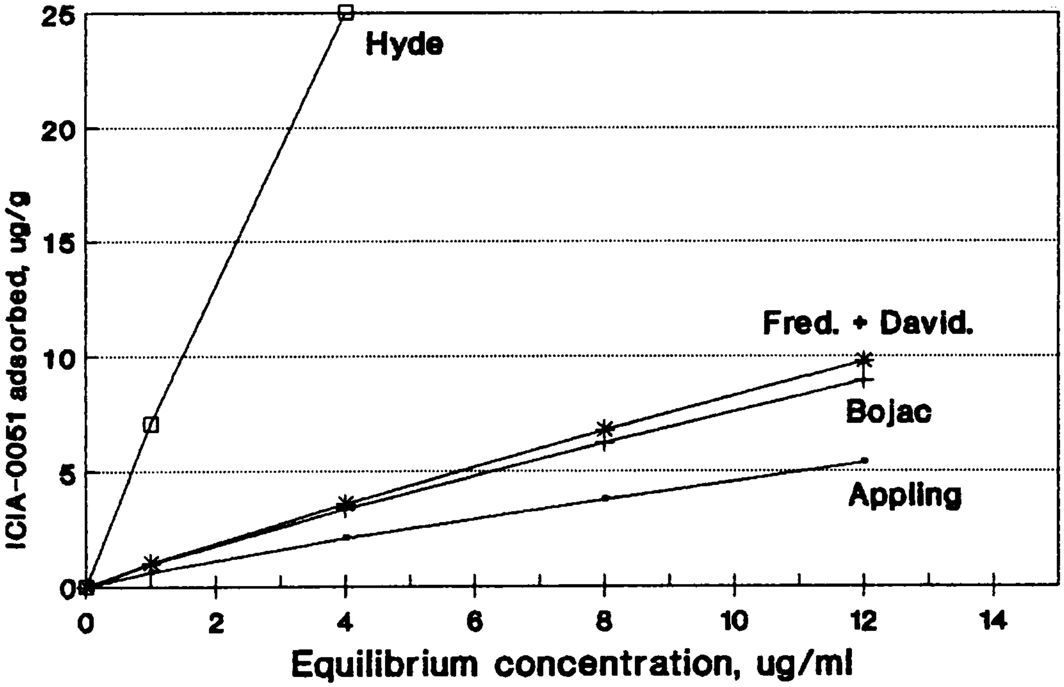

Figure 1. The adsorption of ICIA-0051, at various concentrations, onto five selected soils.

V. PHYTOTOXICITY AND SOIL PERSISTENCE OF ICIA-0051 AS INFLUENCED BY SOIL PROPERTIES.

Abstract. A greenhouse bioassay procedure was used to determine the persistence and biological activity of ICIA-0051 in various soil types. The soils selected were Appling loamy sand, Bojac sandy loam, Davidson clay, Frederick silt loam, and Hyde silty clay loam, predominant corn (Zea mays L.) soils found in Virginia. These soils represented a wide range of organic matter, clay content, pH, and other soil properties. Results from the three cropping periods indicated that ICIA-0051 was more biologically available over time than atrazine in the soils investigated. The order of persistence and availability for ICIA-0051 in the soils as measured by the reduction of crop growth was Appling, followed by Davidson, Bojac, Frederick, and Hyde. The availability of ICIA-0051 appeared to be related to the organic matter, whereby increasing the organic matter decreased the persistence (biological activity) of the herbicide. In all the soils, crop injury decreased over time, particularly at the lower rates, although at the higher rate investigated (1 ppm), significant crop injury occurred after 6 months. The mustard (Brassica kaber L.) growth as a percent of the check, for each soil at the 1 ppm rate after 6 months, was Appling (20%), Davidson (22%), Bojac (33%), Hyde (68%), and Frederick (72%).

INTRODUCTION

The soil persistence and biological activity of a herbicide can influence its potential for carryover to sensitive crops and/or groundwater contamination. Ideally, a preemergence herbicide should persist long enough in the soil to give adequate season-long control or allow the crop canopy to form, but dissipate soon enough to avoid carryover to the next crop. However, due to various soil, environmental, and herbicide interactions, many herbicides have restricted crop rotations due to herbicide residues which persist after the crop is harvested.

Although field testing with ICIA-0051 has been conducted for several years at the university level, little is known about the persistence of this herbicide in the soil. Mayonado (4) reported that in a field experiment from the previous year, where the soil pH of some plots had been amended, lowering the pH increased the persistence of ICIA-0051. However, the influence of organic matter, clay content, and other soil properties on the persistence of ICIA-0051 is currently unknown.

The objective of this study was to investigate the persistence of ICIA-0051, by measuring the biological availability, on a wide variety of soil types under different soil conditions. A soil bioassay procedure was used to measure the soil residue levels of ICIA-0051 and atrazine in five corn soils from Virginia at 0, 3, and 6 months after herbicide application.

MATERIALS AND METHODS

Soil selection and analysis. Five predominant Virginia corn soils were selected which represented a wide range of organic matter, humic matter, clay content, pH, cation exchange capacity, and other soil properties. The soil series selected were Appling loamy sand (clayey, kaolinitic, thermic Typic Hapludult), Bojac sandy loam (coarse-loamy, siliceous, thermic Typic Hapludult), Davidson clay (clayey, kaolinitic, thermic Rhodic Paleudult), Frederick silt loam (clayey, mixed, thermic Typic Paleudult), and Hyde silty clay loam (fine-silty, mixed, thermic Typic Umbraquult). The organic matter and humic matter, respectively, in these soils were as follows: Hyde 11.7 %, 3.7 g/cm^3; Frederick 2.5%, 0.7 $g/100cm^3$; Bojac 2.1%, 0.5 $g/100cm^3$; Davidson 1.9%, 0.2 $g/100cm^3$; and Appling 1.2 %, 0.2 g/cm^3. The pH ranged from 4.5 in the Hyde soil to 6.7 in the Davidson, while the clay content ranged from 5% (Appling and Bojac) to 48% (Davidson).

Persistence studies and herbicides used. The greenhouse procedure used in this experiment was similar to one described by Lavy and Santelmann (1). Each soil was air-dried and than sieved through a 10-mm mesh screen, followed by sieving through a 2-mm mesh screen. Either 500 g of each soil, or 600 g in the case of Appling, were weighed out and placed on a plastic sheet for mixing. Herbicide treatments consisted of ICIA-0051 and a standard, atrazine. Atrazine was chosen as a reference herbicide because of previous persistence work done with atrazine on these same soils, and also because atrazine is often tank-mixed with ICIA-0051 in field research trials (5, 6). ICIA-0051 and atrazine were applied to each soil in a diluted water mixture at 75 ml for each soil, except 90 ml for Appling, which gave herbicide concentrations of 0.125, 0.25, 0.5, and 1 ppm. The 0.25 and 0.5 ppm rates were equilivalent to 0.6 kg/ha and 1.1 kg/ha, respectively, which are recommended preemergence and postemergence field rates. Each herbicide was thoroughly mixed into the soil and then placed in 475-ml plastic cups. The cups were 12 cm in height, with a top diameter of 8 cm, and no holes were cut in the bottoms. An untreated check was also included for each soil. Thirteen seeds of mustard, a sensitive bioassay species, were planted in each cup. The plants were watered each day to maintain adequate growth. All treatments were replicated three times.

Harvesting and timing. After one week, the plants were thinned to 10 seedlings per cup. The plants were grown for three weeks, harvested and dry weights taken. To test the persistence of these herbicides in each soil, the experiment was conducted at 0, 3, and 6 months after application. The 3- and 6-month recroppings involved mixing the harvested plant residues back into the soil and then replanting the indicator species into each cup as previously described. The experiment was repeated twice, although only the 0 and 3-

month croppings were repeated for the second experiment. To evaluate the rate and timing effect, regression models were fitted to each herbicide by soil type and correlations of determination were calculated.

RESULTS AND DISCUSSION

The regression analysis indicated that the best fitted equation for the relationship between herbicide concentration and crop injury was a quadratic curve (Table 1 and 2). R^2 values for both ICIA-0051 and atrazine, within each soil, particularly at the initial planting (0 month), were generally above 80% (Table 1 and 2). However, the r^2 values did decrease in the 3- and 6-month harvests indicating less consistency within the treatments over time.

ICIA-0051, at the 0.5 and 1 ppm rate, caused significant injury to the bioassay plants with all the soils, even after 6 months (Tables 1 through 4). The general order of injury to the mustard bioassay species for both ICIA-0051 and atrazine, from most to least injury, was Appling, followed by Davidson, Bojac, Hyde, and Frederick (Figures 11 and 12). After 6 months, plant growth for each soil at the 1 ppm concentration of ICIA-0051 as a percent of the check was Appling (20%), Davidson (22%), Bojac (33%), Hyde (68%), and Frederick (72%) (Figure 1 through 5). Similarly, atrazine after 6 months at the 1 ppm rate, gave the following plant growth compared to the check for that soil: Davidson (19%), Bojac (34%), Appling (34%), Frederick (48%), and Hyde (128%) (Figures 6 through 10). Based on the biological activity of ICIA-0051 against mustard, a higher potential for crop injury exists in the Bojac sandy loam, Appling loamy sand, and the

Davidson clay soils, due to carryover of herbicide residues. Wilson (7) demonstrated that a positive correlation existed between the soil organic matter and the adsorption of ICIA-0051, while a negative correlation existed between the organic matter level and mobility of the herbicide. Since the cups had no holes in the bottom and allowed no drainage, thus restricting herbicide loss due to leaching, ICIA-0051 persistence was apparently related more to herbicide adsorption to the organic matter. Thus, in a field situation, injury to sensitive rotational crops, due to ICIA-0051 residues, might be enhanced in sandy or high clay soils (low in organic matter) under dry-season conditions, where little or no herbicide leaching occurs.

The persistence of atrazine also appeared to be strongly negatively correlated with the organic matter content of the soil. Previous work by Foy and Wilson (2), using three triazine herbicides applied to the Appling, Bojac, Davidson, and Frederick soils, indicated that significant crop injury to oats (Avena sativa L.) occurred, at the 0.5 and 1 ppm rate in the Davidson soil after 12 months. No injury to the bioassay species was reported in the other soils after the 12-month period (2).

Based on the dose response curves at 0 month, the bioassay species showed a similar sensitivity to ICIA-0051 and atrazine at equal soil concentrations. Comparing plant injury caused by ICIA-0051 and atrazine across all soils, the indication was that ICIA-0051 persisted longer than did atrazine. This difference in injury to mustard between ICIA-0051 and atrazine was most noticeable at the 3- and 6-month harvests in the Bojac soil series (Figures 2 and 7).

Plant growth at the lowest rate of ICIA-0051 (0.125 kg/ha) at 3 months was equal to the check plot, e.g. 95% or better, in the Hyde, Frederick, and Appling soils, while more crop injury occurred in the Bojac and Davidson soils, where growth equalled 75 and 80% of the check, respectively. Therefore, ICIA-0051 residue levels might restrict replanting of sensitive crops after corn at the current preemergence use rates where crop failure has occurred. After 6 months, no significant crop injury occurred with ICIA-0051 in the Hyde and Frederick soils at the 0.125, 0.25, and 0.5 ppm rate, or in the Appling, Bojac and Davidson soils at the 0.125 rate. In fact, within many soils, the treated cups out-yielded the checks, indicating a possible stimulation effect. Work done with triazine herbicides also has shown that, at low concentrations, these herbicides can stimulate growth (3).

Further research in this area is needed to investigate the long term effects (12 months or more after application) of ICIA-0051 compared to atrazine. Mayonado (4) showed that lowering the soil pH increased the availability of ICIA-0051 after 12 months in an amended field site.

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Table 1. Regression models for mustard shoot dry weights at three time periods after application of ICIA-0051 at various rates in greenhouse studies.

Soil type	Timing, after application	Equation[a]	r^2
Appling	0	$Y = 1.23 - 3.76X + 2.57X^2$	0.92
	3	$Y = 1.95 - 6.23X + 4.44X^2$	0.75
	6	$Y = 1.29 - 2.87X + 1.79X^2$	0.94
Bojac	0	$Y = 0.45 - 1.23X + 0.79X^2$	0.79
	3	$Y = 2.36 - 7.06X + 4.72X^2$	0.85
	6	$Y = 0.90 - 1.22X + 0.60X^2$	0.74
Davidson	0	$Y = 0.43 - 0.56X + 0.29X^2$	0.84
	3	$Y = 0.96 - 1.99X + 1.28X^2$	0.91
	6	$Y = 1.17 - 2.37X + 1.43X^2$	0.81
Frederick	0	$Y = 0.57 - 0.90X + 0.44X^2$	0.85
	3	$Y = 1.08 - 1.47X + 0.56X^2$	0.90
	6	$Y = 1.05 - 0.15X - 0.17X^2$	0.60
Hyde	0	$Y = 1.04 - 1.37X + 0.79X^2$	0.72
	3	$Y = 1.00 + 0.30X - 0.82X^2$	0.63
	6	$Y = 1.23 - 0.06X - 0.49X^2$	0.73

[a] Y represents mustard shoot dry weight as a percent of that observed from the check. X represents the concentration of ICIA-0051 in ppm.

Table 2. Regression models for mustard shoot dry weights at three time periods after application of atrazine at various rates in greenhouse studies.

Soil type	Timing, after application	Equation[a]	r^2
Appling	0	$Y = 0.58 - 1.30X + 0.78X^2$	0.88
	3	$Y = 2.22 - 6.03X + 4.11X^2$	0.88
	6	$Y = 1.71 - 3.07X + 1.71X^2$	0.95
Bojac	0	$Y = 0.75 - 1.99X + 1.31X^2$	0.92
	3	$Y = 2.26 - 3.27X + 1.67X^2$	0.74
	6	$Y = 1.11 - 0.33X - 0.45X^2$	0.80
Davidson	0	$Y = 0.39 - 0.42X + 0.22X^2$	0.77
	3	$Y = 1.74 - 4.07X + 2.53X^2$	0.80
	6	$Y = 1.72 - 4.69X + 3.18X^2$	0.82
Frederick	0	$Y = 0.85 - 2.02X + 1.32X^2$	0.92
	3	$Y = 1.97 - 4.22X + 2.40X^2$	0.76
	6	$Y = 1.10 - 1.63X - 0.60X^2$	0.87
Hyde	0	$Y = 0.92 - 0.77X + 0.30X^2$	0.70
	3	$Y = 1.44 + 0.30X - 1.82X^2$	0.81
	6	$Y = 1.50 - 0.26X$	0.55

[a] Y represents mustard shoot dry weight as a percent of that observed from the check. X represents the atrazine concentration in ppm.

Table 3. Correlation coefficients for the persistence of ICIA-0051 in five selected soils.

Soil property	Correlation
	(r^2)
Organic matter (%)	0.67
Humic matter (%)	0.70
Bulk density	0.66
pH	0.20
CEC	0.30
Clay content	0.30
Base saturation	0.02

Table 4. Regression models describing the persistence of ICIA-0051 and atrazine at the 0.5 ppm rate as a function of time.

Soil type	Herbicide	Equation[a]	r^2
Appling	ICIA-0051	$Y = 3.3 + 4.4X$	0.94
Bojac	ICIA-0051	$Y = 2.9 + 6.7X$	0.93
Davidson	ICIA-0051	$Y = 21.9 + 3.2X$	0.98
Frederick	ICIA-0051	$Y = 22.3 + 11.7X$	0.99
Hyde	ICIA-0051	$Y = 64.4 + 6.1X$	0.96
Appling	atrazine	$Y = 11.9 + 7.7X$	0.99
Bojac	atrazine	$Y = 15.2 + 18.4X$	0.96
Davidson	atrazine	$Y = 20.3 + 4.3X$	0.96
Frederick	atrazine	$Y = 16.2 + 12.5X$	0.96
Hyde	atrazine	$Y = 73.3 + 12.5X$	0.88

[a] Y represents the growth parameter as a percent of that observed from the check. X represents the time in months.

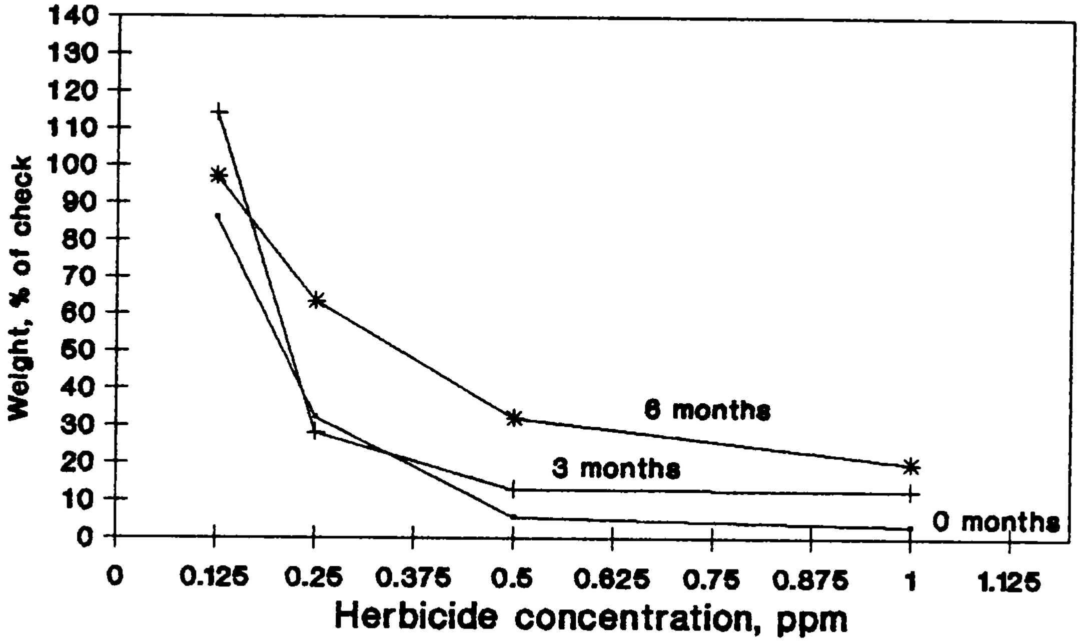

Figure 1. Mustard shoot dry weight in the Appling soil series at 0, 3, and 6 months after application of ICIA-0051 at various rates in greenhouse studies.

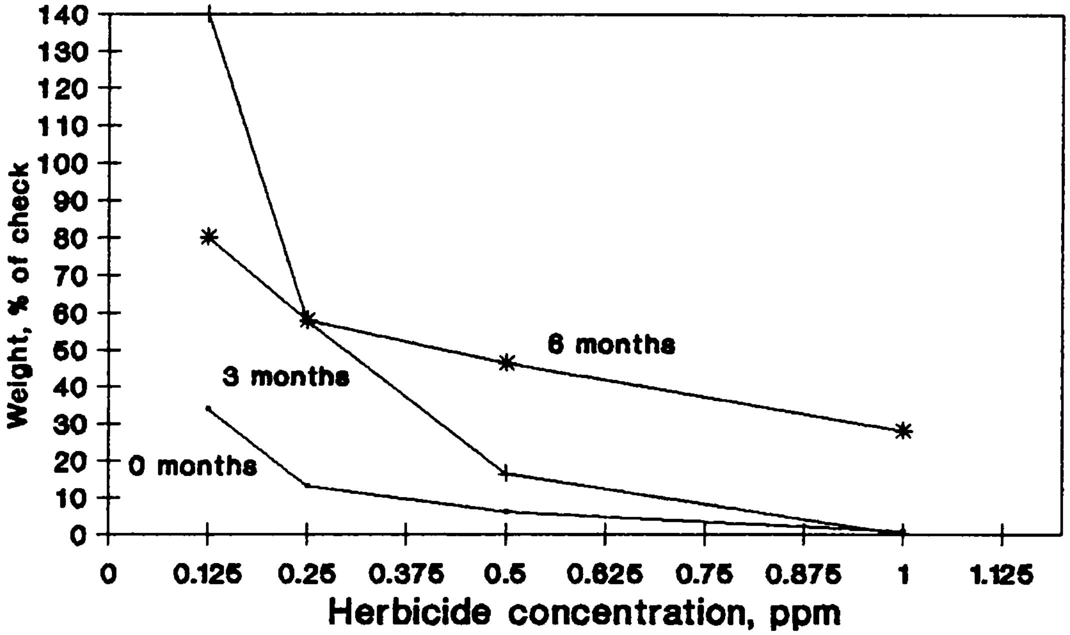

Figure 2. Mustard shoot dry weight in the Bojac soil series at 0, 3, and 6 months after application of ICIA-0051 at various rates in greenhouse studies.

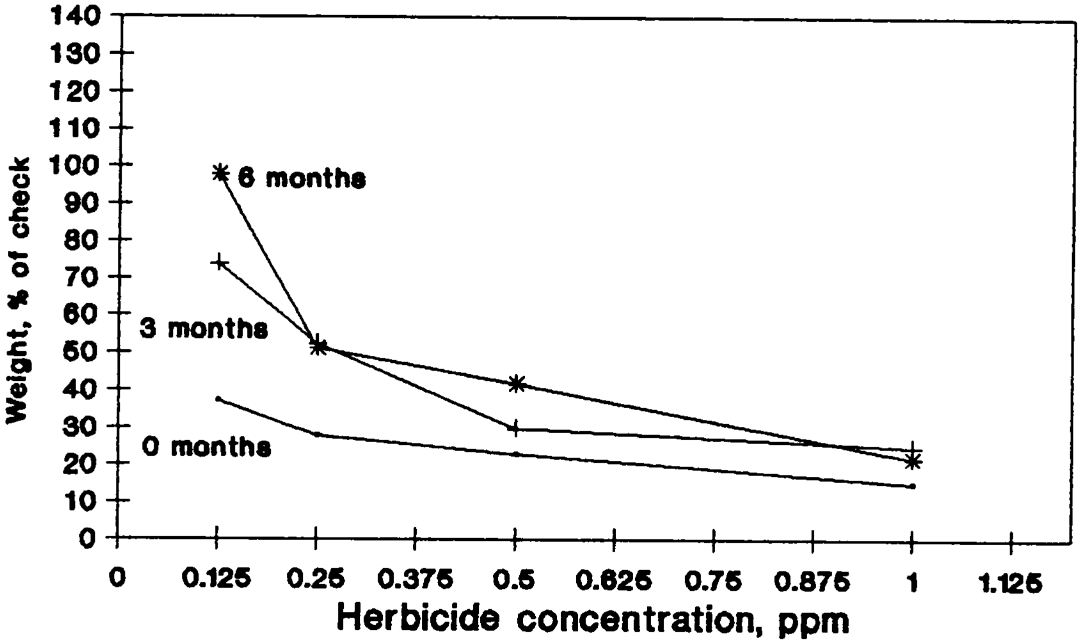

Figure 3. Mustard shoot dry weight in the Davidson soil series at 0, 3, and 6 months after application of ICIA-0051 at various rates in greenhouse studies.

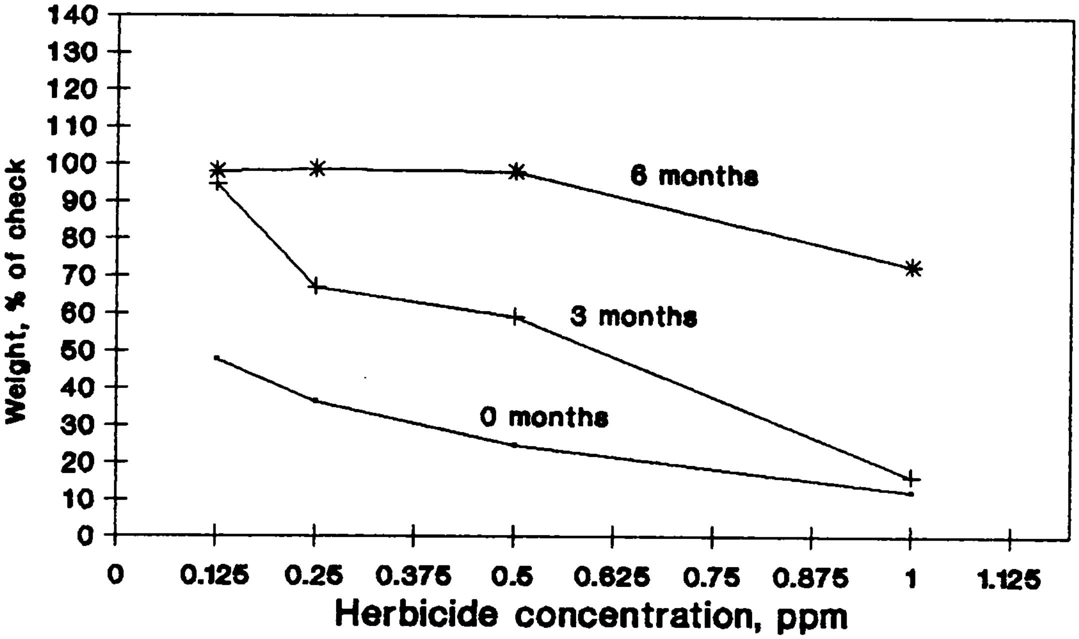

Figure 4. Mustard shoot dry weight in the Frederick soil series at 0, 3, and 6 months after application of ICIA-0051 at various rates in greenhouse studies.

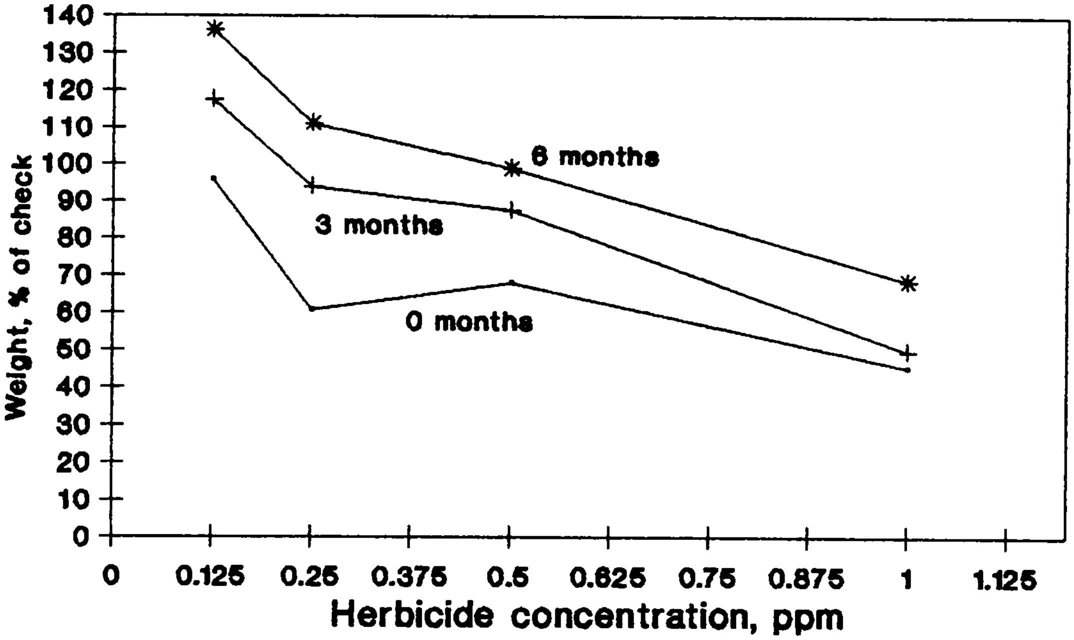

Figure 5. Mustard shoot dry weight in the Hyde soil series at 0, 3, and 6 months after application of ICIA-0051 at various rates in greenhouse studies.

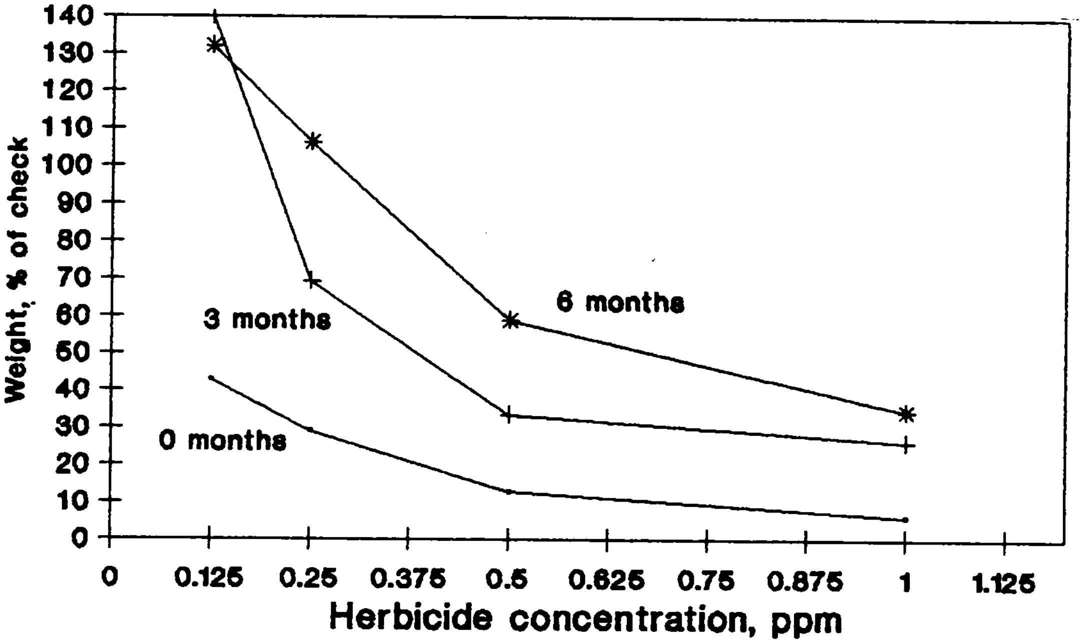

Figure 6. Mustard shoot dry weight in the Appling soil series at 0, 3, and 6 months after application of atrazine at various rates in greenhouse studies.

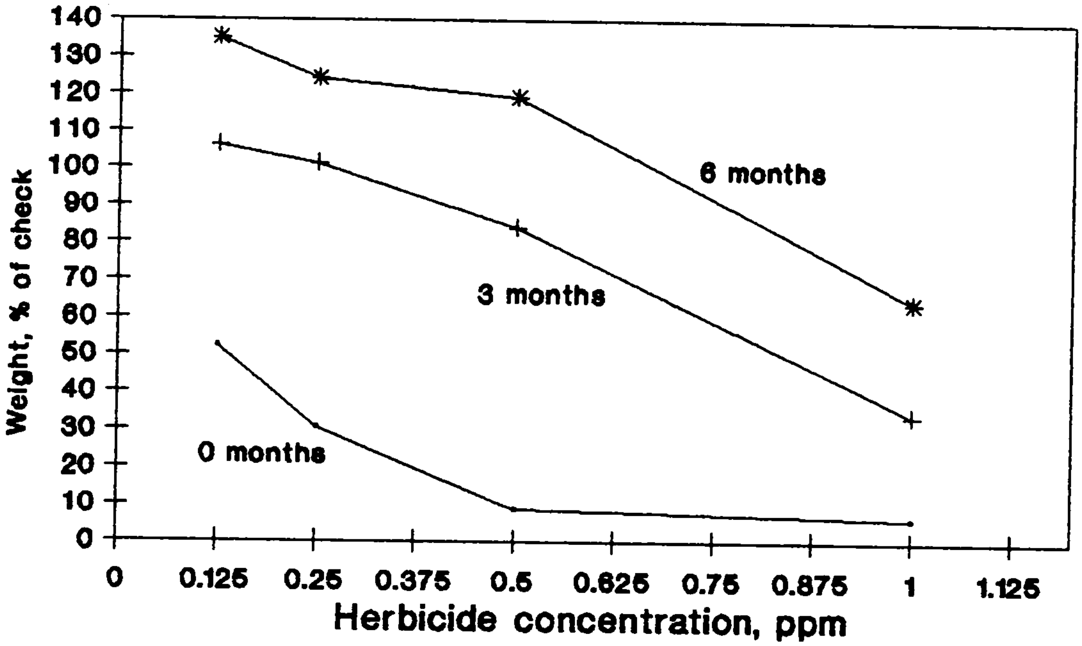

Figure 7. Mustard shoot dry weight in the Bojac soil series at 0, 3, and 6 months after application of atrazine at various rates in greenhouse studies.

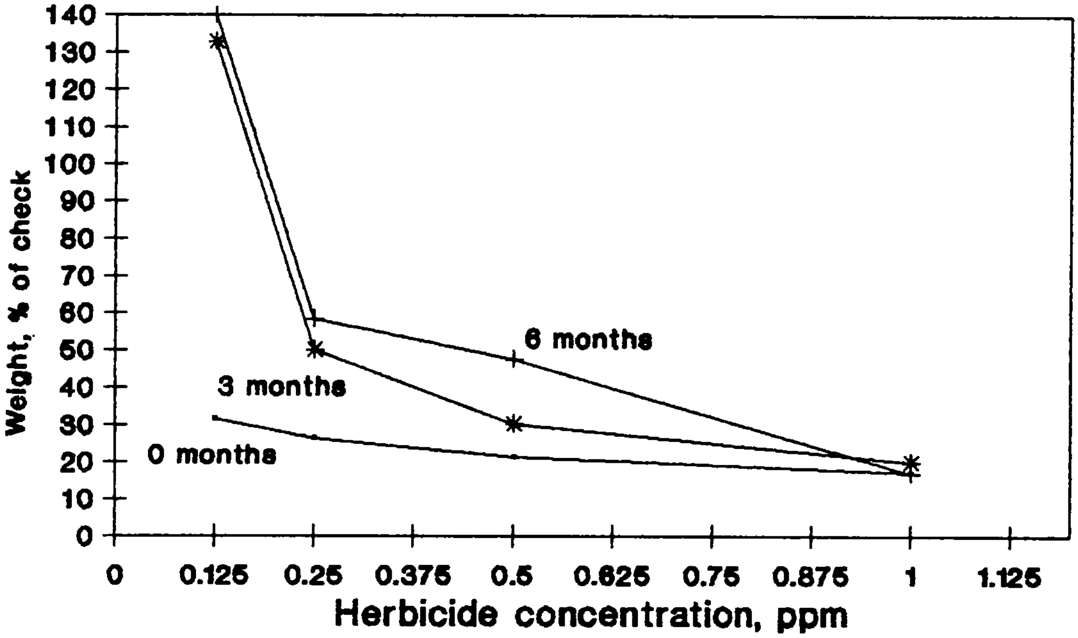

Figure 8. Mustard shoot dry weight in the Davidson soil series at 0, 3, and 6 months after application of atrazine at various rates in greenhouse studies.

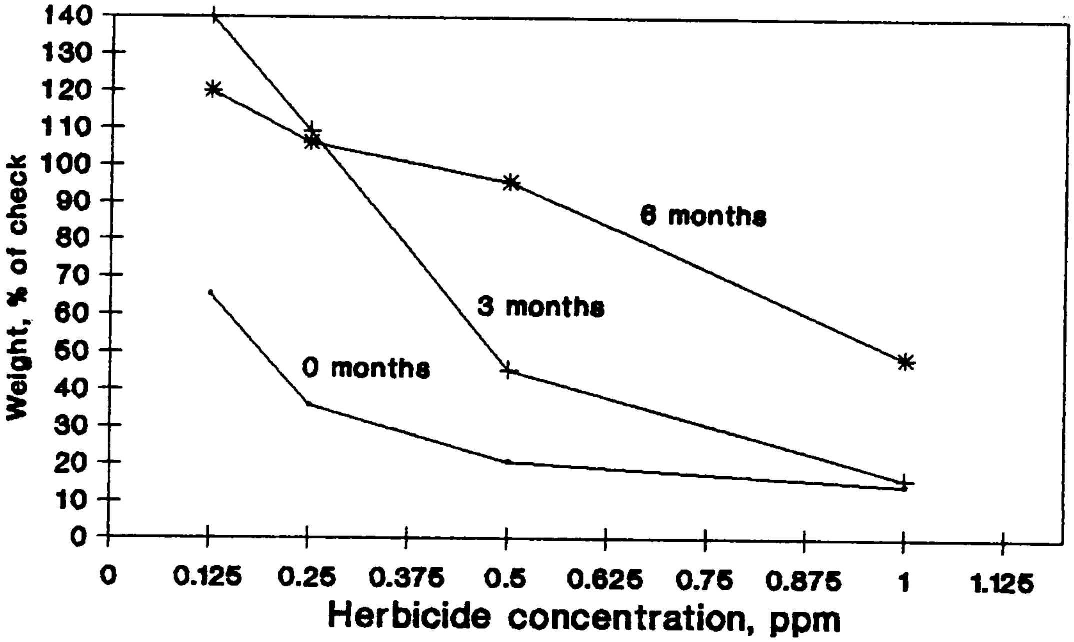

Figure 9. Mustard shoot dry weight in the Frederick soil series at 0, 3, and 6 months after application of atrazine at various rates in greenhouse studies.

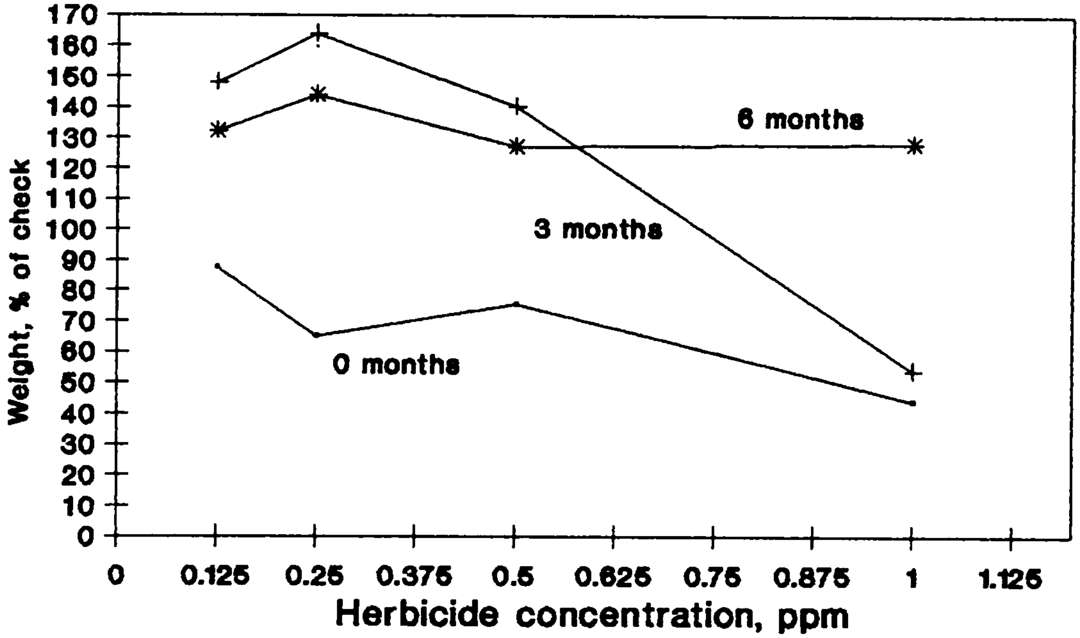

Figure 10. Mustard shoot dry weight in the Hyde soil series at 0, 3, and 6 months after application of atrazine at various rates in greenhouse studies.

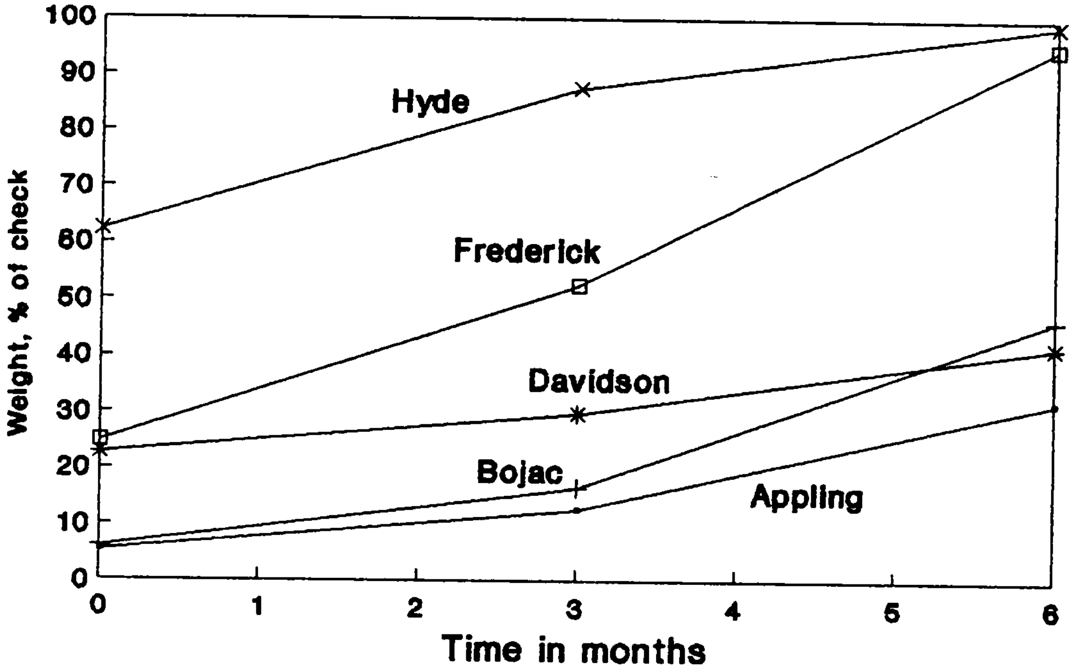

Figure 11. The persistence of ICIA-0051 in all soils at the 0.5 ppm rate over time.

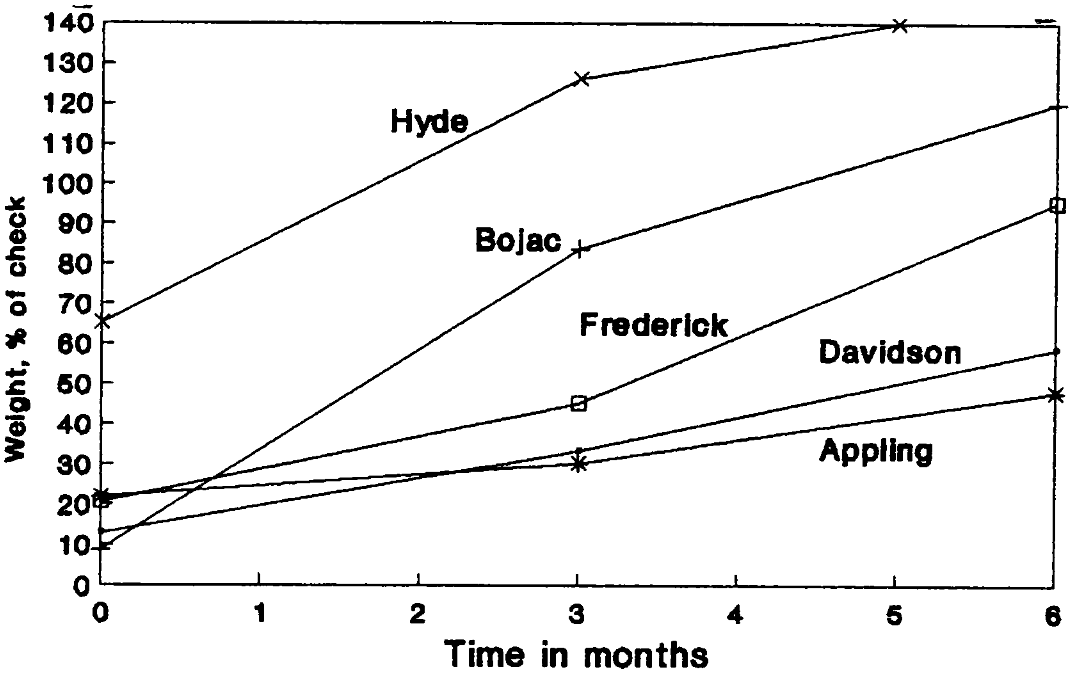

Figure 12. The persistence of atrazine in all soils at the 0.5 ppm rate over time.

SUMMARY AND CONCLUSION

The field efficacy and availability, movement, and persistence of ICIA-0051 herbicide in various soils was investigated. The weed control efficacy of ICIA-0051 and SC-0774 in conventional and no-till systems of corn (*Zea mays* L.) was determined. Results of the preemergence and postemergence applications of ICIA-0051 indicated good control of triazine-resistant smooth pigweed, as well as adequate control of grasses and some broadleaf weeds. SC-0774 gave excellent grass control, but poor control of broadleaf weeds. Because of the ability of ICIA-0051 to control weed species such as triazine-resistant smooth pigweed, which is not currently controlled by standard treatments, it has a good potential for development in the corn markets. However, in many cases, ICIA-0051 may need to be tank mixed with other labeled herbicides (e. g. atrazine), to provide adequate control of many broadleaf species. If ICIA-0051 is to be developed further, it may need to be marketed selectively in problem areas where standard treatments do not provide adequate weed control, or formulated in combination with other herbicides. Although SC-0774 provided adequate grass control its ineffectiveness against broadleaf species, and the potential for crop injury, will limit its development.

The soil mobility, adsorption/desorption, and persistence studies all indicated the organic/humic fraction of the soil was strongly correlated with the behavior of this herbicide. Increasing the organic/humic matter content would thus decrease the availability and mobility of this herbicide. Consequently, soils high in organic matter, such as the midwest soils, might have decreased weed control or a higher potential for carryover. Soil mobility studies also indicated that in most soils investigated both ICIA-0051 and SC-0774 were more mobile than atrazine. This potential to leach, particularly in soils high in sand or clay, might cause groundwater contamination or reduce the season long effectiveness of these herbicides in high rainfall areas or during wet growing seasons. The desorption studies also supported this, since sandy and clayey soils readily released adsorbed ICIA-0051.

Other soil factors such as clay content and pH were only weakly correlated with the herbicide's behavior in the soil. Nevertheless, many soils in the southeast are low in organic matter, and thus other soil factors should also be considered.

ICIA-0051 and atrazine showed similar persistence in the soils investigated, with significant herbicide residues being detected in most of the soils after 6 months. Although no cases of ICIA-0051 carryover have been reported, the potential exists for crop injury, which might limit rotations to sensitive species (e.g. soybeans) following corn.

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