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Date July 30, 1993

TRANSPORT OF IMAZAQUIN THROUGH UNDISTURBED COLUMNS

A Thesis

Presented for the

Master of Science

Degree

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Dolores Marie Chandler

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ABSTRACT

The movement of imazaquin [2-(4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1H-imidazol-2-yl)-3-quinoline-carboxylic acid] through large columns amended with wheat straw was studied.

A tractor-mounted hydraulic sampler extracted six large columns, which were mounted vertically in the laboratory. Wheat straw was applied at the rates of 0 and 4480 kg ha^{-1}. Imazaquin was applied at the recommended rate with pulse and duration times predicted by the FORTRAN program CXTFIT. Bromide was applied as the non-reactive tracer, and used to determine dispersion throughout the columns. This dispersion coefficient was used to determine the retardation factor for imazaquin. The bromide was measured by an ion specific electrode. The imazaquin in the leachate, soil and wheat straw was analyzed by HPLC.

The results showed that imazaquin in the soil and in the wheat straw was at or below detection limits. The imazaquin present in the leachate was in low concentrations over a period of 54 days. Mass balance calculations for the leachate accounted for 43% to 100% of the applied imazaquin. The retardation factor solved for by CXTFIT was higher than predicted using the current literature.

Preferential flow paths were qualitatively analyzed using Rhodamine B dye. The staining patterns were photographed. Preferential flow paths were evident to a depth of 30 - 40 cm.

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CHAPTER I

INTRODUCTION

In the United States, over 50 percent of the total population and 97 percent of the rural population obtain their drinking water from groundwater (Driscoll, 1986). Contamination of this resource by pesticides and fertilizers is a national concern. In response to public and political pressure the United States Department of Agriculture and the Environmental Protection Agency (EPA) have focused their research efforts on assessing the impact of agricultural pesticides and fertilizers on surface and groundwater quality in the United States (Fairchild, 1987). A national survey of drinking water wells conducted by the EPA reported that of the 1347 wells tested, 10 percent of community drinking water wells and 4 percent of the rural domestic water wells had detectable residues of at least one pesticide (USEPA, 1990).

Concern over pesticide contamination of groundwater continues to rise due to increased pesticide usage. In addition, fear over pesticide contamination continues to build as our analytical means result in increasingly lower detection limits. There is also concern that some tillage practices may promote rapid solute infiltration and percolation of pesticides through the soil and into the groundwater (Steenhuis et al., 1990).

Since the publication of Rachael Carson's book *Silent Spring* in 1962, in which she dramatically details the effects of surface water contamination by pesticides that are strongly adsorbed to soil particles, pesticides have been developed that are more rapidly degraded and more mobile. As of 1990, United States pesticide usage had increased to 1 billion kg per year of more than 400

types (Steenhuis et al., 1990). There is concern that pesticide usage may increase with some types of conservation tillage (Helling, 1987).

Conventional tillage practices include many trips across the field for seedbed preparation, fertilization, maintenance of weeds and harvesting. Under conservational tillage, increased pesticide usage reduces the number of trips across the field, resulting in less disturbance of the soil. This minimal soil disturbance preserves the integrity of macropores created by animals and root activity and the soil's structural characteristics. With this increase in macroporosity, there is a probability of increasing solute leaching. During rain events water tends to flow through these macropores and tends to bypass the mass of soil (Thomas et al., 1989). Projections for the year 2010 indicate close to 95 percent of U.S. cropland will include some form of conservation tillage (Myers, 1983). In the southeastern U.S., contamination of groundwater by pesticides may be high due to the presence of well-structured soils. Preferential flow through macropores may decrease reactivity with soil and decrease the travel time of solutes, thereby increasing its likelihood to contaminate groundwater. Ehlers (1975) noted the differences in infiltration and hydraulic conductivity in soils managed under no till verses those managed under more intensive tillage. These differences were attributed to movement of water through large, surface connected, continuous voids or pores in the soil. This phenomena, also referred to as preferential flow has been used to explain the rapid movement of solutes though the soil (Bicki and Guo, 1989).

Preferential flow may be an important factor in the movement of herbicides in the soil. Imazaquin, (Figure 1) whose trade name is Scepter, is an imidazolinone herbicide introduced in 1986 as a soybean herbicide. It is reported by American Cyanamid to have limited movement in the soil. Because

of the presence of acidic and basic functional groups on imazaquin it is amphoteric. The ionizable carboxyl group has a pKa of 3.8.

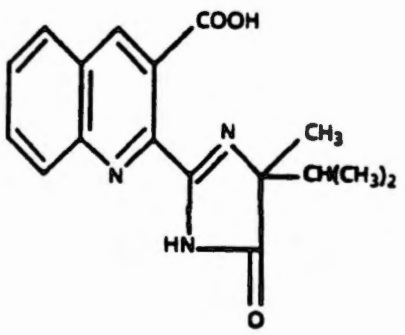


Figure 1. The chemical structure of imazaquin.

At lower a pH, the nitrogen groups on imazaquin may protonate, resulting in a positively charged compound which may bind to soils (Loux et al., 1989). Imazaquin has a molecular weight of 311.3, and a water solubility of 60 to 120 mg L^{-1} (Anonymous, 1986). In general pesticides which have a water solubility greater than 30 mg L^{-1} are likely to leach (Pionke et al., 1988). In a study by Basham and Lavy the half life calculations ranged from 40 days to 11.7 months.

Six variables influence the fate of a herbicide in the soil. These variables include: decomposition as a result of chemical, microbial and light interactions; movement either by plant uptake, volatilization or gravitation; and adsorption.

Adsorption occurs at the clay and/or organic matter surfaces in the soil. In the organic fraction of the soil, adsorption involves interactions with functional groups such as carboxyl, amino and phenolic hydroxyl (Bailey and White, 1970). These interactions are dependent on the nature of the pesticide and the

properties of the soil system. In a study by Loux et al. (1989) the effects of pH on adsorption were observed. At a lower pH, imazaquin's nitrogens protonate resulting in a positively charged molecule which was adsorbed to the ion exchange resin representing the organic fraction of the soil. (Loux, et al., 1989). Generally, increasing soil clay and organic matter content and decreasing soil pH results in reduced activity and reduced mobility of imazaquin (Basham et al., 1987). The lack of adsorption at a near-neutral pH, therefore, could increase the mobility of imazaquin. Wolt et al. (1989) studied the effect of wheat straw incorporation in soil on imazaquin concentration in soil solution. The study simulated what may occur in the soils of the Southeast where reduced tillage systems cause increases in organic matter. Their finding showed imazaquin concentrations were higher in the soil solution than on the adsorption sites. The addition of wheat straw increased solution pH, which earlier reduced adsorption (Wolt et al., 1989). Again the reduction in adsorption could increase imazaquin mobility in soil.

Curran et al. (1992) reported losses by photolysis, or photodegredation to be significant for imazaquin. In this study, using sandy loam (mixed, mesic Typic Udipsamments) herbicide solution was applied to the surface and incubated in a polystyrene tissue culture dish, which was covered and sealed with parafilm. The samples were irradiated for periods of 0, 24, 48 and 120 hours. Because there is a relationship between water content and degradation, this sand and a silty clay loam (fine-silty mixed mesic Typic Haplaquoll) were wetted to a soil water potentials of -10 and -3100 kPa. These soil water potentials corresponded to each soil's water content at field capacity and air dryness, respectively. These were also treated with the herbicide and irradiated for 48 hours. The samples which remained in the dark did not show any difference in imazaquin

concentration throughout the experiment. Therefore, all observed losses were from photodegradation only. The half life of the imazaquin on the sand was 88 hours. In the experiment relating water potentials to losses, the half life of imazaquin was 48 hours. Their study showed greater losses in sand than in silty clay loam soil due to less possibility of adsorption. Herbicide losses were low on both of the air dried soils (Curran et al., 1992).

Basham and Lavy (1988) reported the relationship of microbial degradation and imazaquin availability. Imazaquin was found to be available at higher water contents and therefore more accessible for assimilation by microorganisms. They determined the evolution of $^{14}CO_2$ from five soils (Typic Albaqualf, Mollic Albaqualf, Typic Ochraqualfs Typic Ustifluvent, and a Vertic Haplaquepts) treated with labeled ^{14}C-imazaquin. The soils were incubated for 7 months under a constant water potential (-33 kPa) and temperature (24 C). Their results showed imazaquin degraded most rapidly when incubated at the higher temperature and water content, therefore suggesting that microbial decomposition was a major source of loss from the soil (Basham and Lavy, 1988).

The objectives of this research are to determine imazaquin mobility in intact soil columns and to evaluate the effect of applied wheat straw on imazaquin fate and transport.

CHAPTER II

LITERATURE REVIEW

Leaching, volatilization, photodegradation, microbial degradation and runoff are the five ways in which a chemical may leave the zone of incorporation (Bailey and White, 1970). Leaching is the downward movement of the dissolved chemical. Volatilization is the loss of the chemical from the soil or plant to the atmosphere. Photodegradation and microbial degradation transform the parent chemical to a chemical with different properties due to exposure to light or microbial activity, respectively (Jury and Valentine, 1986).

The potential of a pesticide to leach and enter groundwater is dependent on several variables. In general, pesticides which have a water solubility greater than 30 mg L^{-1}, an adsorptivity (Koc) less than 300 to 500, a soil half life (T 1/2) of greater than 21 days, and are negatively charged have the greatest potential to leach toward groundwater (Pionke et al., 1988).

The field variables which affect the potential of a chemical to leach through the soil include many factors. Among the most important, with respect to application, are the pesticide concentration, timing of application with regard to rainfall and soil hydrologic conditions, and relative intensity of rainfall following application. Soil factors which affect the potential of a chemical to leach include soil water content, pore size distribution, adsorption site density, and depth to groundwater (Jury, 1986; Jury and Valentine, 1986).

Imazaquin, is an imidazolinone introduced in 1986 as a soybean herbicide. It was reported by American Cyanamid to have limited movement in the soil. Because of the presence of acidic and basic functional groups on imazaquin it is amphoteric. The carboxyl group ionizes at a pKa value of 3.8.

Therefore under typical soil conditions it is an anion (Goetz et al., 1986). At lower pH values, the nitrogen groups on imazaquin may protonate resulting in a positively charged compound which may bind to soils (Loux et al., 1989). Imazaquin has a molecular weight of 311.3, and a water solubility of 60 to 120 mg L^{-1} (Anonymous, 1986). According to Pionke et al. (1988), imazaquin would be susceptible to leaching. In a study by Basham and Lavy (1987) the half life calculations ranged from 40 days to 11.7 months. Goetz et al. (1986), in a study on five cultivated soils (two Rhodic Paleudults, and one of each of the following Plinthic Paleudult, Entic Pelludert and one Rendollic Eutrocrept) in Alabama, showed imazaquin to be mobile with K_d values ranging from 0.001 to 0.21 using a batch equilibrium technique. The K_d value (distribution coefficient) represents the affinity of the soil for the compound, and can be expressed by the following equation:

$$K_d = [A(solid)] / [A(aqueous)] \quad (1)$$

Where [A(solid)] is the concentration of the solute A present on the solid phase and [A(aqueous)] is the concentration of the solute present in the aqueous phase. The larger the K_d, the greater the affinity for the solid phase. Using thin-layer soil chromatography the mobility of imazaquin was determined to have R_f values of 0.8 to 0.9. These values reflect the movement of imazaquin along the surface of thin layers of soil. Higher R_f values indicate greater mobility. Field studies conducted in Arkansas suggested that imazaquin losses by leaching are significantly lower under normal summer field conditions due to its rapid deactivation at the soil surface. Using ring-labeled ^{14}C-imazaquin and a batch equilibration technique on selected Arkansas soils (a Mollic Albqualf and a Vertic Haplaquept), Basham et al. (1987) obtained K_ds that ranged from 0.14 to 0.30. Loux et al. (1989), indicated the average imazaquin K_d to be 1.01 with zero

adsorption on some of the 22 soils and 6 sediments used. The soils ranged in clay content from 7 to 60 percent with 4 of the soils primarily montmorillonitic and 6 of the soil primarily kaolinitic. Most all of the sediments were primarily montmorillonitic. This would suggest moderate binding of the herbicide to the soil. A positive correlation with organic carbon content and negative correlation with soil pH was found. Therefore, greater adsorption occurred as organic carbon and clay content increased and as pH decreased. These increased adsorption conditions result in reduced imazaquin activity, higher Freundlich K values, and reduced mobility.

Batch equilibrium studies (one method used to establish the relationship between the solute in the solution phase to the adsorbed phase) do not account for the conditions of the solute in a field situation. Due to the manipulation of the soil by sieving, shaking and centrifugation there is a creation of more available charged surfaces or a removal of various oxides which may mask the charged area under field conditions. Batch isotherms likely overestimate adsorption as compared to the field condition (Sparks, 1989). In the field, where soils are undisturbed, preferential flow results in less exposure to reactive surfaces and therefore less interaction with adsorption sites than observed in batch equilibrium techniques (Jardine et al., 1988). Using large undisturbed soil columns taken from both conventional and no-till plots, Andreini and Steenhuis (1988) showed that no-till exhibited greater preferential flow in the upper layer of the soil. Tillage in the upper disturbed layers resulted in greater contact between the solute and soil reaction sites, thus reducing solute transport.

One dimensional solute transport is often described by the connvective-dispersive equation (Equation 2). This equation relies on the distribution

coefficient concept. It implies a linear relationship between the adsorbed and the solution phase concentration. The equation is

$$R\delta C/\delta t = D\ \delta 2C/\delta x2 - v\delta C/\delta x \qquad (2)$$

where C is the concentration of the solute; t is time; x is distance; R is the retardation factor taken from the literature; D is the dispersion coefficient; and v is the average pore water velocity (defined by q/θ where q is the Darcian fluid flux density, and θ is the volumetric water content). The retardation factor (Equation 3), a variable which accounts for the adsorptive capabilities of the soil, is further defined for linear, equilibrium adsorption by

$$R = 1 + pK_d/\theta \qquad (3)$$

where p represents the soil's bulk density, and K_d is a distribution coefficient. For solute that is not adsorbed to the soil, K_d becomes zero and R reduces to one (Wierenga and Van Genuchten, 1989).

The dispersion coefficient, D, describes the spread, due to diffusion and hydrodynamic dispersion, of the solute front as it moves through the soil. Values for D may be approximated by fitting Equation 2 to actual solute concentrations as a function of time and distance from a known source. Van De Pol et al. (1977) reported that usage of log normally distributed values of v and D can create significant errors in predicting the amount of leaching occurring at a specific soil depth. Their study also indicated that variables such as pore water velocity affect the dispersion coefficient. This complicates the application of Equation 2 to field scale problems in that estimates of v and D are not independent.

In homogeneous systems a correlation coefficient ε, (dispersivity) relates the near linear relationship of the pore water velocity, v, to the dispersion coefficient, D. Wierenga and Van Genuchten (1989), using repacked soil

columns of two different sizes, found greater dispersion in the larger columns. However, there was no clear evidence of an increase in D with depth in the large columns. There was a five-fold increase of ε in the larger columns as compared to the smaller. Butler and Jury (1989) found that the dispersion coefficient was a function of the length of the column, and others have supported this, but the relationship is not clear.

The dispersion coefficient has been noted as a curve-fitting parameter which, depending on the average pore water velocity and the size and distribution of the structural units of the soil, may vary by several orders of magnitude. (White, 1985). Macropore flow, i.e., flow through preferential paths, affects the average pore water velocity causing greater variability in the flow velocity within and between pores, therefore influencing dispersion. White (1985) reported increased concentrations of *Escherichia coli* in column effluent when the soil was undisturbed and had strong preferential flow. In soils were the pore structure was disturbed and preferential flow was minimal, *E. coli* (which are excluded from pore sizes smaller than 15 micrometers) exhibited limited transport through the soils.

The convective-dispersive equation (CDE) in Equation 2 is the most common approach used in mathematically modeling the transport of solute through soil. It must be remembered that the CDE, as with any numerical model, is simply a mathematical representation of the conceptual model for how water and solute move through the soil. It is not reality but a model of reality and includes numerous assumptions and simplifications about the flow and the reactivity.

The foremost assumption of Equation 2 is that water flow is through a ridged porous media under steady state conditions. Under field conditions, soil

water flow is rarely steady state, particularly in the unsaturated zone. Steady state is obtained experimentally in order to match the flow conditions with the CDE. This is typically accomplished by maintaining saturated soil conditions so that the soil water content will be constant with time, $\delta\theta/\delta t = 0$. The alternative is to maintain a constant flux on a soil profile or column. Neither of these experimental conditions represent the real world. Pesticides are not applied to saturated soils. The rainfall events that transport solutes through the soil do not occur under steady input rates for any significant time.

It is generally conceded that most soils exhibit some degree of preferential flow. This is even true for soils thought to consist of homogeneous sand (Selker et al. 1992), and it is especially true for no-tillage conditions. By definition, preferential flow is a result of water moving nonuniformly through the soil. This has been described by a two-region (Selim et al., 1987) form of the CDE and more recently by a multi-region form. The two-region CDE assumes that the soil can be partitioned into mobile and immobile regions with transfer of solute between regions occurring by diffusion. This approach has been applied successfully to saturated soil column studies but its usefulness to field transport has been questioned (Wilson et al., 1992). If preferential flow is evident then an alternative form of the CDE is necessary.

In order to describe solute transport by Equation 2, the parameters R and D must be known. These parameters should be determined independent of one another and independent of the solute breakthrough curve (BTC) to be modeled. This is rarely the case. The most common approach is to experimentally determine BTCs for a nonreactive solute and a reactive solute simultaneously. The CDE, Equation 2, is fit to the nonreactive solute BTC to determine D by setting $R = 1$. The R for the reactive solute may be determined independently by

performing a solute adsorption isotherm to estimate the K_d. However, it is common to fit the CDE to the reactive solute BTC while setting D equal to the fitted D from the nonreactive solute BTC.

Ideally, D represents the dispersion of the average water molecule. However, the 'nonreactive' solute typically used to determine D by BTC fitting is not a water molecule, eg. tritium, but is an anion such as Br^-. Thus it is assumed that the dispersion experienced by this anion is the same as for the water and the same as for the reactive solute. This may not be the case, even for the controlled experimental conditions. Anions are repelled by negatively charged soil particles, thereby concentrating anionic solutes near the center of soil pores. Since the water flow is fastest in pore centers it is probable that the anion will, on average, exit the soil column faster than the average water molecule. This has been found by James and Rubin (1986) to be the case when comparing BTCs for Br^- and tritium. Secondly, the solute of interest by nature of being reactive will be more closely associated with the particle surface where flow is slowest. For this reason, some researchers feel it is best to fit D to the reactive solute BTC and determine R independently. Either approach is better than fitting both D and R simultaneously to a single BTC.

The reactivity for the solute transport represented mathematically by Equation 2 is extremely simplified. Equation 2 is only applicable if there is no volatilization, and no degradation or other chemical or biological transformation of the solute. Equation 2 is a simplifed form of the CDE in that it presumes that the reactivity is described by the single parameter R. Thus this form of the CDE only applies to linear (K_d) type adsorption isotherms. This is true for some solutes; however, many solutes exhibit nonlinear adsorption that must be described by more complex expressions such as a Fruendlich or Langmuir

adsorption model. Reactivity of the solute with soil is typically determined by a batch isotherm analysis. Batch isotherms involve drying the soil sample, followed by grinding, and sieving. A known quantity of this pulverized soil is placed in a flask and a solution of known concentration of solute is added. The soil plus solution is shaken for a prescribed time, a solution sample is removed and filtered, and the solute concentration remaining in the solution determined. Some of the problems with this technique are that the amount adsorbed depends upon: (i) how the soil was prepared (method of grinding), (ii) length of shaking time and method for shaking (ie. continuously or intermittently,) and (iii) the solution matrix. The major problems of the solution matrix are its pH, ionic strength, electrolyte composition, and temperature. The following precautions must be taken in order to reduce errors caused by the solution matrix:

a) The soil sample must be monoionic. To do this, one must saturate the exchange sites with a cation of similar valence as the solute of interest.

b) The pH of the matrix must be buffered due to the pH-dependent charges of some of the colloids.

For reduction in errors in column experiments these precautions apply:

a) Use the same ionic strength in the BTC (miscible displacement) experiment as was used in the isotherm experiment.

b) Saturate the soil column with the same cation used in the isotherm experiment to make soil column monoionic.

c) Use same exchanging cation in the displacing solution passed though the soil column as in isotherm (Ca-Mg exchange).

A further complication is that the adsorption may be kinetically driven. That is, it is assumed in Equation 2 that the solute is at all times in instantaneous

equilibrium with the soil. Adsorption is assumed to be instantaneous and not a function of time. This condition is met in the experimental isotherm determination by first reacting the solute of interest with the soil and sampling the amount adsorbed with time. The change in time, from initial sampling until no further solute can be removed, is used as the reaction time for the isotherm determination. However, the solute transport experiment does not incorporate this time factor, and indeed the BTC may be completed before the time for equilibrium is reached. For such conditions, a kinetic form of the CDE must be applied.

It is further assumed in Equation 2 that all reaction sites are equally accessible to and reactive with the solute. The first point may be true for repacked soil columns which are assumed to represent the plow layer. Traditionally, batch studies have been conducted to determine the reactivity of the solute with soil. This approach facilitates all possible adsorption sites to be made available for reaction. However, new sites may also be created by grinding (Sparks, 1989). Natural soil has Fe- and Al-oxide coatings that mask exchange sites and preferential flow results in the bypassing of many exchange sites by the solute. As discussed previously, soils typically exhibit some degree of preferential flow. This preferential flow not only affects the description of the flow, but also affects the reactivity. Batch isotherms may be representative of transport through repacked soil columns and applicable to the soil condition of the plow layer. However, they do not represent transport through undisturbed soil columns or "natural" soil conditions such as exist below the plow layer or even at the surface under minimum tillage. Jardine et al. (1988) showed that batch-determined adsorption isotherms significantly over predicted adsorption and therefore under predicted transport as compared to undisturbed soil. The

second point, that all sites are equally reactive, may not hold for all soil conditions. Jardine et al. (1988) applied Parker and van Genuchten's (1984) two-site solution of the CDE to describe the transport of NH_4^+. Type 1 exchange sites were assumed instantaneous and reversible, while type 2 exchange sites followed reversible first order kinetics.

Thus it is obvious that there are many assumptions and simplifications inherent in the CDE expression of Equation 2. Additionally, the experimental conditions of batch isotherms to estimate R for describing a BTC are quite restrictive. Thus one must select with caution the CDE expression that is most appropriate for the flow and transport conditions of the soil of interest. We have selected the most simplified form of the CDE with an understanding of the restrictions for which it applies. It was not our objective to accurately predict the transport of imazaquin for field conditions, but rather to gain a knowledge of fate and transport modelling.

CHAPTER III

MATERIALS AND METHODS

A tractor mounted hydraulic sampler, developed by Walker et al. (1990), was used to collect six intact soil columns. The sampling cylinder was a 110 cm long steel pipe with and inside diameter of 34 cm. A tapered steel pipe with an inside diameter of 30.5 cm was welded to the end of the sampling cylinder. Schedule 40 PVC pipe (inside diameter of 30.5 cm) was inserted into the sampling cylinder and rested on the tapered steel pipe. The sampling cylinder was then driven into the soil by the tractor mounted hydraulic sampler. The soil columns were collected from the Ap and the upper portion of the B horizon of an Etowah silt loam (clayey, kaolinitic, thermic Typic Hapludult) which had not been in cultivation for 10 years. Columns were collected from the Plant Sciences Field Laboratory, Knoxville, TN, and transported to the laboratory.

The bottom of each column was fitted with a 33 cm x 33 cm PVC plate with a 1 cm hole drilled in the center and a 10 cm long PVC tube inserted in the hole. These plates were welded onto the bottom of each column using a PVC rod and a hot air welder. Tygon tubing was place over the PVC tube which lead to a vacuum hose and a series of four 500 mL Erlenmeyer flasks to collect the leachate .

Before the tops of the columns were sealed, the soil depth was measured and the top 8 cm of soil was removed, sieved through a 2 mm screen, and replaced. Wheat straw was applied at the rates of 0 and 4480 kg ha^{-1}. A 10 cm length of tygon tubing was inserted through a 1-cm hole drilled in the center of the acrylic sheet for delivery of imazaquin and water. To control the effects of splash, and to encourage radial displacement, Whatman #42 paper was placed on the soil surface directly underneath a tygon tube which applied the water and

imazaquin (American Cyanamid). Using a silicon caulk, a 33 cm x 33 cm acrylic sheet was attached to seal the top.

After sealing the columns, deionized water was applied to the top of each column at 1.8 cm^3 min^{-1} with a Technicon peristaltic pump. After three days, ponded water was syphoned off the columns, and the rate was changed to 0.8 cm^3 min^{-1} and allowed to drain freely for 10 days. This was followed by an 0.8 cm^3 min^{-1} application of 0.01 M KCl to establish an electrical conductivity near that of the bromide tracer. It also prevented the stripping of the soils exchange sites, thereby leaving them essentially monoionic satisfied for the application of imazaquin and the bromide tracer. After steady state flow was achieved, a vacuum of 20 cm of water was applied at the bottom of the columns (Figure 2).

Preliminary pulse duration times were predicted using the FORTRAN program CXTFIT (Parker and Van Genuchten, 1984) that simulates or solves Equation 2. The retardation factor (R) of imazaquin was taken from the K_d values found in the literature and applied to Equation 3, resulting in R values ranging from 1.0032 to 2.4412. The K_ds used ranged from 0.001 to 1.01 (Goetz et al., 1986; Basham et al., 1987; and Loux et al., 1989). Since Br^- is a nonreactive tracer, its R was assumed to equal one. The average column length was 0.584 m. The average pore water velocity of 0.76 cm^3 min^{-1} (v) was defined by the Darcian fluid flux and the volumetric water content. The Darcian flux was monitored and maintained at steady state and the volumetric water content was assumed to be 38% in the prediction equation. Using the column's pore water contents, and lengths plus the Peclet number (Pe) of 0.67 calculated on the same soils in a similar experiment by O'Dell (1989), the estimated dispersion coefficient were calculated from Equation 4.

$$Pe = vL/D \qquad (4)$$



Figure 2. Column leaching assembly.

The input concentration (C) of imazaquin was set to represent the recommended rate for a 7.62 cm depth of incorporation. Using these parameters in the CXTFIT program, a prediction of the solute breakthrough curve was made to test our input parameters for the detectability of imazaquin (Figure 3). The timing of the sampling and concentration of imazaquin were based upon this prediction.

Technical grade imazaquin (92.7 % pure) was applied in 0.1 M KBr (7990.9 mg L^{-1}). The nonreactive tracer Br^- in the column effluent was monitored according to the schedule and measured by an ion specific electrode. The imazaquin in leachate samples was determined by High Performance Liquid Chromatography (HPLC). The HPLC system included Waters model 481 LC spectrophotometer, model 501 pumps and model 740 data module. The mobile phase was a 65:35 (v:v) 4% aqueous acetic acid: acetonitrile at a flow rate of 1 mL min^{-1}. The stationary phase consisted of a reverse phase column, 15 cm long by 4.6 mm inside diameter with 5 micrometer C-packing material. The sample was injected in 50 µL aliquots. The a sensitivity of the method was 1 mg L^{-1}.

To determine the soil water tension of the soil columns, small tensiometers were inserted by drilling into the column's side. A slurry of the soil shavings was made and applied to the porous ceramic cup to insure good soil contact. After insertion, at 12 cm below the surface and 12 cm above the base, the drilled areas were sealed with silicon caulk. Measurements of soil water tension were made with a hand-held pressure tranducer system (Soil Measurement Systems, Tucson, AZ).

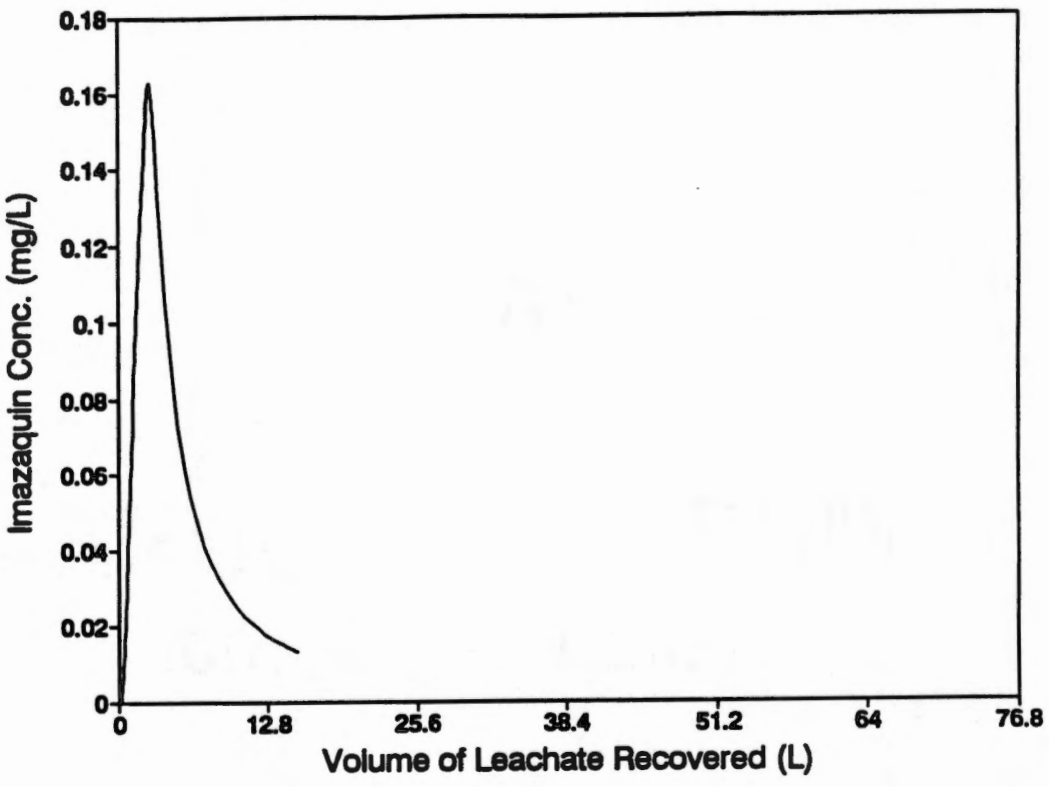


Figure 3. Prediction of imazaquin's breakthrough.

Wheat straw was collected off the tops of the columns and analyzed for imazaquin sorption. Soil samples where collected at depths of 0 to 2.5, 2.5 to 7.5, 7.5 to 15, 15 to 30, 30 to 46, 46 to 60 cm from each column to determine the movement of imazaquin. Samples were extracted and analyzed for imazaquin by a procedure obtained from American Cyanamid (1984). The residues of imazaquin on the straw and in the soil were extracted with deionized water. The pH of the extract was then adjusted to 2.0 and partitioned with methylene chloride. The methylene chloride was then evaporated to dryness and the remaining residue was dissolved in methanol and water. Solid phase extraction cartridges were used to clean up the samples. Imazaquin in the soil and wheat samples were analyzed on a Waters HPLC. The HPLC system included Waters model 481 LC spectrophotometer, model 501 pumps and model 740 data module. The mobile phase was a 60:40:4 (vvv) water, methanol and acetic acid combination. The flow rate of the mobile phase was 1.5 mL min^{-1}. The stationary phase consisted of a reverse phase column, 15 cm long by 4.6 mm inside diameter with 5 micrometer C-packing material. The sample was injected in 50 microliter aliquots. The sensitivity of the method was 5 $\mu g L^{-1}$.

To determine the extent of preferential flow path, one pore volume (determined by the volume of effluent/volume of pores) of reactive dye (Rhodamine B (2 g L^{-1})) was added after the leaching period had ended. The preferential flow paths were documented by taking photographs of the dye staining patterns of the dissected columns.

CHAPTER IV

RESULTS AND DISCUSSION

Bromide and imazaquin transport breakthrough curves varied with each column. The bromide breakthrough curves were used to determine the dispersion coefficients, D in Equation 2. It was assumed the D values (Table 1) obtained from bromide's BTCs represented the dispersion for the average water molecule flowing through the columns. The dispersion coefficients, solved for by using the Br^- BTCs, were entered into the CXTFIT program, along with the effluent concentration of imazaquin with time to solve for R (retardation factor).

Figures 4 to 15 alternately illustrate the BTCs of bromide and imazaquin. The noted difference in the smoothness of the curves of the Br^- breakthrough and the imazaquin breakthrough was considered a result of the higher concentration of Br^- in the effluent as compared to the imazaquin.

The dispersion coefficient in the bromide curves was affected by temperature and depends on pore water velocity, water content, solution viscosity and density, molecular diffusion, and tortuosity. Using the Peclet numbers (Equation 4) for each column, comparisons can be made of dispersion independent of flow rate and column length (Table 1). Pe values of less than 0.4 signify that molecular diffusion is dominant. Pe values between 0.4 and 5.0 signify both molecular diffusion and mechanical dispersion are occurring simultaneously. Pe values of greater than 5 show mechanical dispersion predominates. The Br^- BTCs for columns 1, 4 and 5, presented in Figures 4, 10, and 12, respectively, produced the best fit of D in Equation 2. These columns had similar Pe values of .0376, .0253, and .0237 respectively.

Table 1: Column Information

Parameter	col 1	col 2	col 3	col 4	col 5	col 6
Flow ($cm^3\ hr^{-1}$)	47.58	48.48	25.98	47.94	47.28	46.14
Length (m)	0.558	0.584	0.584	0.610	0.558	0.610
Radius (cm)	15.24	15.24	15.24	15.24	15.24	15.24
Column Area (cm^2)	729.7	729.7	729.7	729.7	729.7	729.7
Density ($g\ cm^3$)	1.58	1.58	1.58	1.58	1.58	1.58
θ ($cm^3\ cm^{-3}$)	0.487	0.329	0.471	0.485	0.408	0.343
q ($cm^3\ cm^{-2}\ hr^{-1}$)	0.065	0.066	0.036	0.066	0.065	0.063
avg v ($q\ \theta^{-1}$)	0.134	0.202	0.076	0.135	0.159	0.184
D ($cm^2\ hr^{-1}$)	2.006	1.453	0.869	3.297	3.784	18.21
r^2 for D	0.955	***	0.439	0.82	0.817	***
Pe	0.038	0.82	0.051	0.025	0.024	0.002
retardation (R)	3.706	8.303	3.143	4.382	6.129	21.47
r^2 for R	0.132	-0.73	0.444	0.572	0.500	0.554
Mass balance of imazaquin ($mg\ L^{-1}$)	0.483	0.282	0.208	0.448	0.428	0.231
% imaz. recov'd	100.8	59	43	94	89	48

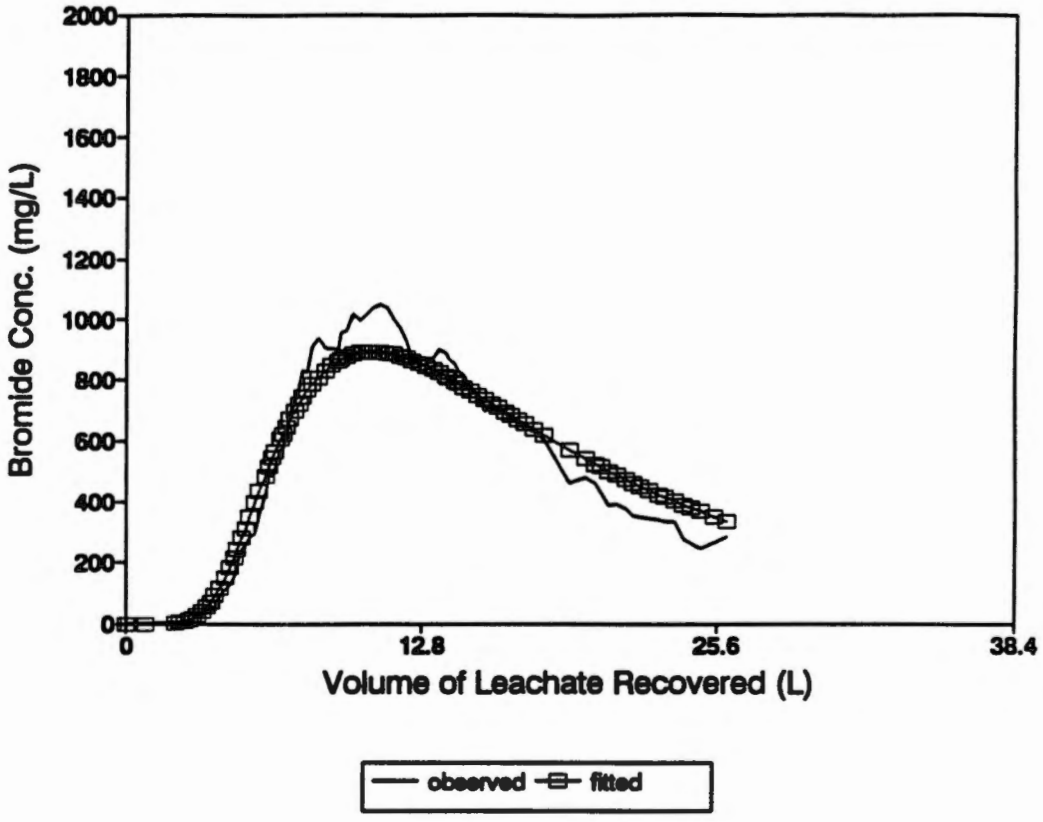


Figure 4. Breakthrough of bromide in column 1 with straw amended surface. The dotted line represents the best fit of the CD model. One pore volume equals 12.8 L.

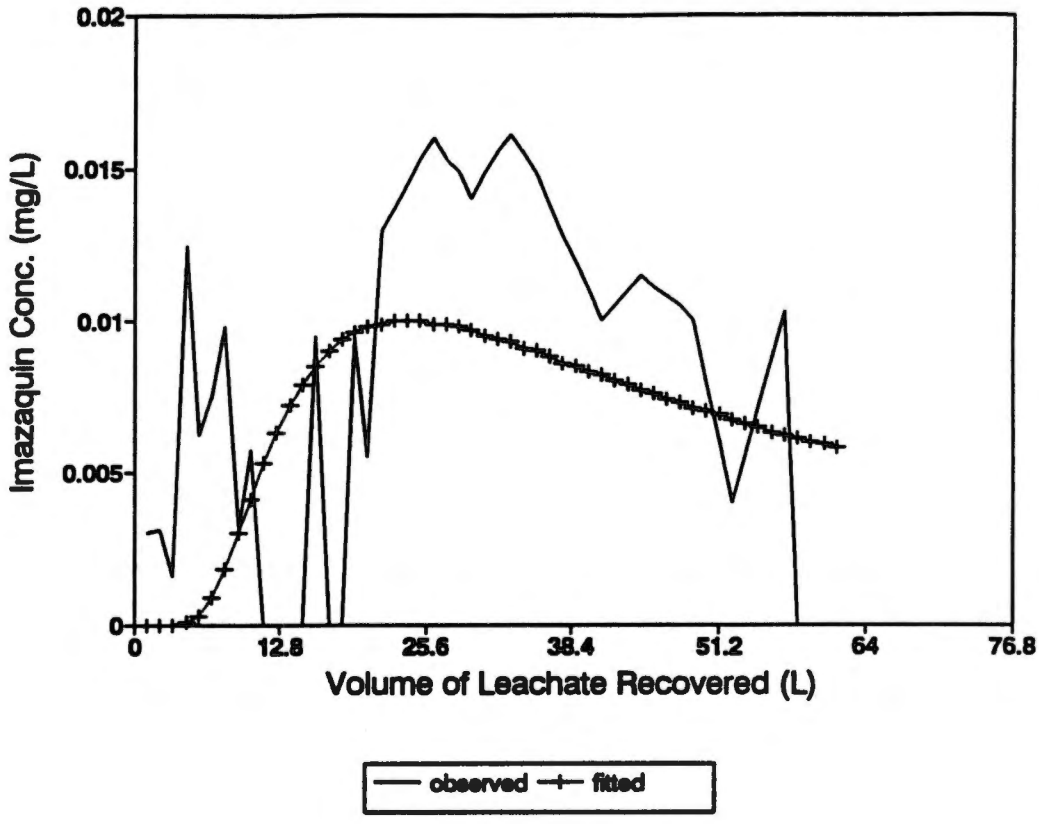


Figure 5. Imazaquin breakthrough curve for column 1 with straw amended surface. Dispersion = 2.006. Retardation factor = 3.706 with an r^2 of 0.132.

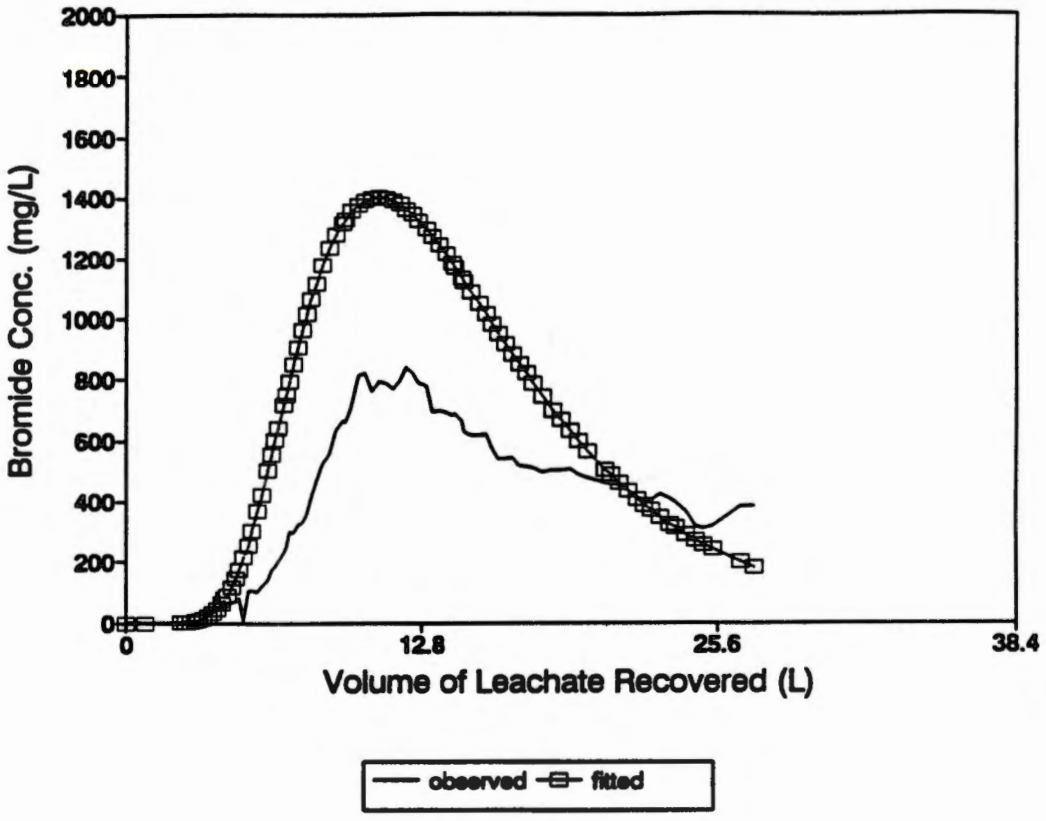


Figure 6. Breakthrough of bromide in column 2. The dotted line represents the best fit of the CD model. One pore volume equals 12.8 L.

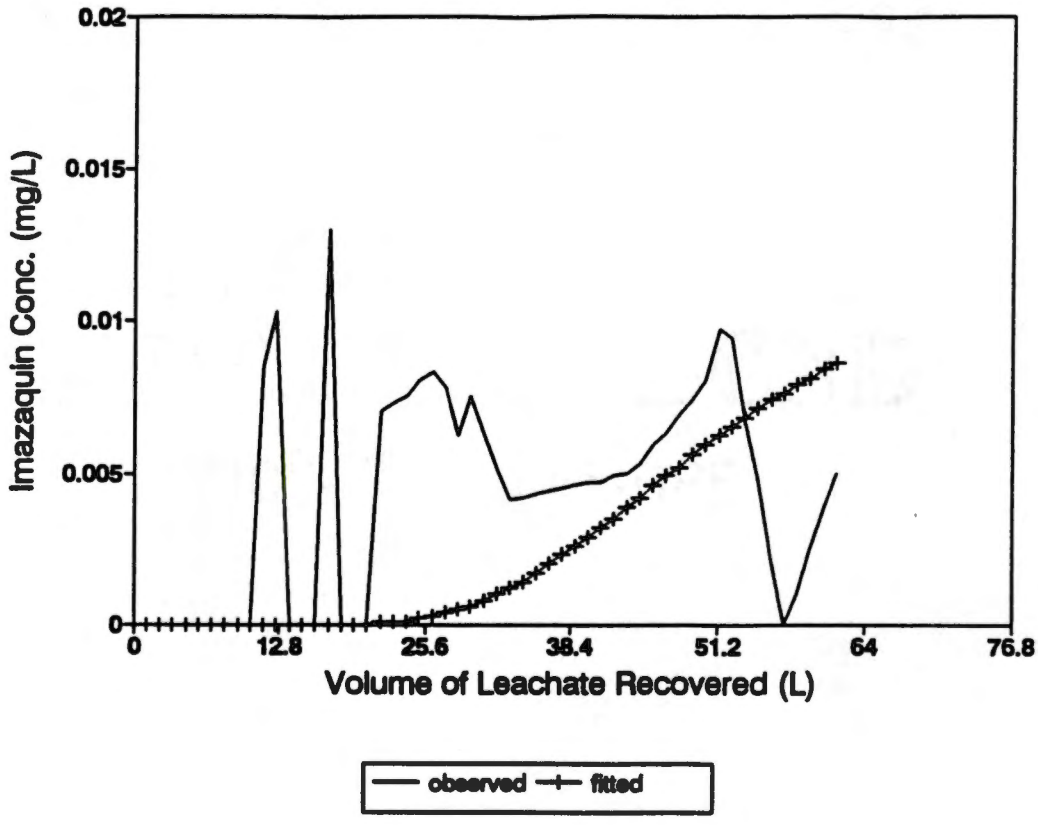


Figure 7. Imazaquin breakthrough curve for column 2 . Dispersion = 1.453. Retardation factor = 8.303 with an r2 of -0.73.

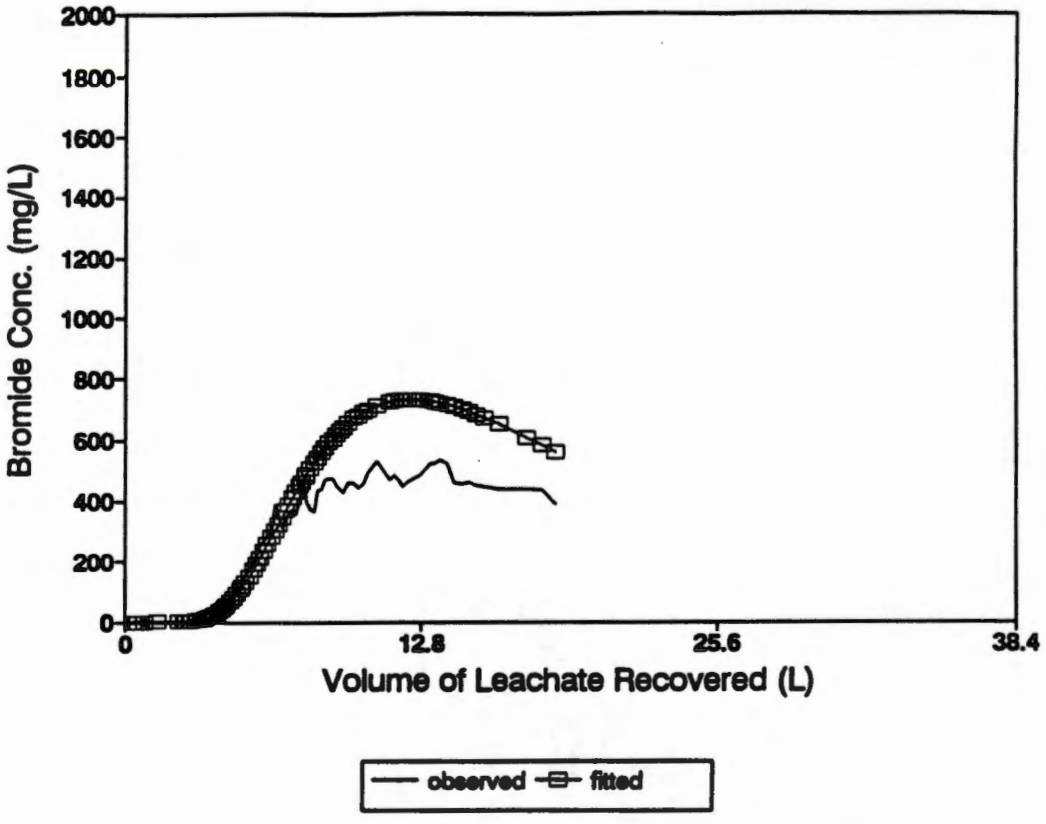


Figure 8. Breakthrough of bromide in column 3 with straw amended surface. The dotted line represents the best fit of the CD model. One pore volume equals 12.8 L

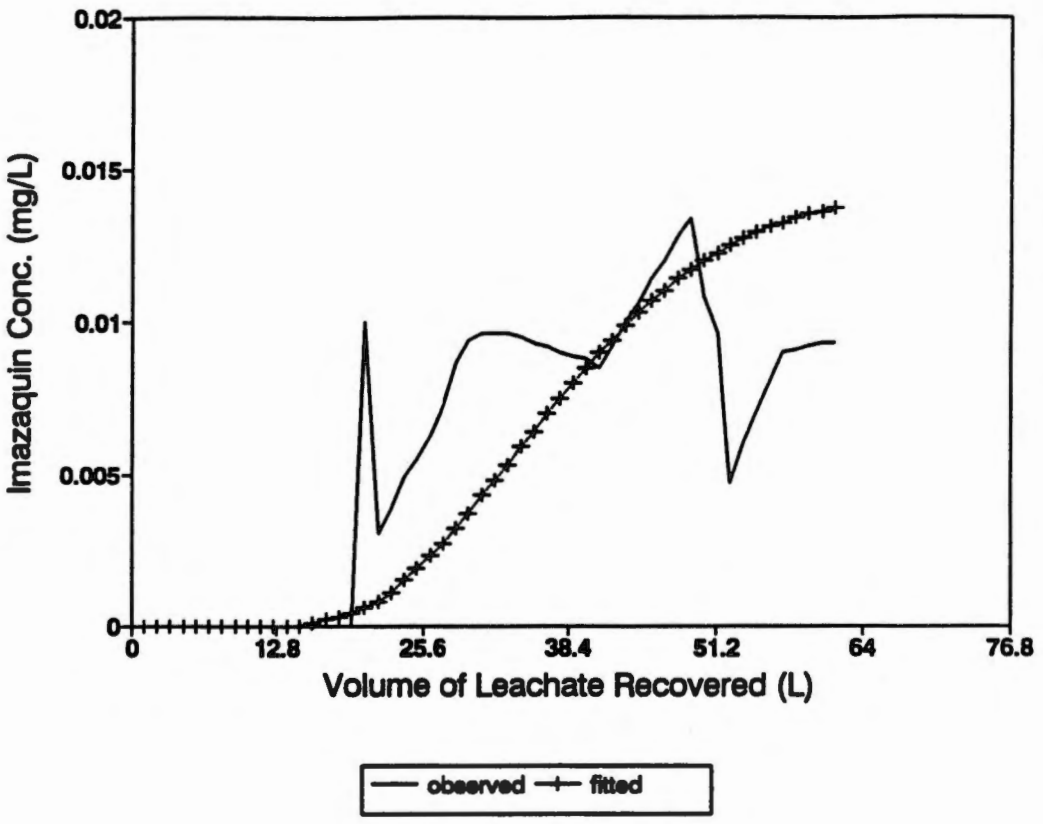


Figure 9. Imazaquin breakthrough curve for column 3 with straw amended surface. Dispersion = 0.869. Retardation factor = 3.143 with an r^2 of 0.444.

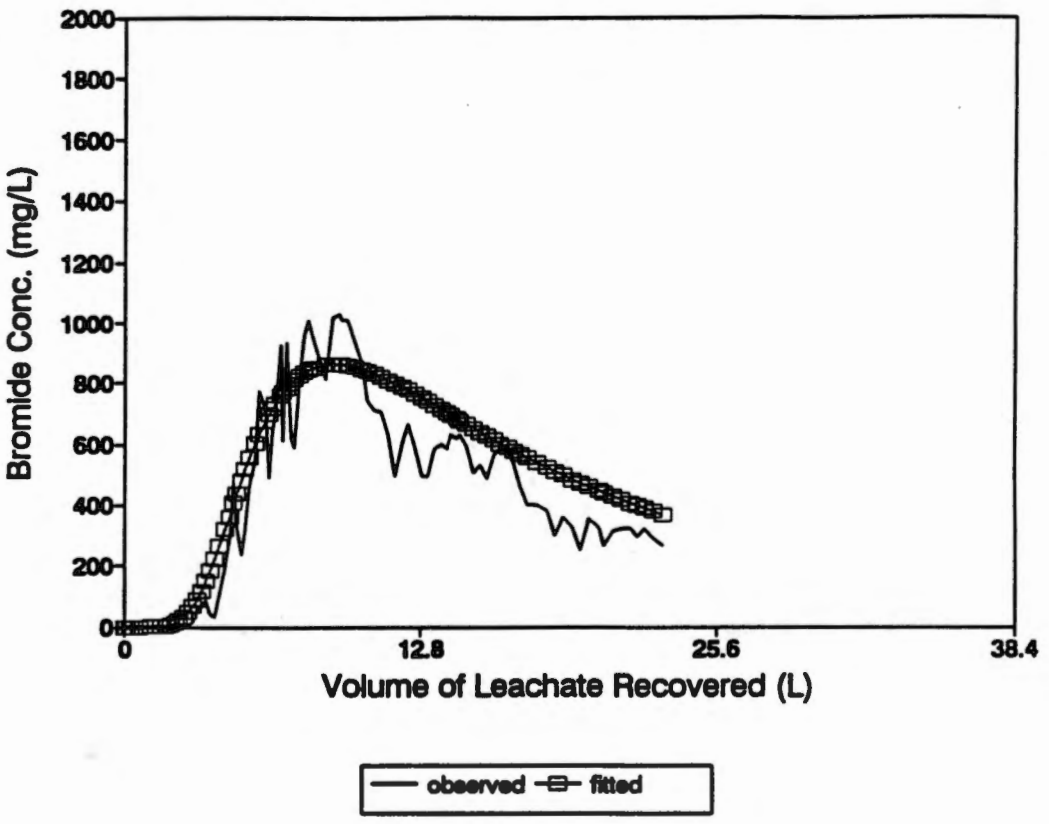


Figure 10. Breakthrough of bromide in column 4. The dotted line represents the best fit of the CD model. One pore volume equals 12.8 L.

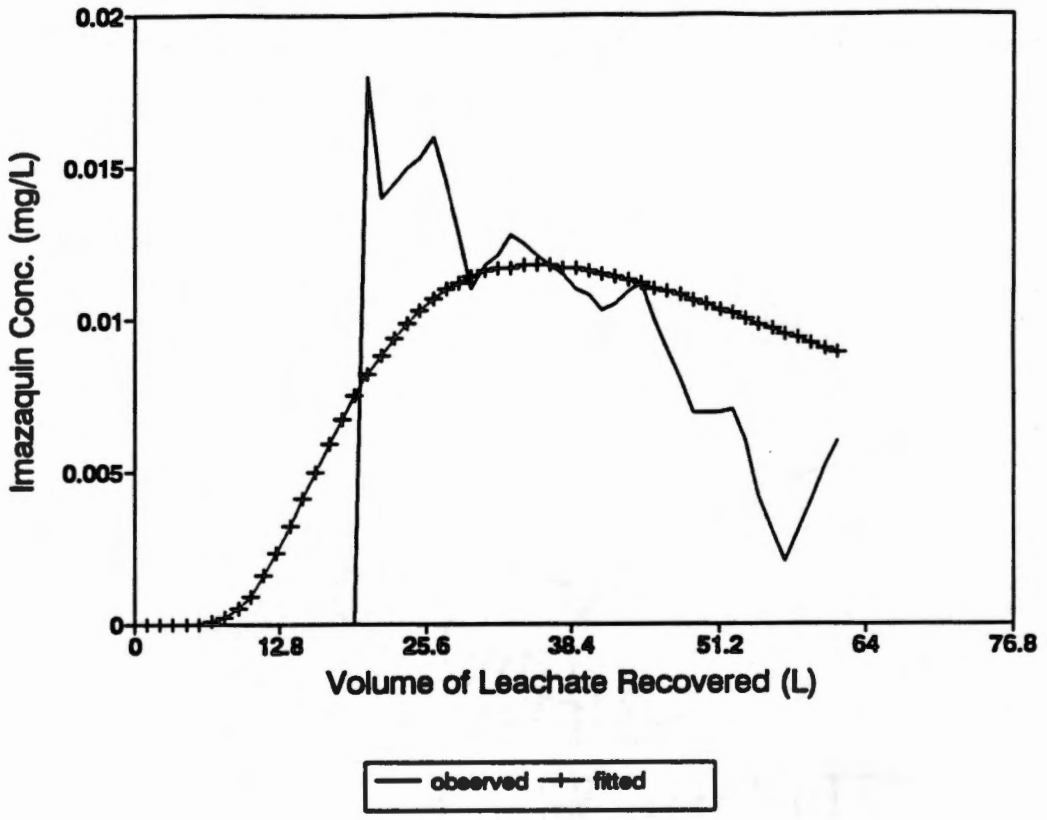


Figure 11. Imazaquin breakthrough curve for column 4. Dispersion = 3.297. Retardation factor = 4.382 with an r^2 of 0.572.

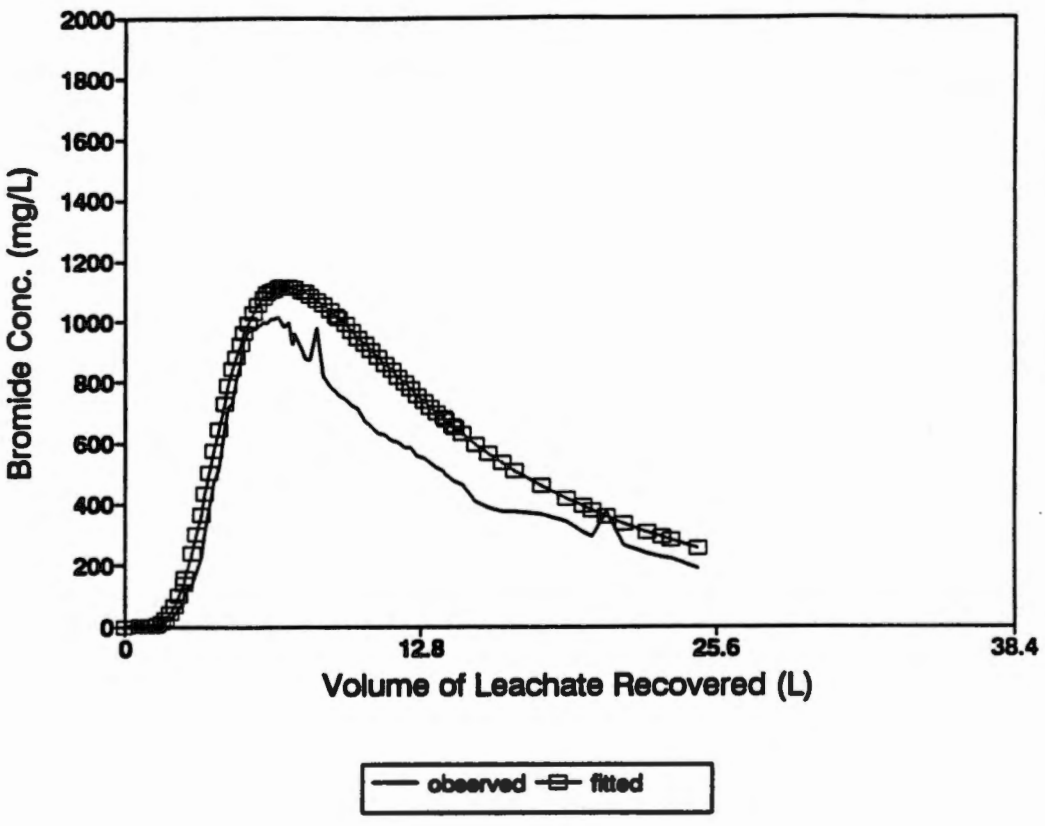


Figure 12. Breakthrough of bromide in column 5 with straw amended surface. The dotted line represents the best fit of the CD model. One pore volume equals 12.8 L.

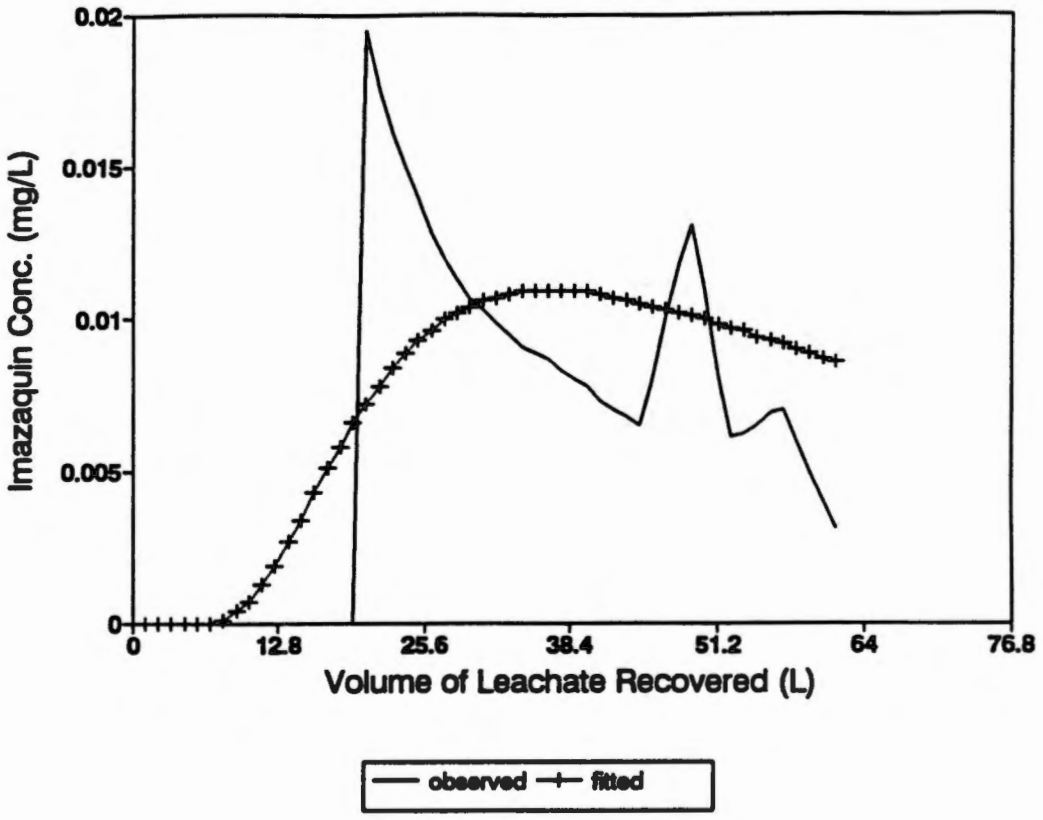


Figure 13. Imazaquin breakthrough curve for column 5 with straw amended surface. Dispersion = 3.784. Retardation factor = 6.129 with an r^2 of 0.5.

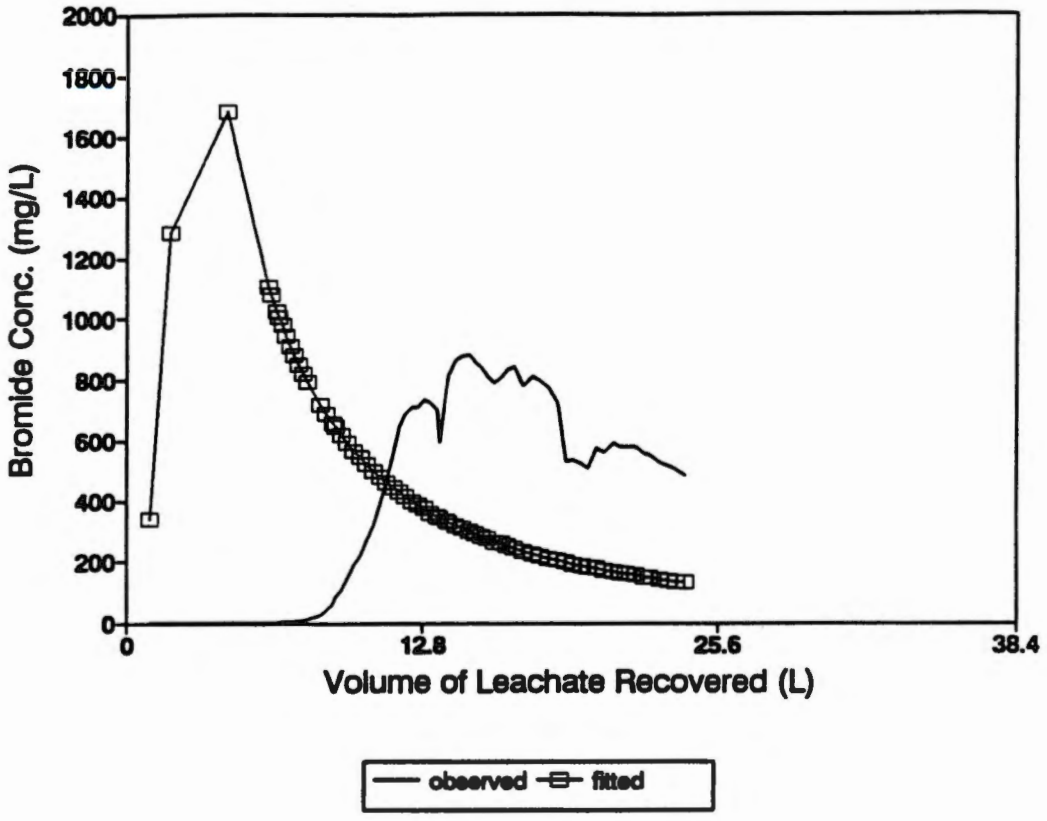


Figure 14. Breakthrough of bromide in column 6. The dotted line represents the best fit of the CD model. One pore volume equals 12.8 L.

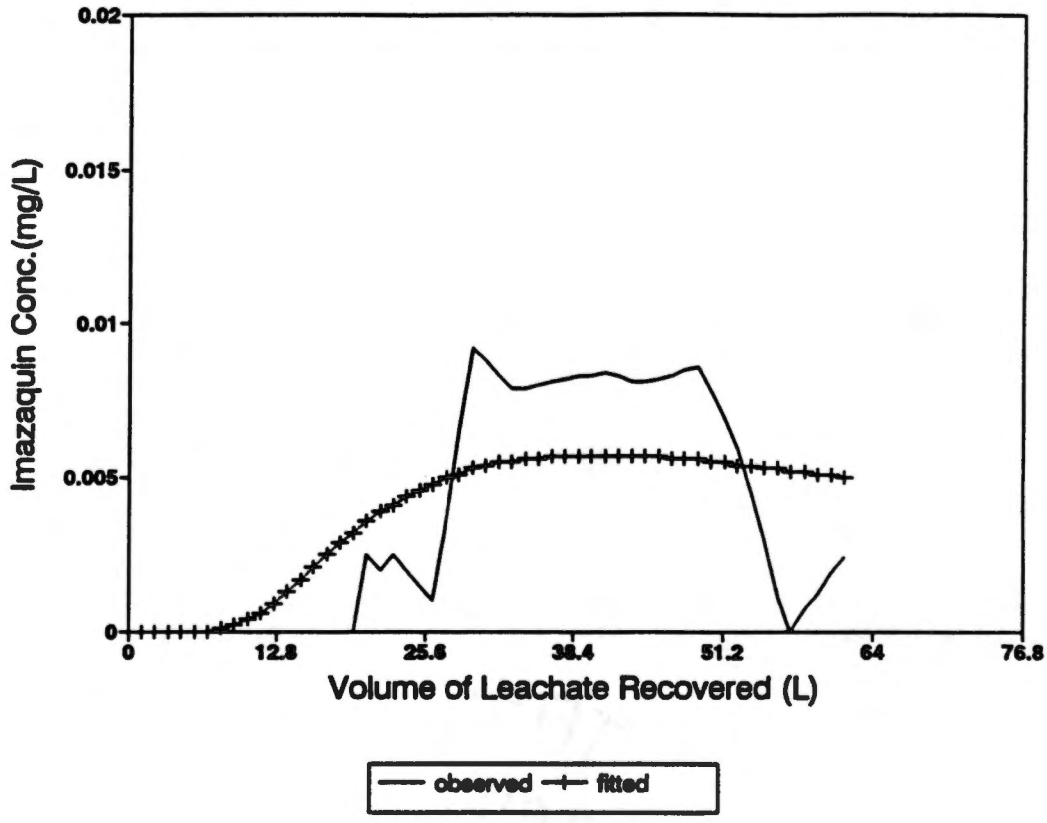


Figure 15. Imazaquin breakthrough curve for column 6. Dispersion = 18.21. Retardation factor = 21.47 with an r^2 of 0.554.

The Br^- BTC for column 3 (Figure 5), which was distinguished by its slow flow rate, had a Pe value of .0513. The Br^- BTCs for columns 2 and 6 (Figures 4 and 8, respectively) did not converge. Their Pe numbers, based on the dispersion coefficient of the last iteration of the CXTFIT program, were the highest and lowest of all the columns (.0820 and .0062, respectively).

It was speculated that the nonconvergence for columns 2 and 6 could be an artifact of the averaging of the water content in the determination of v in Equation 2. Using the CXTFIT program D and v were solved for simultaneously. The results suggested in order to obtain a convergence the water content of column 2 should have been equal to 0.616 $cm^3\ cm^{-3}$ nearly twice as high as the measured 0.329 $cm^3\ cm^{-3}$. For column 6 the water content should have been 0.535 $cm^3\ cm^{-3}$ in order to converge as compared to its measure water content of 0.343 $cm^3\ cm^{-3}$. The revised water contents were not realistic.

The dispersion coefficients from the Br^- BTCs were entered into the CXTFIT program, along with the effluent concentration of imazaquin with time to solve for R. However, due to the very low concentrations of imazaquin found in the leachate the R values were not fit with high confidence—as evidenced by their r^2 values (Table 1). The closer r^2 is to 1.0 the better the fit to the model. The r^2 values ranged from negative numbers to a high of 0.357. The model, however, does not take into account degradation of imazaquin over time; this factor is lumped into the R variable. Thus, low concentrations of imazaquin in the effluent over time, produced higher R values and subsequently larger r^2 values.

The concentration of imazaquin in the leachate was often at or below the detection limits of the analysis. This was illustrated in the noise of the imazaquin BTCs. In the analysis each sample was injected into the HPLC three times and

the average concentration was plotted. If the average was below detectable limits and could not be replicated it was recorded as 0.0 mg L^{-1}. The method of analysis was checked for linearity down to 0.01 mg L^{-1}, below this point reproducibility was rarely maintained. Expecting imazaquin concentrations near that of the outcome predicted by the model and best estimations (Figure 3), the method was retained. After effluent analysis found extremely low concentrations of imazaquin, it was speculated that adsorption or degradation of the compound was occurring. The soil was then analyzed by a method with sensitivity down to 0.005 mg L^{-1}.

The soil's imazaquin concentrations were close to the detection limits of the extraction method used (columns 5 and 6 were not analyzed due to the presence of Rhodamine B, the die used in the tracer analysis). This reduced the confidence of the analysis.

Although the concentration of imazaquin in the soil was near the detection limits of the analysis and therefore, unreliable, some trends were evident. In columns which had a wheat straw cover, more imazaquin was found in the 2.54 - 7.62 cm layer (Appendix A). This could have occurred due to the higher organic matter content of the layer. Imazaquin adsorption increases as organic matter increases (Basham et al., 1987). In the columns which had no straw on the surface, there is also a slight increase at the top of 7.62 - 15.24 cm layer, due to organic matter content. As imazaquin moved further into the soil, decreases were seen over all columns with the exception of the 15.24 - 45.72 cm layer. In this layer imazaquin concentration levels off. This could be a reflection of the increase in clay content of the argillic horizon. Imazaquin adsorption is positively influenced by clay content (Basham et al., 1987).

Since the concentration of imazaquin found in the leachate and the soil was low, a mass balance was calculated to determine the amount of imazaquin recovered. The mass balance was calculated by multiplying the amount of imazaquin present in the analyzed sample by the quantity of leachate or soil the sample represented. The mass balance results were then summed for each column. The mass balance for the leachate of columns 1, 4 and 5 accounted for 100.8, 93.5 and 89.4 percent respectively of the applied imazaquin. Columns 2, 3 and 6 accounted 58.9, 43.3 and 48.2 percent, respectively, of applied imazaquin.

Because the concentration of imazaquin in the soil and in the wheat straw were at or below detection limits, the mass balance summations were deceptive. Concentrations which were below detection limits were often analytically reproducible. However, these results summed in the mass balance equation produced exorbitantly high concentrations, beyond what was applied to the soil. The conclusion reached concerning this issue, was that the imazaquin concentrations found below or at detection limit should have been recorded as zero.

The preferential flow paths of soil columns 5 and 6 were qualitatively determined by adding Rhodamine B dye (2 g L^{-1}) at the same rate as the tracer. After application of one pore volume (12.8 L), the columns were dissected and flow paths were identified by the dye staining pattern. On the surface the dye distributed in a radial manner from the point of application. This radial pattern disseminated further throughout the 2.54 - 7.62 cm layer. This was the disturbed layer created by the removing soil and repacking. Below this layer some preferential flow was seen. However the pattern of flow was terminated near the 30.48 - 40 cm depth (Appendix B).

CHAPTER V

SUMMARY AND CONCLUSION

Imazaquin was found in the leachate of the columns and were not present in significant concentrations in either the soil or the wheat straw addition. The effluent concentrations were low, yet prevalent over the 54 day experimentation period. The prediction by CXTFIT of imazaquin's breakthrough, based upon best estimations and the K_d values from literature, overestimated the concentration at breakthrough as well as underestimating the retardation factor. Using Equation 3, the K_d values for these soils had a calculated range of 0.638 to 1.52 as compared to those in the literature with a range of 0.001 to 0.27. However, the r^2 values for fitting R in this experiment were not high.

Wheat straw incorporation appeared to have no effect on the transport of imazaquin through the soil. The data for the soil and wheat straw analysis showed insignificant amounts of adsorbed imazaquin.

CHAPTER VI

FUTURE RESEARCH AND RECOMMENDATIONS

The conditions under which this experiment was performed had many routes by which imazaquin may have been lost. In light of this, several suggestions need to be made for future experiments of this type. The rate of imazaquin applied could have been increased by several times the chosen rate of application. This could have provided better analytical results. The tops of the columns could have been opaqued to insure little loss by photodegredation. ^{14}C-labeled imazaquin could have been used, so that the fate of imazaquin could have been traced in microbial degradation and volatilization. Also, air traps could have been placed in the collection system, and a porous plate installed at the bottom of the column to further reduce losses by volatilization. Infiltration rates could have been reduced to more closely simulate field conditions.

In summary, the results of this research into the fate and movement of imazaquin through undisturbed columns showed slow gradual movement through the columns.

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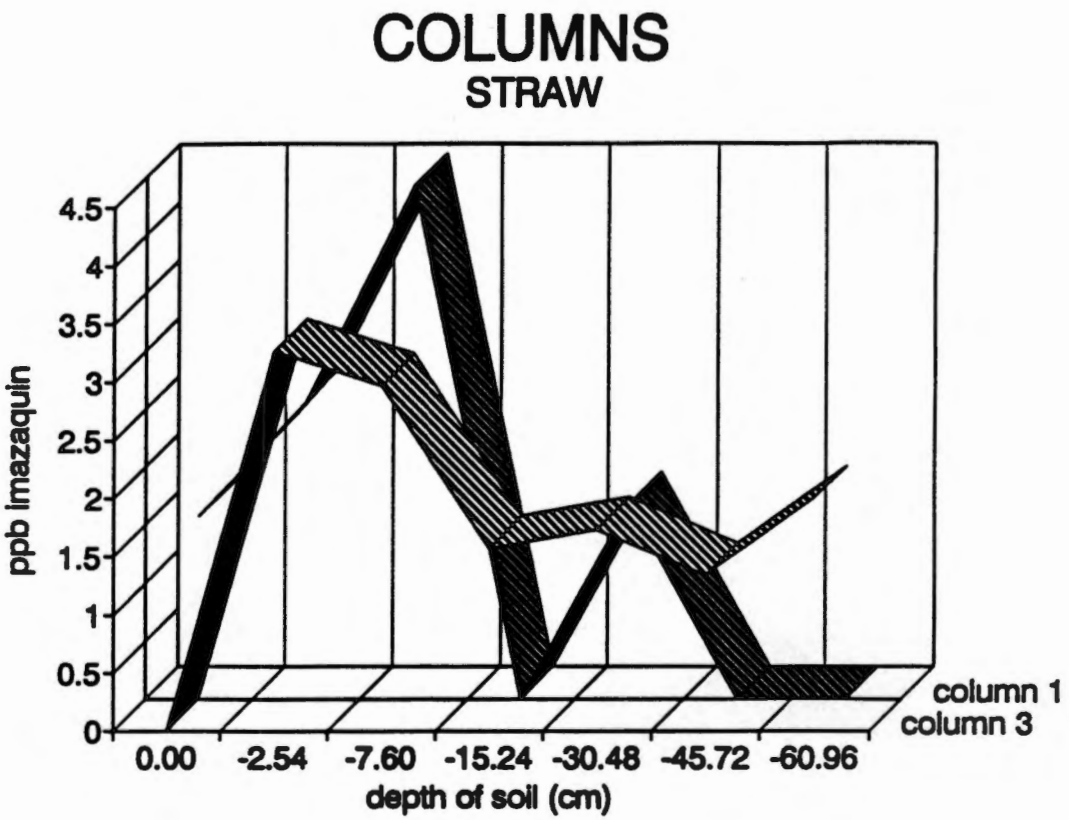
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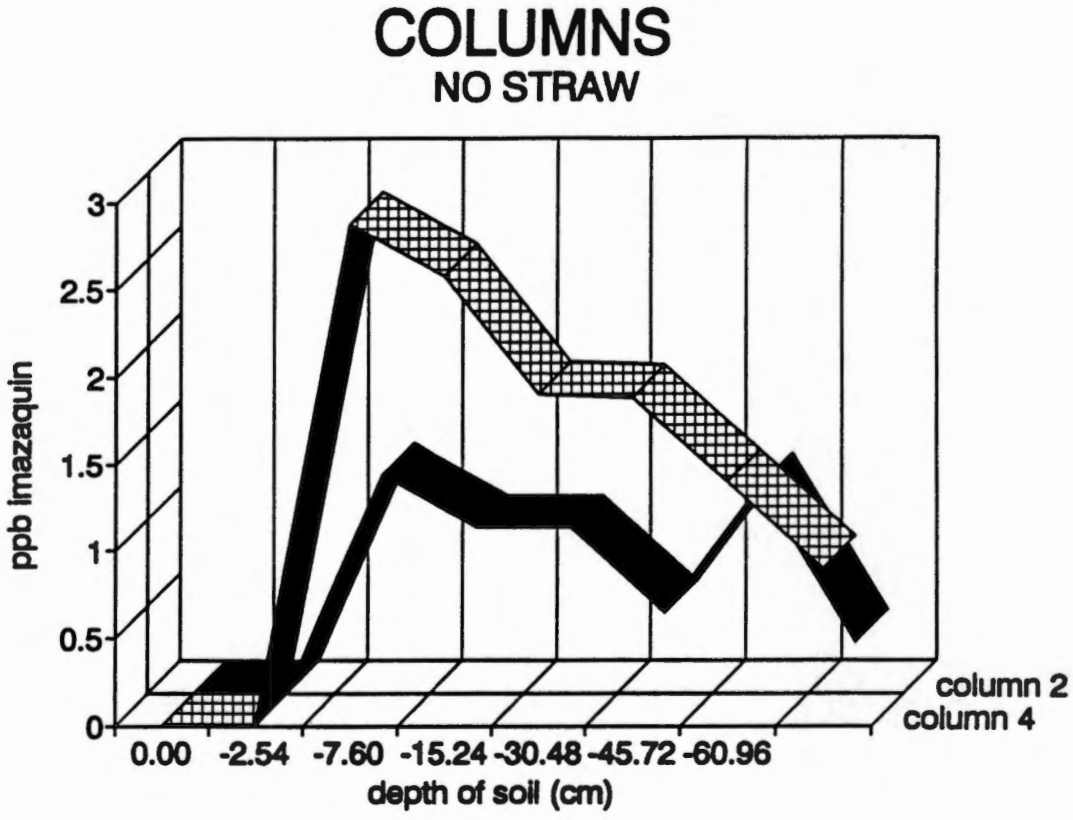
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APPENDICES

APPENDIX A

GRAPHS OF IMAZAQUIN IN THE SOIL


COLUMNS
STRAW
ppb Imazaquin
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
0.00
-2.54
-7.60
-15.24
-30.48
-45.72
-60.96
depth of soil (cm)
column 1
column 3


COLUMNS
NO STRAW
ppb imazaquin
3
2.5
2
1.5
1
0.5
0
0.00
-2.54
-7.60
-15.24
-30.48
-45.72
-60.96
depth of soil (cm)
column 2
column 4

APPENDIX B

PHOTOGRAPHS OF FLOW PATTERNS IN COLUMNS 5 AND 6



Rhodamine B dye breakthrough for column 5 (right) and column 6 (left).

VITA

Dolores Marie Chandler was born in Knoxville, Tennessee on March 24, 1961. She attended St. Joseph Elementary school in Knoxville, and graduated in 1979 from Norris High School in Norris, Tennessee. After several years of work, she entered the University of Tennessee in 1984 to obtain her Bachelor of Science degree in Plant and Soil Science. In 1988 she received this degree, and a teaching assistantship to continue her studies in Soil Science.

Throughout the first 4 years of her masters program she taught laboratory procedures for Introductory Soil Science, and Soil Fertility. In 1992, she accepted a position as research associate from the Department of Forestry at the University of Tennessee. In this position she carried out the remainder of an optimum nutrition experiment at Oak Ridge National Laboratory.

In 1993, she moved to Hillsborough, North Carolina, where she completed work on her masters thesis. She received the Masters of Science Degree in Plant and Soil Science with an emphasis in environmental soil physics in August 1993.