THE INFLUENCE OF TILLAGE AND LANDSCAPE ON IMAZETHAPYR PERSISTENCE AND SORPTION IN SOIL

BY

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for the Degree of

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THE UNIVERSITY OF MANITOBA

FACULTY OF GRADUATE STUDIES

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ABSTRACT

Anderson, Amber Lynn. M.Sc., The University of Manitoba, October, 2006. The Influence of Tillage and Landscape on the Persistence and Sorption of Imazethapyr in Soil. Major Professor; Annemieke Farenhorst.

The persistence and sorption of imazethapyr in conventional and reduced tillage soils in different soil landscapes was examined. Soils from two one-year field studies were used in bioassay and sorption batch equilibrium experiments. The bioassays indicated that herbicide bioactivity was most strongly influenced by herbicide concentration, organic carbon content, and clay fraction in Fairland clay loam and Hallboro sandy loam soils in the 1999 field study, and herbicide concentration, and organic carbon content in Newdale clay loam and Varcoe clay loam soils in the 2000 field study. No significant trends were observed between imazethapyr soil activity and tillage system or landscape position. The batch equilibrium results indicated that imazethapyr sorption was greater in soils under conventional tillage than reduced tillage, and Freundlich Kf values were greater in the lower landscape positions than the upper slope positions in both conventional and reduced tillage fields. No correlation was observed between imazethapyr sorption and either soil pH, organic carbon, or clay fraction.

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1.0 INTRODUCTION

Agriculture systems in Canada have undergone many changes in the last few decades. The dominance of conventional tillage and monoculture grain crops has lessened with the introduction of conservation or reduced tillage and crop rotations. Increasingly, agriculture producers are preparing the land using conservation tillage methods, which leaves some residue on the surface to protect the soil from wind and water erosion. Between 1991 and 2001, no-till seeding and conservation tillage in Canada increased by 23% and 6%, respectively, while conventional tillage decreased by 29% (Statistics Canada 2003).

The sustainability of reduced tillage is largely dependent on the ability to manage weed communities in the cropping system. As weeds are no longer eradicated by tillage, herbicides are needed for adequate weed control. Between 1995 and 2000, herbicide application to arable land increased by 11.3% and 4.2% in Canada and Manitoba, respectively (Statistics Canada 2003). Increased herbicide use over the last three decades have led to herbicide tolerance amongst varying weed species with the number of herbicide-tolerant weeds increasing from a single reported case in 1978 to 188 herbicide-tolerant weed types reported in 42 countries in 1997 (Heap 1997). Rotating herbicide groups will slow the onset of herbicide resistant weeds. However, the potential for some herbicides to persist in soil beyond a growing season needs to be considered prior to usage in the cropping system.

The potential for Pursuit® and Odyssey® to persist in soil has been a concern for producers, particularly since the introduction of Clearfield® canola, a variety with induced resistance to imazethapyr, an active ingredient in both these herbicide products. Imazethapyr is persistent in soil, with previous studies documenting the damaging effects of its residues on subsequent crops in rotation (Goetz et al. 1990; Loux et al. 1989; Mills and Witt 1989; Renner et al. 1988). Imazethapyr persists longer in soils with increased clay and organic matter contents due to increased sorption (Cantwell et al. 1989; Goetz et al. 1993; Loux et al. 1989), thus its use on Manitoba soils with higher clay and organic matter contents needs to be examined prior to inclusion in a herbicide rotation.

The aim of this study was to determine the potential for imazethapyr to persist in cultivated soils under conventional and reduced tillage. Field studies were conducted in 1999 and 2000 to evaluate the bioactivity of imazethapyr in soil over time. A laboratory study was also conducted to evaluate the sorption behavior of imazethapyr in soil.

2.0 LITERATURE REVIEW

2.1 Canadian Agricultural Production Systems

Conventional tillage is a traditional farming practice that uses cultivation as a method of weed control, and to prepare soil for seeding or summerfallow. Conventional tillage can be classified as either high or low disturbance. High disturbance action disturbs the entire soil surface resulting in the disappearance of the majority of plant residues, while low disturbance action leaves some of the soil undisturbed allowing some plant residues to remain on the surface. While conventional tillage has various benefits for cropping, problems with soil aggregation (Chang and Lindwall 1989; Dormaar and Lindwall 1989), soil erosion (Chang and Lindwall 1989; Lindstrom 1986; McGregor et al. 1975), and losses of organic matter (Carter and Rennie 1982; Dormaar and Lindwall 1989; Hamblin 1980) and soil moisture (Phillips et al. 1980; Voroney and Rennie 1985) forced producers to search for more sustainable methods.

Reduced tillage is a general term used to describe several types of residue management practices that minimize the intensity or frequency of tillage operations, but involve at least one significant cultivation practice (Locke and Bryson 1997; Saskatchewan Interactive 2002). In reduced tillage systems, some or the entire previous crop residue is left on the soil surface, and subsequent crops are seeded directly into the standing stubble.

Reduced tillage helps to restore and maintain soil quality, leading to an agronomically and economically more viable farming practice (Lafond and Derksen 1996). Benefits of reducing tillage include increases in organic matter content (Blevins et al. 1983; Dormaar and Lindwall 1989; Tyler et al. 1983) and soil moisture (Slack et al. 1978; Voroney and Rennie 1985) and decreases in soil erosion (Chang and Lindwall 1989; Lindstrom 1986).

The 2001 Agriculture census revealed that about 60% of all agriculture land in Canada was under no-till or reduced tillage. In Manitoba, conventional tillage remained the most prevalent form of tillage occurring on over half the cropland in the province in 2001, but less than the nearly two-thirds of conventionally tilled cropland in 1996.

Canola production is a major part of agriculture rotations as the oilseed crop helps reduce disease, insect and weed cycles common to continuous cereal cropping. The total value of canola seed, oil, and meal exports in Canada is approximately $2 billion (Canola Council of Canada 2004a). Canola is grown primarily in the Prairies, Ontario and British Columbia. In Manitoba, 2.5 million acres of canola was harvested in 2003 (Canola Council of Canada 2004a).

2.1.1 Tillage management and weed control

Weed communities present in cultivated fields are the result of the interaction of agronomic, environmental, and ecological selection pressures (Derksen 1996). These factors play an important role in weed community composition and subsequent control measures. Currently, the primary management strategies that can be used to control weeds are tillage, herbicides, and cropping practices (Derksen et al. 1996). Individually, these control measures can be effective, but the adaptation of two or more of these practices in cropping systems provides for better weed control (Clancey et al. 1993; Loeppky and Derksen 1994).

For many years, prior to the introduction of chemical herbicides into the cropping system, weed control was mainly achieved by destroying weeds through physical means. Tillage kills weeds through total plant burial; depleting food reserves by removing top growth; exposing root systems to drying and frost; and encouraging rotting of weed roots and stems (Government of BC 2001). The control of annual weed species by tillage is to prevent seed production and depletes current seed reserves in the soil. For perennials, the aim is to destroy roots in conjunction with the prevention of seed production and reduction in seed reserves (Government of BC 2001). However, problems with soil erosion and loss of soil fertility resulted in widespread adoption of more sustainable cropping practices, particularly reduced tillage systems (Saskatchewan Interactive 2002).

Weed management programs in reduced tillage systems rely largely on herbicides (Moyer et al. 1994). Herbicides replace tillage operations as the main tool in weed control. Some producers have reported using up to 20% more herbicide use in reduced tillage fields in comparison to conventional tillage fields (Baker 1985; Hanthorn and Duffy 1983; Lafond et al. 1993). Increased use of herbicides in reduced tillage systems is generally attributable to pre-plant application of herbicides such as glyphosate, paraquat, or 2,4-D to control weeds previously controlled by tillage passes. Studies show very little difference in the total amount of herbicide used in-crop in conventional or reduced tillage systems (Crosson 1981; Fawcett et al. 1994).

The type of tillage system affects weed community composition (Ball 1992; Blackshaw 1994), but changes in the nature and extent of weed populations are difficult to generalize. Green foxtail [*Setaria viridis*] was associated with reduced tillage in Ontario (Frick and Thomas 1992), but was prevalent in both reduced and conventional tillage fields in Saskatchewan (Hume et al. 1991). Yellow foxtail [*Setaria glauca*] and witchgrass [*Panicum capillare*] densities were greater in conventional tillage (Frick and Thomas 1992), while downy brome [*Bromus tectorum*] and wild oats [*Avena fatua*] were more common in reduced tillage (Derksen et al. 1993; Douglas et al. 1990). Some reports and surveys indicate that perennial quackgrass [*Agropyron repens*] showed no preference for either tillage system (Derksen et al. 1993; Frick and Thomas 1992), while other studies found quackgrass was more associated with reduced tillage (Legere et al. 1993; Mahli et al. 1988). Other perennial weeds such as Canada thistle [*Cirsium arvense*], sowthistle [*Sonchus arvensis*], dandelion [*Taraxaum officinale*], and foxtail barley [*Hordeum jubatum*] were more prevalent in fields under reduced tillage (Blackshaw et al. 1994; Derksen et al. 1993; Frick and Thomas 1992; Hume et al. 1991; Izaurralde et al. 1993). Broadleaf weeds more common in conventional tillage systems include lamb's quarters [*Chenopodium album*], stinkweed [*Thlaspi arvense*], and wild buckwheat [*Polygonum convolvulus*] (Blackshaw et al. 1994; Derksen et al. 1993; Legere et al. 1993; Moyer et al. 1994), while wild mustard [*Sinapis arvensis*] and flixweed

[*Descurainia sopia*] have been associated with reduced tillage (Derksen et al. 1993; Moyer et al. 1994).

Though it was predicted that grasses, perennial broadleaves, and volunteers would increase under conservation tillage (Froud-Williams et al. 1981), research indicates that generalizations regarding the response of weed species, or even weed communities, to a particular tillage practice are difficult to make (Derksen et al. 1996), because other agronomic practices, like crop rotation, may have a greater impact on weeds than tillage (Froud-Williams 1988).

2.1.2 Canola production and weed control

Canola [*Brassica napus* and *Brassica rapa*] is an edible type of rapeseed that was developed by Canadian researchers through traditional plant breeding methods in the 1970's (Berglund and McKay 1998; Busch 1994). The oil component is less than 2% erucic acid, and the meal component is less than 30 micromoles per gram glucosinolates (Canola Council of Canada 2004a). Since its introduction into the market place, canola oil has steadily become recognized as the healthiest oil available to consumers with low saturated and high monosaturated fatty acids, and a unique level of omega3 fatty acids and alpha-linolenic acids (Gianessi et al. 2002).

Canola was grown on approximately 11.6 million acres in Canada in 2003 with an approximate yield of 0.6 tonnes per acre (Canola Council of Canada 2004a). The average acreage seeded with canola in Canada between 1995 and 2003 was 11.3 million, in comparison to 8.4 million between 1986 and 1994 (Canola Council of Canada 2004a).

The increase in acreage is partially attributable to the commercialization of herbicide tolerant canola varieties in 1995, as well as the agronomic benefits of including canola in crop rotations (Gianessi et al. 2002). Currently, Canada's annual exports of canola seed, oil and meal are valued at over 2 billion dollars (Canola Council of Canada 2004a).

Weeds are a major limiting factor in canola production. Canola is a cool season crop that is most productive when seeded early in the spring from mid-April to mid-May (Gianessi et al. 2002). Canola is non-competitive against weeds in the early growth stages because it is slow growing and is slow to cover the ground, but the crop becomes very competitive against weeds once established (Berglund and McKay 1998; Gunsolus and Oelke 2000). Weeds affect canola production in two major ways. First, weeds reduce yields by competing with canola for light, moisture, and nutrients. Second, weed seeds, particularly from the mustard family, which are similar in size and shape, reduce the quality of canola oil and meal due to higher levels of erucic acid and glucosinolates (Davis 1999). Wild mustard [*Sinapis arvensis*] densities in canola as low as 20 plants per square meter resulted in wild mustard seeds accounting for 5% of the total harvested seed, which is the maximum tolerance of wild mustard in any grade of canola before subsequent price discounting and market rejection (Canola Council of Canada 2004b).

Weeds that cause problems in canola are the perennial types, shade tolerant weeds, and tall-growing, early germinating weeds such as wild oats [*Avena fatua*] (Canola Council of Canada 2004b). The more troublesome weeds include wild mustard, wild oats, volunteer wheat [*Triticum aestivum*] and barley [*Hordeum vulgare*], Canada thistle [*Cirsium* *arvense*], sowthistle [*Sonchus arvensis*], and quackgrass [*Agropyron repens*] (Canola Council of Canada 2004b). Wild oats is a major weed in small-grain cereals. As these crops are predominant in most rotations, wild oats has become a major weed in canola production included in these rotations. Left unchecked, 8 wild oat plants per square meter can reduce canola yields by 9% (Canola Council of Canada 2004b). Volunteer barley and wheat are weed pests in canola that is sown into cereal stubble. Both plants are highly competitive, but barley is more competitive than wheat. Volunteer wheat and barley at 7 to 8 plants per square meter can reduce canola yield by 10% to 13% (Canola Council of Canada 2004b). Canada thistle is a strong competitor against canola. In North Dakota, more than 60% of canola acres are infested with Canada thistle (Coleman and Jenks 1999). Twenty Canada thistle shoots per square meter can reduce canola yields by 24% (University of Manitoba 2005). Sowthistle is commonly found with Canada thistle, and can reduce canola yields by 27% at 20 plants per square meter (Canola Council of Canada 2004b). Quackgrass grows most vigorously in cool, moist spring conditions, which can seriously impact canola establishment. The economic threshold of quackgrass in canola is approximately 20-25 shoots per square yard (University of Manitoba 2005).

The most predominant herbicides used for weed control in canola include trifluralin, ethalfluralin, sethoxydim, and quizalofop (Canola Council of Canada 2004b; Gianessi et al. 2002; USDA 1999). Trifluralin and ethalfluralin are the only preplant soil incorporated herbicides presently labeled for weed control in canola (Canola Council of Canada 2004b; Gianessi et al. 2002). Both herbicides will control most annual grasses and some broadleaf weeds such as kochia, Russian thistle [*Salsola kali*], and redroot pigweed [*Amaranthus retroflexus*] (Canola Council of Canada 2004b; Gianessi et al. 2002; Gunsolus and Oelke 2000). Control of wild oat densities in canola using trifluralin has been contradictory, with some areas in Canada and the United States obtaining excellent control and other areas obtaining poor to fair control (Gunsolus and Oelke 2000).

Post-emergent herbicides sethoxydim, quizalofop, fluazifop, fenoxaprop, and clethodim are available for grassy weed and volunteer cereal control in canola. Clopyralid is a broadleaf herbicide that provides good control of Canada thistle, sowthistle, and wild buckwheat [*Polygonum convolvulus*], while ethametsulfuron controls stinkweed [*Thlaspi arvense*], and smartweed [*Polygonum pennsilvanicum*], and is currently the only herbicide available that will selectively control weeds closely related to canola, such as wild mustard, without significant crop injury (Canola Council of Canada 2004b; Gianessi et al. 2002; Harker 1995). Other challenges that threaten canola production include herbicide carryover and herbicide resistant weeds (Gianessi et al. 2002). Canola is sensitive to many herbicides used on previous rotational crops. Drift from adjacent fields may also cause crop injury by herbicides like imazethapyr (Moyer and Esau 1996).

Herbicide resistant canola was introduced into the Canadian market in 1995. Tolerance to specific herbicides has been introduced as a novel trait into canola to provide farmers with additional means to control weeds without damaging their crops. There are two genetically modified herbicide tolerant canola systems currently available, Roundup

Ready® and LibertyLink®. Clearfield® canola is a third type of herbicide resistant canola, but it is not transgenic.

Clearfield® canola is resistant to the imidazolinone class of herbicides. The mode of action of imidazolinones is the inhibition of acetohydroxyacid synthase (AHAS) (also referred to as acetolactate synthase or ALS), a major enzyme in the biosynthesis of valine, leucine, and isoleucine amino acids (Miflin 1971). The activity of AHAS in Clearfield® canola is tolerant of inhibition by imidazolinones. This tolerance was developed through traditional plant breeding methods, namely mutagenesis (Swanson et al. 1989). Mutagenized microspores were cultured in the presence of imidazolinone herbicides. Susceptible microspores were discarded, while tolerant microspores were selected and allowed to develop into embryos from which tolerant plants grew (Swanson et al. 1989). As a result, Clearfield® canola is internationally recognized as a non-genetically modified organism since it contains no introduced genetic material.

Herbicide resistance of LibertyLink® and Roundup Ready® canola was derived from the introduction of genetic material from another organism. LibertyLink® canola is resistant to glufosinate, a non-selective, non-residual, contact herbicide that effectively controls annual broadleaf and grass weeds, and can suppress perennial weeds (Berglund and McKay 1998). The gene that imparts resistance to the herbicide glufosinate, isolated from the bacteria *Streptomyces hygroscopicus*, encodes for phosphinothricin acetyl transferase (PAT), an enzyme that catalyzes the metabolic inactivation of glufosinate (Gianessi et al. 2002).

Roundup Ready® canola is resistant to glyphosate, a non-selective, non-residual, systemic herbicide capable of controlling most annual and perennial weeds (Berglund and McKay 1998). Glyphosate phytotoxicity is derived from the inhibition of 5-enolpyruvyl-shikimate-3-phosphate synthase (EPSPS), an enzyme that is essential to the synthesis of aromatic amino acids. Roundup Ready® canola carries a gene that codes for a glyphosate-insensitive (EPSPS) enzyme that allows Roundup Ready® canola to remain unaffected by glyphosate application (Gianessi et al. 2002).

Since its introduction into the market place in 1995, herbicide resistant canola acreage in Canada has risen rapidly. Increased yields up to 10% and an average increase in net return of $5.80/acre have propelled many producers to switch to herbicide tolerant canolas (Canola Council of Canada 2004a). Currently, transgenic canola systems LibertyLink® and Roundup Ready® account for approximately 70% of the canola acreage in Canada, while Clearfield® varieties account for approximately 18%, and conventional varieties 12% (Canola Council of Canada 2004).

2.2 Imazethapyr

2.2.1 Chemical properties and characteristics

Imazethapyr herbicide products were introduced in 1983, and initially marketed for use in soybean crops in the United States. Currently, in Manitoba, it is registered for use in field peas, dry beans (pinto, pink, and red), soybeans, seedling alfalfa, established alfalfa and chickling vetch for seed production, and Clearfield® canola varieties (Guide to Crop Protection 2005). It is marketed by BASF Canada under the label names Pursuit® (240 g/L imazethapyr), Pursuit Ultra® (240 g/L imazethapyr, 450 g/L sethoxydim), Odyssey®

(35% imazethapyr, 35% imazamox), and Absolute® (35% imazethapyr, 35% imazamox, 360g/L clopyralid). As a selective, systemic herbicide, imazethapyr provides flexibility in both its application and control. It offers effective weed control in low doses, and can be applied as a pre-emergent, pre-plant incorporated or post-emergent herbicide (Table 2.1). As either a foliar or soil application, it provides significant activity for immediate and long-term control of noxious weeds.

Table 2.1 Weed control by imazethapyr in Clearfield® canola

Weed	Control*
Chickweed	Good
Cleavers	Good
Green foxtail	Good
Lamb's quarters	Suppression
Redroot pigweed	Good
Smartweed	Good
Stinkweed	Excellent
Volunteer barley	Suppression
Volunteer canola (non-Clearfield® varieties)	Good
Volunteer wheat	Suppression
Wild buckwheat	Good
Wild mustard	Excellent
Wild oats	Fair

* As indicated in the Manitoba Guide to Crop Protection 2005

Imazethapyr belongs to the imidazolinone chemical class of compounds, which also includes herbicidal chemicals imazaquin, imazapyr, imazamox, and imazamethabenz-methyl. The imidazolinones contain three structural sections: the imidazolinone ring, the acid or acid equivalent, and the backbone. Differences in the acid equivalents or substituents on the backbone distinguish these compounds from one another. Imazethapyr is an amphoteric molecule possessing both an acidic carboxyl, and basic imidazolinone and pyridine functional groups (Figure 2.1).

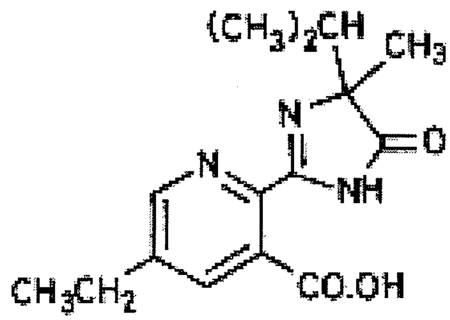

Figure 2.1 Imazethapyr molecule

The chemical has both a low water solubility and low vapor pressure, while its octanol-water partitioning coefficient is pH dependent (Table 2.2). Imazethapyr has two ionization constants. The first constant, pK_1, corresponds to the protonation of the imidazolinone ring, while the second constant, pK_2, corresponds to the ionization of the carboxylic acid group. The half-life of imazethapyr in soil ranges from 3.2 to 12 years depending on environmental conditions (Shaner and O'Connor 2000).

Table 2.2 Selected physical and chemical properties of imazethapyr.

Chemical Formula	$C_{15}H_{19}N_3O_3$
Molecular weight	289.34
Melting point	169 - 173°C
Vapour pressure @ 60°C	$<1 \times 10^{-7}$ mmHg
Water solubility	0.14g / 100mL
Ionization constants	$pK_1 = 2.1$
	$pK_2 = 3.9$
Half-life	3.2 - 12 years
Partition coefficient @ 25°C	11 @ pH 5
(*n*-octanol/water)	31 @ pH 7
	16 @ pH 9

2.2.2. Interaction with plants

Imazethapyr is a systemic herbicide that can be applied pre- or post-emergently to a cropping field to control weeds. It is an ideal chemical for translocation in plants due to its intermediate permeability and weak acid nature. Imazethapyr present in the soil solution could enter plants through the root absorption, and be translocated to the leaf surface via the xylem water transport system in the apoplast. If imazethapyr is foliar applied, it enters through breaks in the leaf cuticle, and moves into the phloem carbohydrate transport system in the symplast. The herbicide is then transported in the phloem tissue to the meristematic regions, the herbicide site of action.

The mode of action of imidazolinones is the specific inhibition of the Acetohydroxy acid synthase (AHAS) enzyme. AHAS is the essential enzyme in the synthesis of branched-chain amino acids, and is located in the stroma of the chloroplasts in green tissue, and the plastids of non-green tissue.

Imazethapyr binds to AHAS and inhibits its activity leading to reduced amounts of valine, leucine and isoleucine in the plant. Anderson and Hibberd (1985) found that free amino acid pool sizes for leucine and valine in corn decreased by greater than 50% when exposed to imidazolinone herbicides while the levels of other free amino acid pools increased. The effects of imidazolinones on free amino acid pools and soluble protein pools suggest that imidazolinones disrupt protein synthesis by changing the turnover rate of pre-existing proteins. The decrease in the synthesis of branched-chain amino acids following herbicide application may signal the plant that nitrogen is limited, resulting in an increased rate of protein turnover (Shaner and O'Connor 2000). Mature plant tissue has larger amino acid pools as well as larger protein reserves that can be catabolized for new amino acids. In contrast, the younger meristimatic regions of the plant, where AHAS is most active, require higher levels of amino acids for protein synthesis, cell division and growth yet contain fewer protein reserves. As a result, symptoms caused by application of imidazolinones appear first in younger tissue rather than mature tissue in a growing plant. These symptoms include chlorosis followed by necrosis of the meristems; purple coloration of the leaves and stems; root growth inhibition; and slow necrosis of mature tissue.

Starvation of branched-chain amino acids causes secondary factors that lead to plant mortality. Susceptible plants treated with imazethapyr show inhibition of cell division and growth. Rost et al. (1990) found imazethapyr halted cell division in pea roots during interphase, and subsequent supplementation of the three branched-chain amino acids prevented the inhibition of cell division by the herbicide. These results suggest a close relationship between inhibition of the synthesis of the branched-chain amino acids and DNA synthesis, cell division, and growth (Shaner and O'Connor 2000). Another factor that contributes to plant mortality is the build up of sugars in leaves, and the unavailability of carbohydrates to other parts of the plant. The cessation of phloem transport caused by AHAS inhibition disrupts the translocation of photosynthates to the roots thereby cutting off their only energy supply, which ultimately leads to plant death. A third factor is the interference with plant hormones. The meristems are the source and site of action of growth and development regulating hormones such as auxins and gibberellins. The inhibition of AHAS disrupts the plants' metabolism at the meristems thereby causing changes in the hormonal balance, which may indirectly affect plant growth.

Approximately three to five days after treatment, phloem translocation of imazethapyr ceases due to the limiting nature of the mode of action of the imidazolinones. Translocation of these herbicides is limited by the availability of the branched-chain amino acids since a deficiency of these amino acids causes the cells in the meristems to stop dividing, thereby decreasing the need for photosynthates. Therefore, phloem transportation to the meristems ceases, and the herbicide does not reach the site of action. The inhibition of phloem transport reduces the usefulness of imazethapyr for control of perennial weeds whose roots must be affected by the herbicide for adequate control.

Selectivity of imazethapyr among crops and weeds is dependent on the rate of break down of the herbicide to non-toxic transformation products. Many grasses and broadleaf weeds are controlled in leguminous crops due to differences in metabolic rates of herbicide transformation between crops and weeds. Imazethapyr metabolizes to non-toxic forms via hydroxylation of the ethyl substituent on the pyridine ring followed by conjugation to glucose (Shaner and O'Connor 2000). The hydroxylation of imazethapyr occurs in a wide range of plant species, but the ensuing metabolite retains herbicidal capabilities. The imazethapyr molecule becomes non-toxic only after the hydroxylated metabolite is conjugated with glucose. However, rapid hydroxylation of the herbicide immobilizes it at the site of application, thereby reducing its toxicity since only a limited amount reaches the growing points. A susceptible plant metabolizes the herbicide very slowly, and is therefore controlled by the herbicide. Some species show intermediate tolerance by being able to hydroxylate the herbicide, but unable to conjugate it with glucose. Tolerant species, like soybeans and Clearfield® canola, rapidly metabolize imazethapyr to non-toxic forms.

2.2.3 Behavior in soil

Imazethapyr in soil is subjected to three main processes: sorption, transformation, and transportation. Sorption refers to the binding of the herbicide onto soil mineral fractions, mainly clay, and other soil constituents such as soil organic matter. Transformation of imazethapyr occurs due to chemical degradation and biodegradation processes. Biodegradation requires the presence of microorganisms capable of degrading the herbicide. Abiotic degradation refers to chemical reactions in soil (without the need for soil fauna) or at the soil surface under the influence of sunlight (photodegradation). Transportation of imazethapyr includes volatilization (the evaporation of herbicides), leaching, and transport by wind and water erosion.

2.2.3.1 Sorption

Soil behavior of ionic herbicides, like imazethapyr, is greatly influenced by soil pH, clay content and organic matter content. These properties affect the availability of imazethapyr in soil solution, and thus, its biological activity. The more the herbicide binds to soil colloids, the less available it becomes for uptake by plant roots, thereby reducing its efficacy against target organisms.

Soil constituents impact the sorption of ionic herbicides by providing charged adsorption sites on colloid surfaces. Soil solids are made up of mineral (inorganic) material and organic matter at various stages of decomposition. Inorganic material consists of sand, silt and clay particles. Clay minerals are made up of sheets of silica tetrahedra and aluminum octahedra, referred to as layered silicates. The source of negative charge of clay minerals arises from isomorphic substitution, the replacement of the Si or Al cations with cations of lesser charge, and occurs predominantly in 2:1 minerals, montmorillonite, vermiculite, and illite, with little substitution in 1:1 minerals, like kaolinite (Tisdale et al 1993). This inherent negative charge is uniform over the surface of the particle, and unaffected by soil pH. Another source of charge arises from broken edges of layered silicates, but this charge is pH dependent. Soil organic matter provides another source of pH dependent charge. The negative charge originates from the dissociation of hydrogen ions from the carboxylic acid and phenolic groups (Tisdale et al. 1993). Increases in soil pH cause increases in negative charge on the surface of organic matter as hydrogen ions are neutralized.

Soil pH greatly influences the sorption and efficacy of ionic herbicides by affecting the ionic character of the soil organic matter, clay surfaces, and the ionization of the herbicide molecule. Sorption of ionizable herbicides is greatest near their dissociation constants (Best et al. 1974; Kells et al. 1980). The acidic and basic pKa's of imazethapyr are 3.9 and 2.1, respectively (Shaner and O'Connor 2000). At near-neutral soil pH, imazethapyr will be primarily in an anionic form, and repulsed from negatively charged organic matter and clay surfaces. As a result, greater concentrations of the herbicide should be available in soil solution for plant uptake. Conversely, as pH decreases, protonation of the nitrogen atoms on the imidazolinone ring occurs, resulting in a neutral or positively charged molecule, thereby increasing the potential for sorption (Stougaard et al. 1990). When studying the effects of pH on imidazolinone sorption, Shaner and O'Connor (2000) observed that small differences in pH levels, 0.2 to 0.4 units, can double the Freundlich adsorption coefficient (Kf) of imazethapyr in the range of pH 3 to 6 because lowering the pH in small increments greatly increases the amount of imazethapyr with weak positive charges on its nitrogen atoms.

Loux et al. (1989) examined the sorption of imazethapyr and imazaquin on 22 samples with varying soil properties. Multivariate regression analysis of the Freundlich Kf values and soil properties indicated that organic matter and pH were important variables in imazaquin sorption, while imazethapyr sorption was strongly influenced by clay content and pH. Greater sorption of both herbicides occurred at pH levels below 6, with regression models reflecting an increased effect of pH on adsorption in the pH range of 4 to 6. Che et al. (1992) examined sorption of imazaquin and imazethapyr on illite, kaolinite, hectorite and two humic acids. The study determined that sorption of both herbicides increased as pH decreased, but the increased sorption was most evident as soil pH decreased from 5 to 3. The study also showed that the humic acids had a much greater affinity for both herbicides than the clay minerals, though imazethapyr had a greater affinity to clays than imazaquin. However, the researchers concluded that the extent of sorption on the clay minerals indicated that soil clay content could affect availability to plants. Similar conclusions were reached by Stougaard et al. (1990) after observing that both imazaquin and imazethapyr sorption was greatest in a finer silty clay loam soil than a sandy loam soil, and the herbicides were more strongly sorbed, less mobile, and less efficacious at lower pH levels in both soils. A study by Renner et al. (1988) determined that sorption of imazethapyr and imazaquin increased as soil pH decreased, with significantly more imazethapyr sorbed than imazaquin at pH levels 3.0 and 5.5, while Goetz et al. (1990) examined imazethapyr persistence in two soils with similar pH values observing greater sorption in the soil with the higher clay and organic matter content. Contrarily, Gennari et al. (1998) found no correlation between sorption coefficients and clay content when studying imazethapyr sorption on clays and humic acids at pH levels ranging from 3.6 to 8.3. However, for both the clays and humic acids, adsorption was promoted by low pH values.

Soil moisture also plays an important role in herbicide adsorption. Soil moisture affects the amount of herbicide in soil solution, and thus, its availability for plant uptake. Researchers observed that binding of imazaquin to soil colloids increased as soil moisture decreased resulting in reduced herbicidal efficacy (Shaner and O'Connor 2000). Allen and Casely (1986) examined the efficacy of imazamethabenz-methyl on blackgrass. Soil moisture did not significantly influence the dry weight of blackgrass when treated pre-emergence or late post-emergence (two leaves, one tiller) with imazamethabenz-methyl. However, when imazamethabenz-methyl was applied early post-emergence (one leaf), increased injury to blackgrass occurred as soil moisture increased. Pillmoor and Casely (1982) observed that imazamethabenz-methyl activity was greater in waterlogged soils, particularly if the soil was waterlogged on the day of treatment, though the extent of injury was dependent on the species' susceptibility to the herbicide. Further studies by Pillmoor and Casely (1984) examined the importance of foliar and soil activity of imazamethabenz-methyl applied to wheat under waterlogged conditions. The herbicide caused very little injury to wheat when applied only to foliage, but caused significant injury when applied to the soil or to foliage and soil. Furthermore, the researchers observed that when waterlogged conditions were maintained for seven days, beginning either one day before herbicide treatment, one day after treatment, or seven days after treatment, significant injury to wheat occurred even when waterlogging was delayed until seven days after herbicide treatment. Subsequent treatments of ^{14}C- imazamethabenz-methyl to wheat under field capacity and waterlogged conditions revealed that the plants under waterlogged conditions took up a greater amount of herbicide, indicating greater herbicide efficacy with increasing moisture. Field studies by Loux and Reese (1993) found that sufficient soil moisture resulted in rapid dissipation of imazethapyr, and the herbicide was less persistent than the previous year when the soil was significantly drier. Imazapyr sorption studies by Dickens and Wehtje (1986) observed that as soil moisture decreased imazapyr sorption increased. These studies reveal that sufficient moisture is required to bring herbicides into soil solution, or to promote desorption of herbicides from soil colloids and become available to plants.

The effect of rainfall on herbicide sorption varies depending on rainfall amount, intensity, and the timing of the herbicide application. For example, rain following application promotes herbicide efficacy, as more herbicide is available in soil solution for plant uptake. In general, rainfall effects are more significant in coarse-textured soils than finer soils with high clay and organic matter content. Ayeni et al. (1998) studied rainfall influence on imazethapyr bioactivity. The researchers observed that rainfall amount had a strong influence on the herbicidal activity of imazethapyr especially in coarse-textured soils. Under rainfall intensities greater than 25 mm/hr, herbicide activity in sandier soils declined as imazethapyr leached beyond the rooting zone as the small clay and organic matter content was insufficient to bind the herbicide. In contrast, rainfall intensities up to 25 mm per hour following imazethapyr application to these coarse-textured soils did not significantly influence herbicidal action. Furthermore, the efficacy of imazethapyr decreased with increasing time between herbicide application and rainfall, particularly for coarse-textured soils and to a lesser extent for fine-textured soils.

2.2.3.2 Transportation

Volatilization and leaching can reduce the amount of herbicide in the soil available for plant uptake. However, imidazolinones have low vapor pressures ensuring that volatilization from soil will be minimal and not an important dissipation process for this class of herbicides. Goetz et al. (1990) examined the volatilization of imazethapyr from a silty loam and a silty clay soil using formulated and ^{14}C-labeled imazethapyr. The researchers observed that only 2% of applied imazethapyr volatilized from the soil surface, with no significant difference in volatilization losses between soil types. In addition, Weber (1994) concluded that imazethapyr is not lost from the soil surface through volatilization.

Dissipation and downward movement of imazethapyr in soil has been examined by several researchers who have concluded that imazethapyr has a small leaching potential

(Shaner and O'Connor 2000). Jourdan et al. (1998) studied the effects of rainfall (12.5 mm/h, 3 times/week) on imazethapyr distribution in soil. Ninety days after imazethapyr application, the greatest concentration of imazethapyr was still in the top 15 cm of soil, and no imazethapyr was detected below 30 cm. After examining imazethapyr mobility in soil under three watering regimes, Basham and Lavy (1987) concluded that under typical field conditions, leaching is not a major dissipation factor of imazaquin. Similarly, Renner et al. (1988) found that 150 days after application, imazaquin residues remained predominantly in the top 5 cm of the soil profile. Researchers conducting an experiment on the downward movement of imazethapyr in soil under conditions that would promote leaching (maximum rainfall expected in the area, coarse-textured soil, unrealistically high herbicide application rates) detected imazethapyr residues in the 30 to 45 cm layer on only two sampling dates, and detected no imazethapyr below 45 cm (Shaner and O'Connor 2000). The researchers concluded that imazethapyr soil residues have limited potential to contaminate groundwater through leaching regardless of soil texture or rainfall amount.

Many studies have observed that the largest quantities of herbicide lost in runoff are during rainfall events soon after application (Gaynor et al. 1992; Myers et al. 1995). These studies concluded that intensity and timing of rainfall, as well as the solubility of herbicides in water are extremely important in influencing the extent of herbicide lost in runoff. The study by Ayeni et al. (1998) found that intensity of rainfall did not significantly influence imazethapyr efficacy, indicating that the herbicide did not move to any great extent from the site of application, while rainfall following application was actually beneficial for herbicidal action. Furthermore, imazethapyr has low water solubility, indicating that surface runoff is not a major route of transportation for imazethapyr.

2.2.3.3 Transformation

Transformation of herbicides in soil occurs from abiotic and biotic processes that convert the herbicide into secondary metabolites. Biodegradation of imazethapyr is dependant on the presence of microbial populations in soil capable of degrading the chemical. Cantwell et al. (1989) studied the biodegradation characteristics of imazaquin and imazethapyr, and concluded that the primary mechanism of imidazolinone degradation in soils is enzymatic. The study showed that after 12 weeks of incubation, 95% of radio-labeled herbicide extracted from the sterilized soil was unaltered parent material, in comparison to non-sterilized soils that showed extensive herbicide degradation. Thus imazethapyr degradation occurs primarily through biotic processes. The results also showed a good correlation between the amount of herbicide in soil solution and the amount degraded, and that degradation was dependant on soil type. Basham and Lavy (1987) examined imazaquin microbial degradation in five soil types over a period of 7 months. The researchers concluded that degradation of imazaquin is accomplished primarily through microbial activities, with greater degradation occurring in the silty loam soils compared to the silty clay soil that had the highest amount of organic matter. Goetz et al. (1990) examined the degradation and field persistence of imazethapyr. They also observed that soil type significantly influenced the total $^{14}CO_2$ evolved and that $^{14}CO_2$ evolution was inversely related to clay and organic matter content. Since the soils had similar microbial respiration, the researchers concluded the increase in $^{14}CO_2$ from the coarser soil was probably due to lesser herbicide sorption and increased availability of imazethapyr to microorganisms. The study also revealed that degradation of imazethapyr was influenced by moisture and temperature, similar to other studies on herbicide degradation, which found that increasing soil moisture content and temperature increased the rate of degradation (Basham and Lavy 1987; Parker and Doxtader 1983).

Goetz et al. (1990) also examined photodecomposition of imazethapyr and observed that significantly more imazethapyr photodegraded as irradiation time increased, though less photodegradation occurred in comparison to imazaquin, as observed by Basham and Lavy (1987). When imazethapyr was applied to the soil surface and irradiated for 43 hours (approximately four days of normal exposure), the maximum photodecomposition losses were 10%. It was concluded that rainfall within 4 days of application would significantly minimize photolysis of imazethapyr because of the movement of imazethapyr below the 0-1 cm soil surface.

2.3 Influence of tillage management on herbicide behavior in soil

Tillage can influence herbicide dissipation and distribution in the soil by affecting the amount of plant residues on the surface and through soil mixing (Curran 1992). In a study by Mills et al. (1989), less clomazone was recovered from an untilled soil with wheat residue (mainly stubble) than a tilled soil with limited wheat residue. An experiment by Koppatschek et al. (1989) recorded greater recovery of metribuzin from soils with low levels of corn residue then soils with higher levels of residue, while Curran et al. (1992) found that plant residues did not influence herbicides reaching the soil surface.

Tillage can also dilute herbicide concentration by mixing the topsoil with a larger volume of subsoil, thereby lessening subsequent crop injury (Shea 1985). Burnside (1974) observed less oat injury by pre-plant-applied trifluralin and nitralin when the soil was plowed prior to seeding. Corn injury by trifluralin residues was reduced in tilled soils versus no-till soils (Hartzler 1989). In contrast, Curran et al. (1992) found corn displayed greater levels of injury by clomazone, imazaquin, and imazethapyr when the herbicides were pre-plant incorporated than when the herbicides were applied pre-emergently.

Imazethapyr efficacy and mobility in soil is largely dependent on availability of herbicide residues in soil solution, which is affected by pH, organic matter content, and clay fraction (Stougaard et al. 1990). The type of tillage practiced on the soil can influence these soil properties. Generally, soils under conventional tillage have decreased organic matter content (Hamblin 1980; Dormaar and Lindwall 1989; Carter and Rennie 1982) and soil moisture (Voroney and Rennie 1985; Phillips et al. 1980), and higher pH (Dick 1983; Levanon et al. 1994) than those under reduced tillage. These differences can strongly influence herbicide efficacy, mobility, and persistence.

The focus of this study is to examine imazethapyr behavior in soil by conducting field and laboratory experiments on soils with various textures derived from flat and undulating landscapes in western Manitoba under conventional and reduced tillage.

3. THE INFLUENCE OF TILLAGE AND LANDSCAPE ON THE RESIDUAL ACTIVITY OF IMAZETHAPYR IN SOILS

3.1 Abstract

Effects of tillage and landscape position on the persistence and residual activity of imazethapyr were investigated in two 1-year field studies. Two herbicide products containing imazethapyr, Odyssey® and Pursuit®, were applied at recommended field rates to conventional and reduced tillage soils under varying slope positions during two growing seasons. Oat bioassays indicated that herbicide bioactivity (%HB) was most strongly influenced by herbicide concentration, organic carbon content, and clay fraction in the 1999 field study, and herbicide concentration, and organic carbon content in the 2000 field study. No consistent trends were observed between imazethapyr activity in soil and tillage system or landscape position. Pursuit® efficacy was greater than Odyssey® efficacy in all four soil series. Modest dissipation and degradation of imazethapyr was observed during the winter in reduced tillage soils in comparison to conventional tillage soils where herbicide bioactivities noticeably decreased. These decreases were attributed spring tillage events. Herbicide carry-over from one year to the next was small in most treatment soils and not influenced by tillage system or slope position.

3.2 Introduction

Imazethapyr is an effective pre- or post-emergent herbicide capable of controlling a broad spectrum of noxious weeds throughout the growing season. It belongs to the imidazolinone chemical class of compounds. The systemic herbicide is used primarily in leguminous crops, but also in conjunction with Clearfield® canola, a non-genetically modified herbicide-tolerant canola (HTC) hybrid resistant to imidazolinone herbicides. Currently, more than half the canola grown in Canada is of an herbicide-tolerant variety including Clearfield®, Roundup Ready®, and Liberty Link®. Clearfield® canola accounted for 18% of the total amount of canola seeded in Canada in 2004 (Canola Council of Canada 2004b). As imidazolinones are the only chemical option for weed control in Clearfield® canola crops, applications of herbicides containing imazethapyr have consequently increased.

Following application, most imazethapyr residues remain in the top 0-15 cm soil layer (Jourdan et al. 1998) because the herbicide has relatively low water solubility and is not mobile in soil. Imazethapyr is soil-active and residues present at one year following application caused injuries to corn (Loux and Reese 1993; Curran et al. 1992), wheat (Stougaard et al. 1990), cucumber, and grain sorghum (Vencill et al. 1990).

Imazethapyr persistence and bioavailability to plants and microorganisms are influenced by the extent of imazethapyr sorption onto soil (Cantwell et al. 1989; Goetz et al. 1990). Imazethapyr persistence increases with decreasing soil pH, particularly at pH levels below 6 (Loux et al. 1989a). Greater imazethapyr sorption in acidic soils increased imazethapyr persistence, relative to soils with a near-neutral soil pH (Loux and Reese

1993). Imazethapyr sorption also increases with increasing clay fraction and soil organic matter content (Goetz et al. 1990; Senesi et al. 1997). Imazethapyr persistence decreases with increasing soil temperature and moisture content (Jourdan et al. 1998; Vischetti 1995) as both chemical and microbial decomposition rates increase with high temperature and moisture levels (Goetz et al. 1990).

Soil characteristics are influenced by tillage operations and landscape position (Mallawatantri et al. 1992; Elliot and Efetha 1999). Reduced tillage soils tend to have a higher amount of organic carbon in the surface soils (Andruilo et al. 1987; Doran 1980; Lal et al. 1994), as well as a greater amount of specific soil organic components, like amino acids and carbohydrates (Arshad et al. 1990; Angers et al. 1993). Levanon et al. (1994) found that the pH of reduced tillage soils is often more acidic than conventional tillage soils, and thus, reduced tillage soils would more strongly sorb weak bases and amphoteric molecules, like imazethapyr. An increased aggregate size and higher proportion of water-stable aggregates associated with reduced tillage soils results in better tilth and water-holding capacity of the soil (Anger et al. 1993). As a result, these soils tend to have higher moisture levels than conventional tillage soils.

Soil properties can differ considerably between cropping system and topography. Subsequently, herbicide carryover can fluctuate significantly within fields and regions. Though the effects of various physical and chemical characteristics of soil on imazethapyr bioactivity have been well documented (Goetz et al. 1990; Loux and Reese 1993), there has been limited research on the effects of tillage and landscape variability on imazethapyr soil activity, particularly on near-neutral Canadian Prairie soils.

The objective of this study was to evaluate the effects of tillage and landscape position on the persistence of imazethapyr in agricultural soils of varying textures.

3.3 Materials and Methods

3.3.1 Description of Sites and Soils

Field studies were conducted during the summer months of 1999 and 2000. In May 1999, a field study was set up to examine the persistence of imazethapyr in coarse- and fine-textured soils under conventional and reduced tillage. Four sites were chosen based on soil textural characteristics and tillage system. Two sites were located south of Brandon, Manitoba and two sites were located near Neepawa, Manitoba. The reduced tillage sites selected for this field study had been under reduced tillage for at least seven years. The conventional tillage soils were medium to high disturbance tilled for at least five years with a minimum of two tillage passes per year. The topography at all locations was level (< 2% slope). The two Brandon soils were imperfectly drained, Orthic Black Chernozems from the Fairland soil series with a clay surface texture for the reduced tillage plot and a clay loam surface texture for the conventional tillage plot. The Neepawa soils were well-drained, Orthic Black Chernozems from the Hallboro soil series with sandy loam surface textures for both the reduced and conventional tillage plots.

In June 2000, a field study set up to examine the persistence of imazethapyr in upper and lower slope areas under conventional and reduced tillage. Four sites were chosen for this study based on soil topography and tillage system. The sites were located north of

Brandon, on the Manitoba Zero-tillage Research Farm, and on an adjacent conventional tillage farm. The topography at the sites was hummocky with slopes between 2 – 5%. The soils from the upper slope were well drained, Orthic Black Chernozems from the Newdale soil series. The lower slope soils were imperfectly drained, Gleyed Rego Black Chernozems from the Varcoe soil series. All four treatment soils had a clay loam surface texture.

3.3.2 Field Studies

3.3.2.1 Site Preparation. Field experiments were set up in a split block design with reduced and conventional tillage as main plots, and the two herbicide products, Pursuit® and Odyssey®, as subplots. All plots were 16 m x 14 m in size. The subplots measured 5 m x 10 m with a 2 m perimeter around each subplot to avoid herbicide drift (Figure 3.1). This allowed for imazethapyr persistence to be observed in four treatment plots for each field season. In 1999, the four plots were Fairland clay loam soil under conventional (FCT) and Fairland clay soil under reduced tillage (FRT), and Hallboro sandy loam soils under conventional (HCT) and reduced tillage (HRT). In 2000, the four plots were Newdale clay loam soils under conventional (NCT) and reduced tillage (NRT), and Varcoe clay loam soils under conventional (VCT) and reduced tillage (VRT).

Prior to application of Odyssey® and Pursuit®, the plots were sprayed with RoundUp®, and rogued to ensure uniform ground coverage of herbicide spray. The herbicide products were applied to the soils at recommended field rates: 0.085 L/acre for Pursuit® and 0.017 kg/acre for Odyssey®, with Pursuit® containing 20% more imazethapyr than

Odyssey®, and Odyssey® also containing 35% imazamox. The herbicides were applied with a tractor-mounted sprayer, using a 5 m offset boom, and 8001SV nozzles. These nozzles were used to prevent boom bounce and provide more uniform ground coverage.

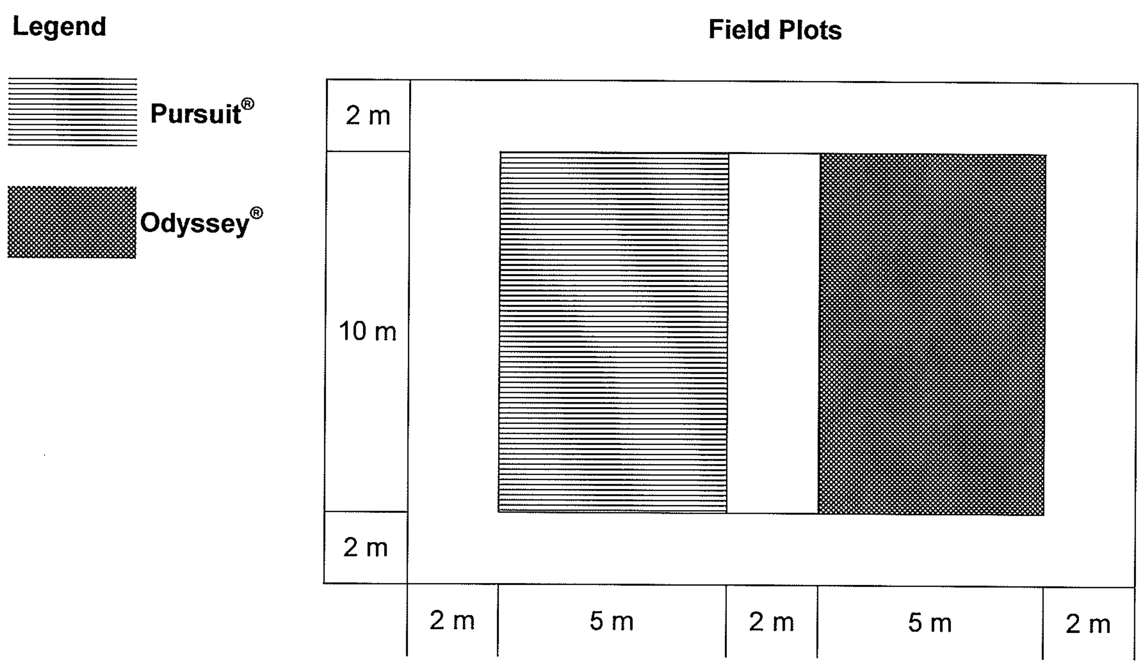

Figure 3.1 Split block design for all field sites

3.3.2.2 Soil Sampling and Preparation. Sampling of the plots commenced four days following herbicide application. The soil samples were collected using 5 cm radius PVC pipe hammered into the soil to a depth of 10 cm. Five random, composite samples were obtained for each subplot, and sealed in plastic freezer bags. Clean soil samples were collected outside the plots, as well. The samples were placed in coolers with ice packs for transport to the laboratory. All samples were placed in freezers until analyzed. In 1999, the plots were also sampled on days 8, 16, 32, 64, 96, and approximately one year following application (323 and 303 days for Fairland clay loam soils and Hallboro sandy loam soils, respectively). In 2000, subsequent sampling of the plots occurred on days 12, 24, 48, 96, and 304. Rainfall events in the days following application in 2000 prohibited sampling until 12 days after application.

Prior to analysis, soil samples were removed from the freezer and allowed to thaw at room temperature. Once thawed, samples were collected for moisture content and bulk density analysis, and the remaining soil was homogenized and sieved through a 4 mm sieve, and air-dried for a minimum of 48 hours. Soils were then used to determine selected physical and chemical properties, and herbicide bioassay experiments as described below.

3.3.2.3 Soil Physical and Chemical Properties.

Soil texture was determined by particle size analysis using the pipet method as outlined in Gee and Bauder (1986).

Bulk densities were derived to a depth of 10 cm. Three, 100 g sub samples from each plot were analyzed gravimetrically for water content in order to calculate the oven-dried weight of the soil.

Soil organic carbon contents were determined to a depth of 10 cm for all soils. A Leco CHN CNS 2000 Elemental Analyzer model was used to determine the soil organic carbon by dry combusting 0.2 g of oven-dried soil previously treated with 6N hydrochloric acid and deionized water, as described in Tiessen et al. (1983).

The pH measurements were determined using 50 g of soil in a 1:1 soil-water suspension that was stirred and allowed to stand for 15 minutes. The samples were stirred again after 15 minutes, and readings were taken using an Accumet 925 pH/ion meter with a glass electrode.

Field capacity of each soil was derived by determining the percentage of water (mass basis) in the soil by measuring the wet weight and oven-dried weight of the soil. The calculated amount of water needed to attain field capacity was then added to specific volumes of dry soil in PVC pots and observed for water loss. Experiments involving soils which had water loss were repeated using less water until no more water loss was observed. This was determined the field capacity of the soil. Additional amounts of water were added to soils showing no initial water loss until a visible amount of staining was noted on the bottom of the soil, thus deriving the field capacity. Multiple replicates for each soil were used with more or less water being added as needed to each pot to accurately establish field capacity.

3.3.3 Herbicide Bioassays.

Numbered PVC pots were filled with sieved and air-dried, clean and treated soil samples to a depth of 10 cm. Five pots of each treatment soil and clean soil were prepared for each sampling day. Each pot was seeded with 10 oat seeds (*Avena sativa*), pregerminated in an incubator at 25°C for 24 hours. Oats was chosen as the indicator species as it is highly sensitive to imazethapyr. The sample pots were watered to field capacity, and then placed in growth chambers in an order derived from a random numbers chart. The growth chambers were set to maintain a temperature of 25°C +/- 2°C, a relative humidity of approximately 90% and a 12-hour light/dark cycle. The sample pots were watered every day to maintain field capacity, and remained in the controlled environment chambers for two weeks. After the two-week period, the number of shoots and the fresh-weight biomass was measured for all soil cores. The residual activity of imazethapyr remaining in the treated soils was determined by comparing the fresh weight of oat shoots to those grown in clean soil from the same field site, and the percent herbicide bioactivity (%HB) was calculated by:

$$\text{(1)} \qquad \%HB = 100 - \{(W_h / W_c)*100\}$$

where Wh = fresh weight of plant shoots grown in a herbicide treated soil, and Wc = fresh weight of plant shoots grown in a control soil.

3.3.4 Statistical Analysis of Data

The effect of tillage, texture and landscape on imazethapyr persistence in soil was assessed by ANOVA (Analysis of variance) using SigmaStat® software from the SPSS Institute. ANOVA tests were performed for each sampling day, and for each herbicide product. Graphical illustrations using mean values obtained from each plot were used to separately demonstrate the differences in imazethapyr persistence over time, and between herbicide concentrations. Errors bars indicate the standard deviation from the mean for each treatment soil.

3.4 RESULTS

3.4.1 Variations in imazethapyr persistence under different tillage systems

3.4.1.1 Fairland. Odyssey® applied to Fairland clay soil under reduced tillage maintained greater soil activity than in Fairland clay loam soil under conventional tillage after day 4 (Figure 3.2). As expected, the herbicide was most effective following application where the %HB was 73.2% in the conventional tillage soil, and 66.8% in the reduced tillage soil. However, over time, Odyssey® persisted longer under reduced tillage maintaining greater than 50% herbicide bioactivity up to 64 days after application.

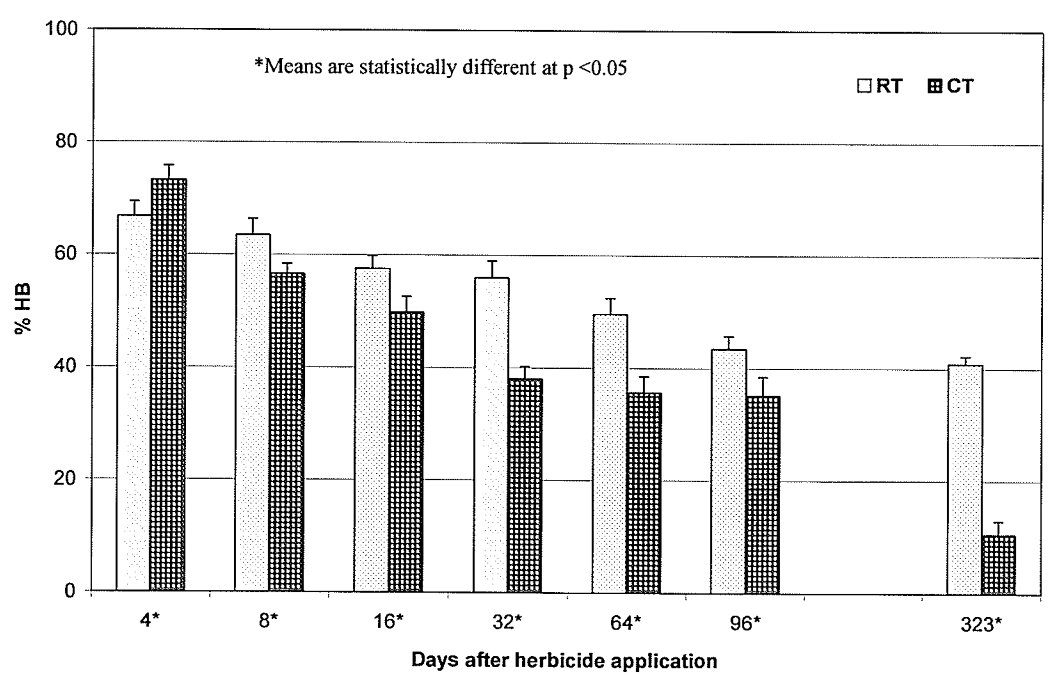

Figure 3.2 Herbicide bioactivity of Odyssey® applied to Fairland reduced tillage clay soil and conventional tillage clay loam soil.

The %HB in the conventional tillage soil dropped below 50% by day 16. The differences in herbicide bioactivity for the FRT soil versus the FCT soil were statistically significant at $p < 0.05$ for sampling days 4, 8, 16, 32, 64, and 323, although there was a greater variability in the results of the FCT soil for each sampling day. The %HB for the FRT soil on days 96 and 323 were quite similar, 42.1% and 39.5%, respectively, indicating no significant degradation during the winter. In contrast, there was a noticeable difference in %HB for days 96 and 323 in the FCT soil, 30.8% and 17.7%, respectively, which may be a result of spring tillage. Imazethapyr was still prominently active in soil, especially in the reduced tillage plot, one year following application where the %HB was still greater than 40%.

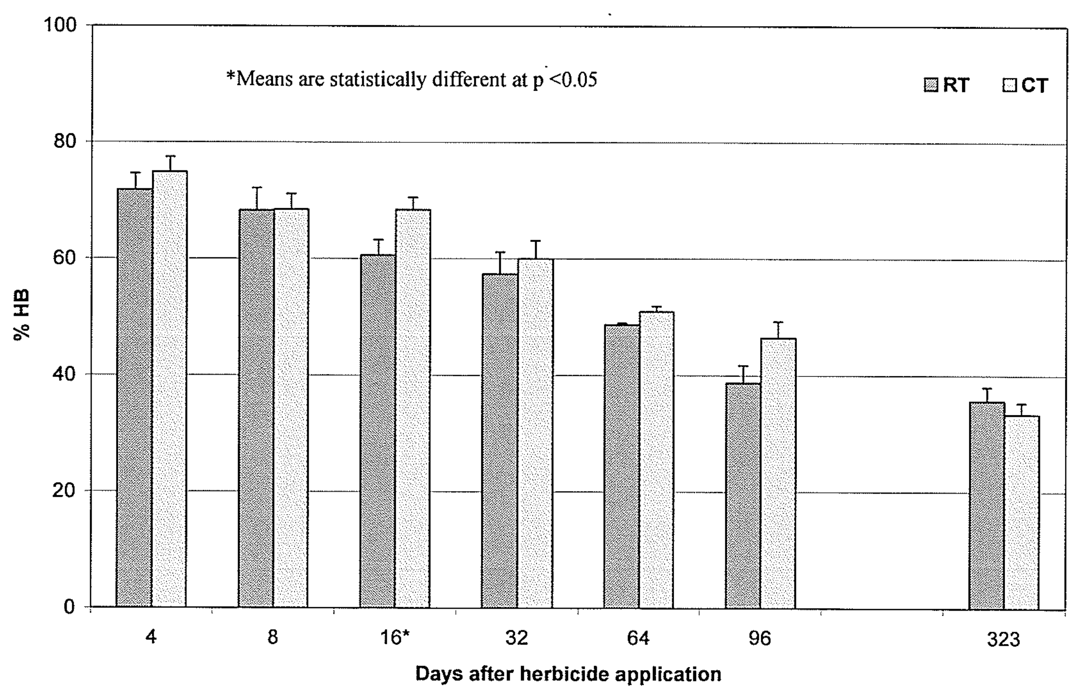

Figure 3.3 Herbicide bioactivity of Pursuit® applied to Fairland reduced tillage clay soil and conventional tillage clay loam soil.

The residual activity of Pursuit® in both the Fairland clay loam conventional and clay reduced tilled soils was relatively similar throughout the sampling period (Figure 3.3). Initially following application, the herbicide was very effective in both the FRT and FCT soils, with a %HB >70%. The bioactivity of Pursuit® throughout the growing season in FCT soil and FRT soil was statistically different for only day 16 ($p = 0.01$). Between day 96 and 323, the %HB decreased by only 13.2% and 3.1% in the FCT and FRT soils, respectively, indicating very little dissipation or degradation of imazethapyr during the winter.

The results for the Fairland soils sprayed with Pursuit® differed significantly from those sprayed with Odyssey®. Pursuit® maintained greater bioactivity in the conventional tillage soils than Odyssey® as the %HB remained above 50%, 64 days following Pursuit® application in comparison to 36% for plots sprayed with Odyssey®. The largest discrepancy between the two herbicides occurred one year following application where the %HB were 33.3% and 10.5% for Pursuit® and Odyssey®, respectively, indicating Pursuit® maintained greater residual activity in the soil for a greater period of time. In the reduced tillage plots, Pursuit® was a more effective herbicide than Odyssey® immediately following application. However, by day 32, Pursuit® efficacy had decreased by a greater percentage than Odyssey®.

In summary, Odyssey® maintained greater soil activity in the FRT soils than the FCT soil. The results indicated that Pursuit® showed no partiality for either tillage system, though efficacy in both soils was greater than that of Odyssey®.

3.4.1.2 Hallboro. The results from the coarse-textured Hallboro sandy loam soil bioassays indicated only slight differences in persistence of imazethapyr between conventional and reduced tillage soils (Figure 3.4). The difference in herbicide bioactivity was statistically significant (p = 0.03) immediately following Odyssey® application indicating greater soil activity in the conventional tillage soil. However, by day 8, the results from the reduced tillage soil had a %HB value approximately 20% greater than that of the conventional tillage soil. Herbicide activity decreased dramatically between day 4 and day 16 where the %HB was 66.7% and 38.5% in the HRT soil, and 71.7% and 36.3% in the HCT soil, respectively.

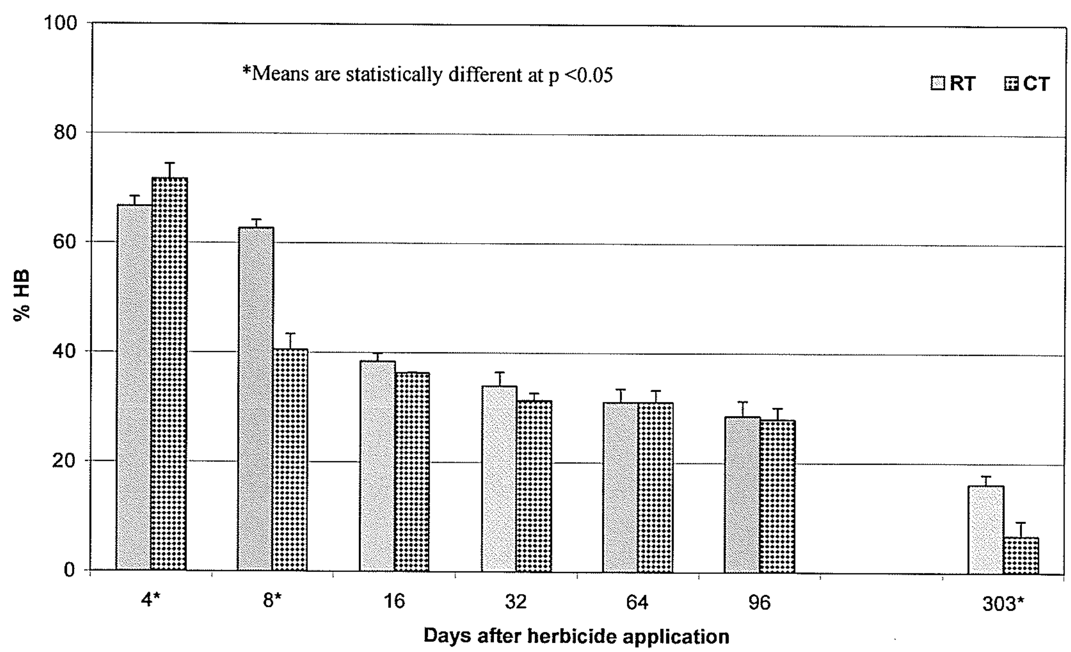

Figure 3.4 Herbicide bioactivity of Odyssey® applied to Hallboro reduced tillage and conventional tillage sandy loam soils.

The results from soil samples collected after day 16 indicate the soil activity remained relatively constant from day 16 to 96 following application (Figure 3.4). There was a noticeable difference in %HB for day 96 and 303 in both the HRT and HCT soils, indicating a decrease in herbicide persistence during the winter.

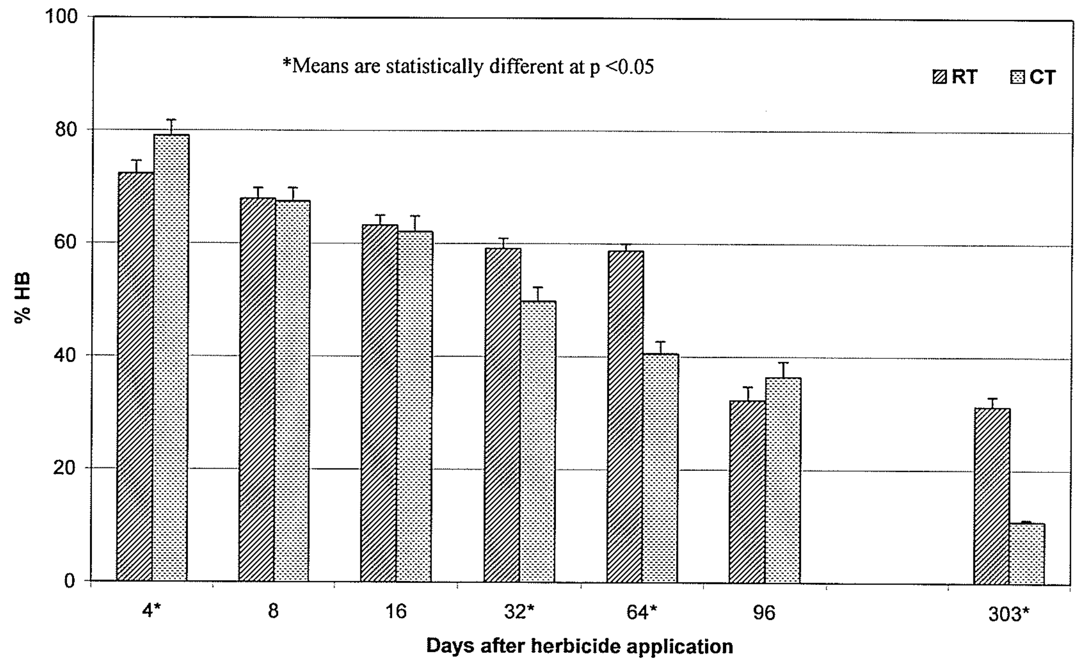

Figure 3.5 Herbicide bioactivity of Pursuit® applied to Hallboro reduced tillage and conventional tillage sandy loam soils.

Herbicide activity in plots sprayed with Pursuit® was initially greater in the conventional tillage soil than the reduced tillage soil (Figure 3.5). The %HB differed significantly on day 4 ($p = 0.02$), but was similar on day 8 ($p = 0.81$) and 16 ($p = 0.57$). The %HB in the HCT soil declined noticeably between day 16 and 32, after which the rate of decrease accelerated until the last sampling day.

In contrast, the %HB in the HRT soil remained relatively stable in the first two months after application, decreasing by less than 15%. The %HB for the HRT soil was significantly higher than the HCT soil for days 32 and 64 ($p < 0.05$). By day 96, the mean values were comparable due to a 26% reduction in herbicidal activity in the HRT soil. Over the winter, there was a noticeable reduction in Pursuit® activity in the HCT soil, but not in the HRT soil, likely attributable to fall or spring tillage in the conventional tillage plots.

In the conventional tillage soil, Pursuit® soil activity was much greater than Odyssey® for all sampling days. Odyssey® persistence in the soil declined considerably between sampling day 4 and 8 (Figure 3.4), while the results showed Pursuit® efficacy decreased gradually over time (Figure 3.5). Both herbicides showed susceptibility to degradation and dissipation during the winter as indicated by the decreases in %HB between day 96 and 303.

When applied at recommended field rates to the reduced tillage plots, Pursuit® persisted longer in the soil than Odyssey®. The most noticeable differences in herbicidal activity for the two herbicides were observed between day 16 and 64 post-application. While Pursuit® soil activity declined gradually up to day 96, the results from plots sprayed with Odyssey® indicated a major decline in soil activity between sampling day 8 and 16. Odyssey® results showed considerable degradation and dissipation during the winter, while the %HB for Pursuit® remained relatively stable.

Overall, Pursuit® provided more prolonged and effective herbicidal activity than Odyssey® in both the conventional and reduced tillage Hallboro sandy loam soils. Though Pursuit® showed better activity in the RT soil, Odyssey® results indicated no advantage for either tillage system over time.

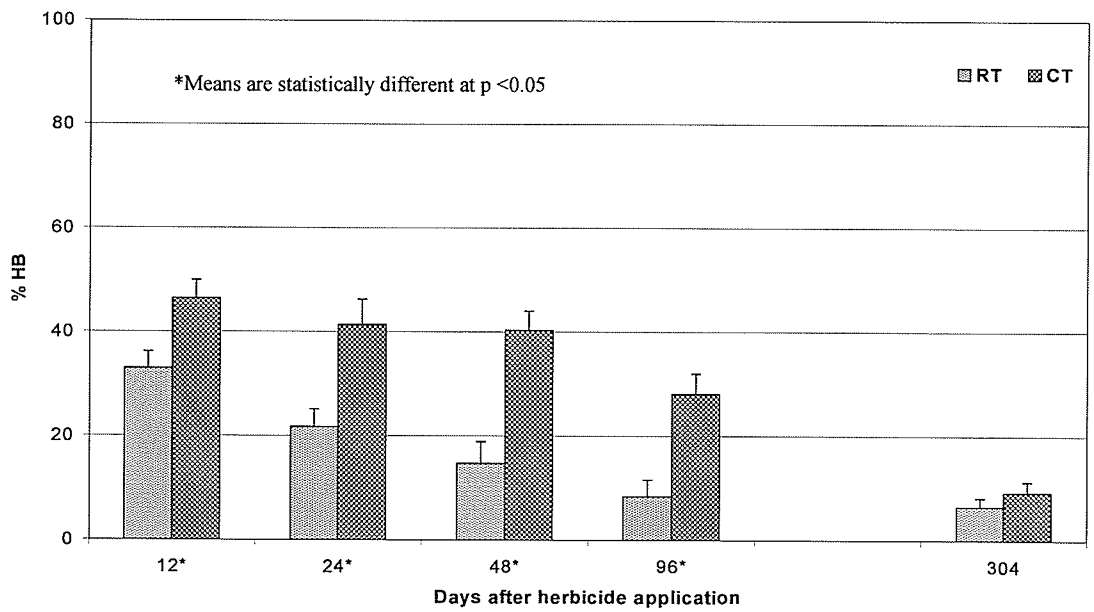

Figure 3.6 Herbicide bioactivity of Odyssey® applied to Newdale reduced tillage and conventional tillage clay loam soils.

3.4.1.3 Newdale. The differences in herbicide bioactivity in the Newdale clay loam soils indicate that Odyssey® applied to soil under conventional tillage maintained greater soil activity than soil under reduced tillage (Figure 3.6). As expected, the herbicide was most effective directly following application and decreased gradually over time. Odyssey® persisted longer under conventional tillage than under reduced tillage maintaining a %HB between 40% and 50% up to sampling day 48. In contrast, the %HB for the reduced tillage soil was below 40% by day 12, and below 20% by day 48. The differences in herbicide bioactivity for NRT and NCT soils were statistically significant at $p < 0.05$ for sampling days 12, 24, 48, and 96.

The %HB for the NRT soil between day 96 and 304 remained relatively stable, 8.4% and 6.5%, respectively. In contrast, there was a clear difference in %HB for days 96 and 304 in the NCT plots, 28.2% and 9.2%, respectively, indicating a noticeable reduction in Odyssey® persistence possibly due to tillage activities. Soil activity of Odyssey® one year following application was very low in both the conventional or reduced tillage soil.

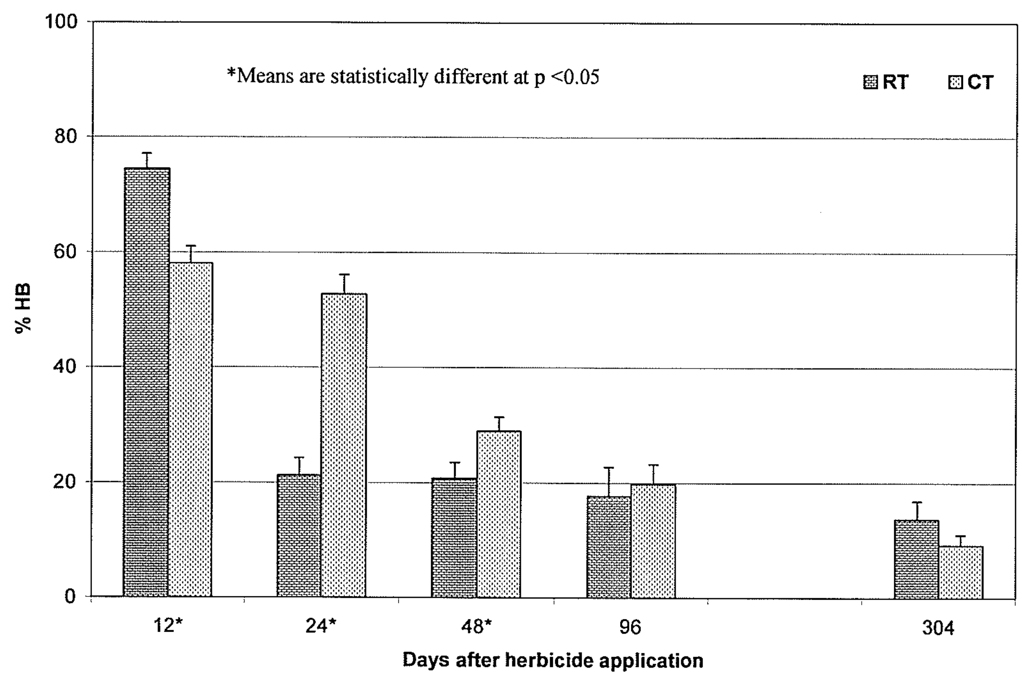

Figure 3.7 Herbicide bioactivity of Pursuit® applied to Newdale reduced tillage and conventional tillage clay loam soils.

The results for the Newdale clay loam bioassays indicated considerable differences in soil activity of Pursuit initially following application (Figure 3.7). Twelve days post-application, the soil activity in the reduced tillage soil was significantly greater ($p<0.05$), where the %HB declined by approximately 74.5% in comparison to 58.1% in the conventional tillage soil. However, by day 24, herbicidal bioactivity in the NRT soil decreased to 21.3% while the %HB in the NCT soil remained relatively stable at 52.8% before decreasing to 29.0% by day 48. The %HB for the NRT and NCT soils was significantly different at $p < 0.05$ for sampling days 12, 24, and 48. Over the winter, there was no major decrease in Pursuit® activity in either the conventional or reduced tilled soils.

Pursuit® was a more effective herbicide than Odyssey® when applied to the reduced tillage Newdale clay loam soil. Herbicidal bioactivity was approximately 75% for Pursuit in comparison to 33% for Odyssey® at day 12. Between sampling day 24 and 96, both herbicides were equally effective, and neither herbicide showed significant bioactivity one year following application. In the conventional tillage Newdale clay loam soil, Pursuit® showed greater herbicidal activity than Odyssey® initially following application. Pursuit® activity was stronger than Odyssey® on days 12 and 24, but by day 48, Odyssey® was a more effective herbicide than Pursuit®. There was a noticeable reduction in persistence for both herbicides following the winter months, with Odyssey® showing a greater susceptibility to degradation and dissipation.

Overall, Pursuit® provided more effective soil herbicidal control following application than Odyssey® in both the conventional and reduced tillage Newdale clay loam soils. Persistence of both herbicides was greater in the conventional tillage soil than the reduced tillage soil.

3.4.1.4 Varcoe. Odyssey® was not a very effective herbicide in either the conventional or reduced tillage Varcoe clay loam soils (Figure 3.8). The results for the first sampling day following application indicate that the herbicide bioactivity was approximately 45% in both soils. Further declines in activity were detected in the VRT soil between day 12 and 24, and in the VCT soil between day 24 and 48, where the %HB decreased by 18.5% and 18.9%, respectively. Odyssey® activity was significantly greater in the VCT soil than the VRT soil on day 24 ($p = 0.01$), and significantly greater in the VRT soil than the VCT soil on day 48 ($p = 0.02$). The %HB for the VCT soil was lower than for the VRT soil for days 96 and 304, but the differences were not significant due to the high variability of the results for these sampling dates.

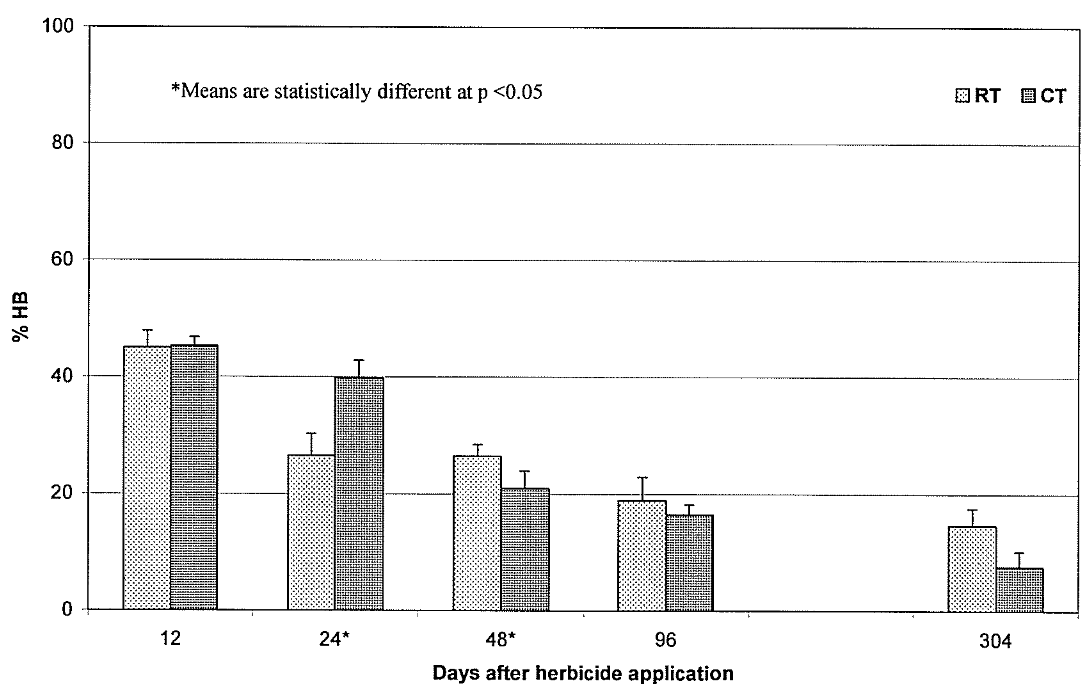

Figure 3.8 Herbicide bioactivity of Odyssey® applied to Varcoe reduced tillage and conventional tillage clay loam soils.

Pursuit® applied to Varcoe clay loam soils under conventional tillage maintained greater soil activity initially following application as indicated by the %HB for sampling day 12 and 24 (Figure 3.9). The difference in herbicide activity for each soil was statistically significant for day 24 ($p = 0.03$), but not day 12 ($p = 0.34$). By day 48, Pursuit® activity in the VCT soil declined considerably with the %HB decreasing from 52.9% to 21.5%, which was significantly different from the %HB in the VRT soil ($p < 0.05$). The %HB for the VCT soil on days 96 and 304 were quite similar, 12.9% and 12.5%, respectively, indicating no significant degradation or dissipation of the herbicide during the winter. In comparison, there was a noticeable difference in the herbicide activity for these same days for the VRT soil, 25.8% and 8.9%, respectively.

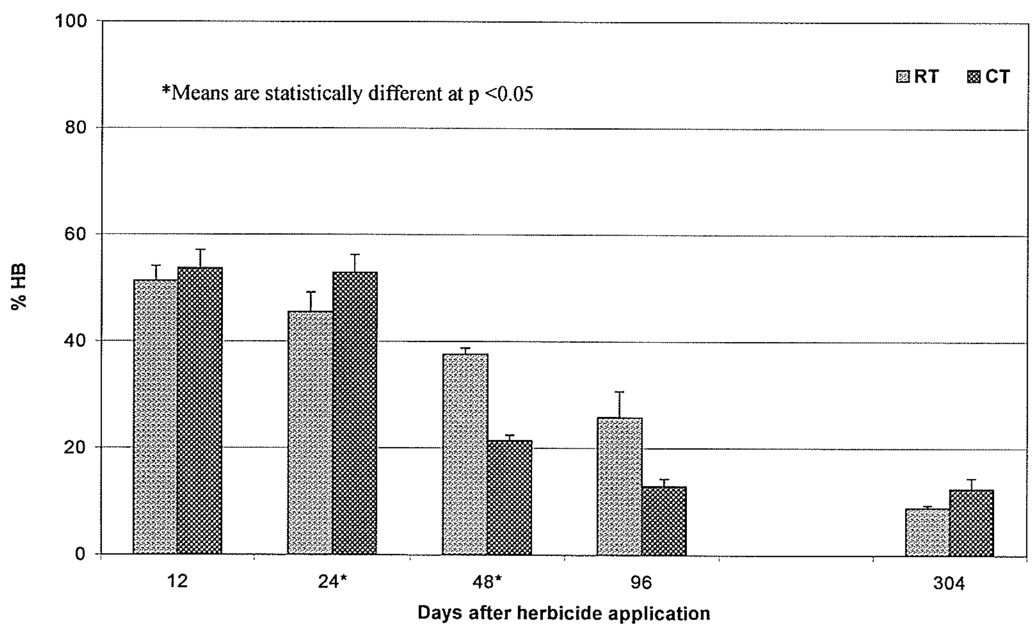

Figure 3.9 Herbicide bioactivity of Pursuit® applied to Varcoe reduced tillage and conventional tillage clay loam soils.

Pursuit® was more persistent in the reduced tillage soil than Odyssey®, but herbicide activity for both products only attained a maximum of 52%. Pursuit activity declined gradually over the sampling period in comparison to Odyssey®, which decreased rapidly between day 12 and 24, then steadily dissipated over the remainder of the sampling days. Pursuit® showed greater susceptibility to degradation during the winter than Odyssey®. In the conventional tillage soil, Pursuit® showed much greater activity than Odyssey® immediately following application on days 12 and 24. The bioactivity of each herbicide did not differ substantially following sampling day 24. One year following application, neither Pursuit® nor Odyssey® had maintained effective herbicidal capabilities in the conventional-till soil.

In general, Odyssey® and Pursuit® persisted longer by maintaining greater soil activity in the VRT soil than the VCT soil. The results from plots sprayed with Pursuit® showed greater activity than Odyssey® in both soils.

3.4.2 Influence of landscape position on Odyssey® and Pursuit® persistence in soil

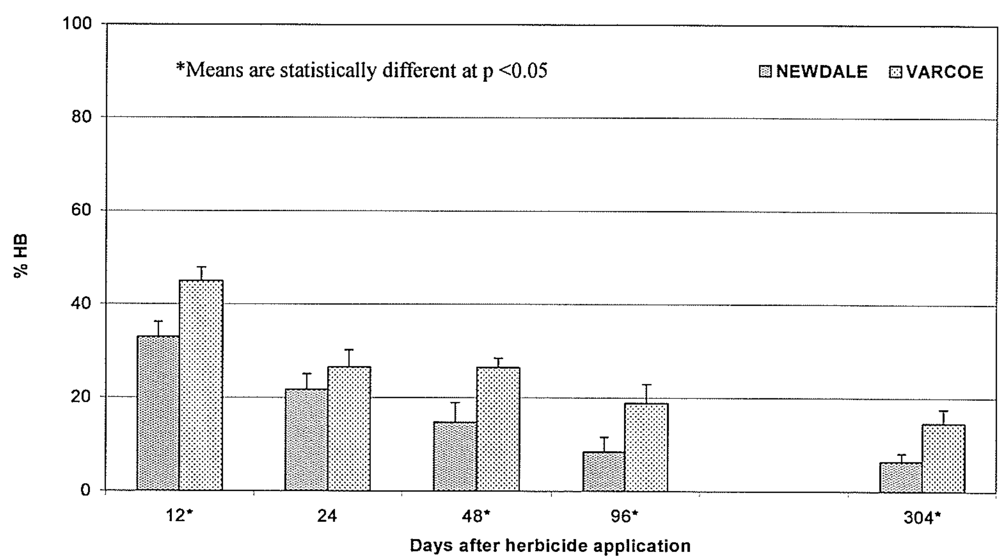

Figure 3.10 Herbicide bioactivity of Odyssey® applied to upper slope (Newdale) and lower slope (Varcoe) soils under reduced tillage.

In year 2, the experiments in the Newdale clay loam soil (upper slope) and the Varcoe clay loam soil (lower slope) were done in the same field, as such; the influence of landscape position on the persistence of Pursuit® and Odyssey® was also examined.

Odyssey® applied to soil in the lower slope under reduced tillage maintained greater soil activity than in the upper slope (Figure 3.10). The herbicide was most effective initially following application where the herbicide bioactivity was 45.0% in the lower slope and 33.0% in the upper slope. The differences in %HB for the upper slope and the lower slope were statistically significant at $p < 0.05$ for all sampling days except day 24, due to the high variability of the results for this day. Soil activity of imazethapyr one year following application was very low in both slope positions.

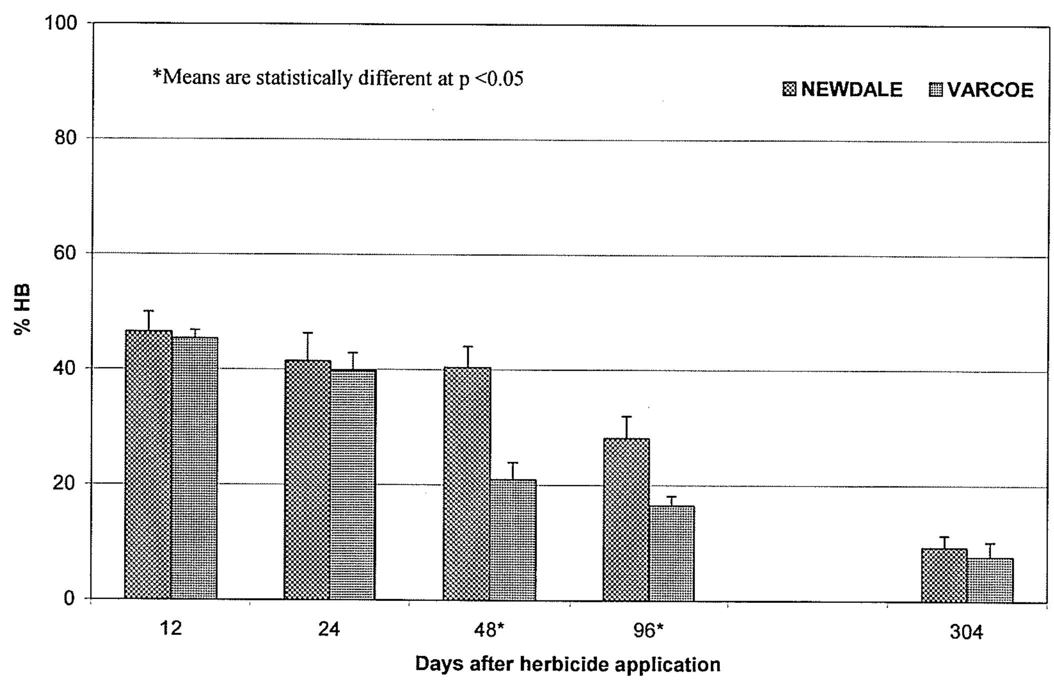

Figure 3.11 Herbicide bioactivity of Odyssey® applied to upper slope (Newdale) and lower slope (Varcoe) soils under conventional tillage.

The residual activity of Odyssey® in both the upper and lower slope under conventional tillage was comparable immediately following application, with no significant differences in herbicide activity observed on day 12 and 24 (Figure 3.11). However, the %HB in the upper slope was approximately 20% greater than in the lower slope ($p < 0.05$) by day 48, and approximately 10% greater on day 96 ($p = 0.04$). There was a noticeable reduction in persistence at both slope positions over the winter months, though Odyssey showed greater susceptibility to degradation and dissipation in the upper slope.

Herbicide bioactivity in plots sprayed with Pursuit® under reduced tillage was initially greater in the upper slope than the lower slope position (Figure 3.12). The difference in %HB was statistically significant ($p < 0.05$) 12-days following herbicide application. However, between sampling day 24 and 96, the %HB in the lower slope was greater than in the upper slope, but the differences were only significant on day 24 and 48 ($p < 0.05$).

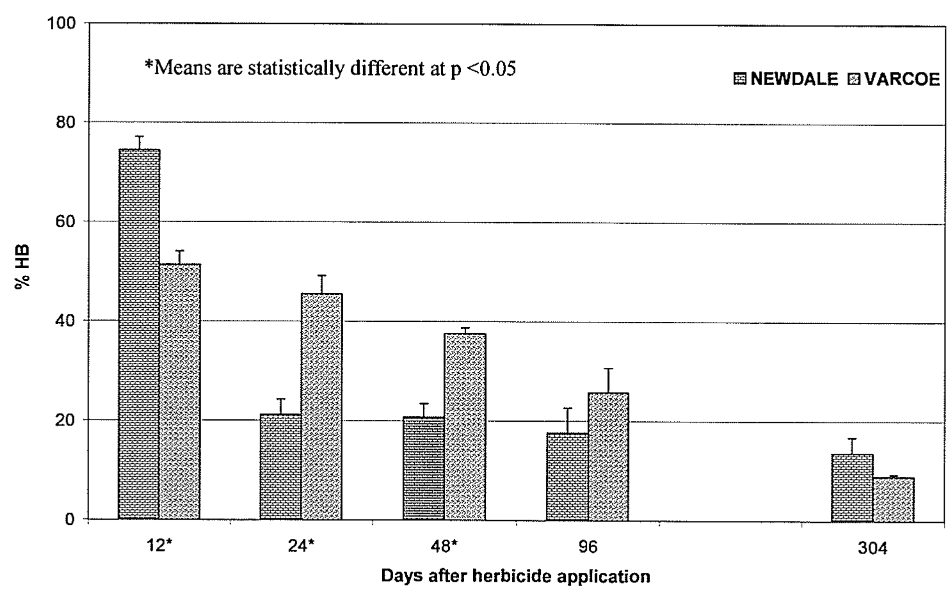

Figure 3.12 Herbicide bioactivity of Pursuit® applied to upper slope (Newdale) and lower slope (Varcoe) soils under reduced tillage.

The %HB for the upper slope on days 96 and 304 were quite similar, 17.7% and 13.7%, respectively, indicating no significant degradation during the winter. In contrast, there was a noticeable difference in %HB for days 96 and 304 in the lower slope, 25.8% and 8.9%, respectively, indicating greater herbicide dissipation in the lower slope position over the winter months.

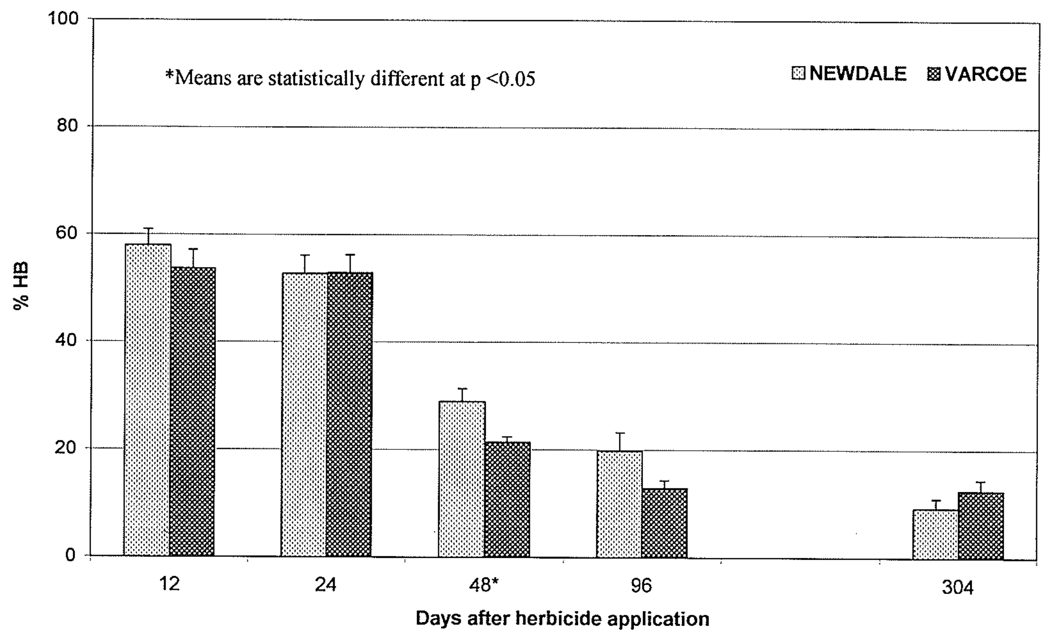

Figure 3.13 Herbicide bioactivity of Pursuit® applied to upper slope (Newdale) and lower slope (Varcoe) soils under conventional tillage.

The residual activity of Pursuit® in both the upper and lower slope soils under conventional tillage was comparable immediately following application (Figure 3.13). For sampling days 12 and 24, the herbicide was equally effective in both the upper and lower slope positions, with a %HB >50%. There was a substantial decrease in herbicide bioactivity between day 24 and 48 in both slope positions. For day 48 and 96, the %HB in the upper slope was greater than in the lower slope, but the difference was only significant on day 48 ($p = 0.02$). Over the winter, there was a noticeable reduction in Pursuit® activity in the upper slope soil, but not in the lower slope soil.

Overall, the %HB was generally greater for Pursuit® than Odyssey® in the various treatment plots, and herbicide bioactivity over the growing season was less persistent on the upper slope RT and lower slope CT than for the upper slope CT and lower slope RT.

3.5 DISCUSSION

The results of the field study experiments provided evidence that imazethapyr maintains residual herbicide activity in soil following application. These data confirm previous work that imazethapyr has the potential to control multiple weed flushes during the growing season and to injure subsequent crops in rotation (Curran et al. 1992; Stougaard et al. 1990; Vencill et al. 1990). The potential and extent of crop injury is dependent on the residual activity of the herbicide, which is mainly influenced by physical-chemical properties of the soil. Selected soil properties of the treatment soils are listed in Table 3.1 and Table 3.2.

Herbicide bioactivity was consistently greater for Pursuit® than Odyssey®. As such, the type of herbicide product was the strongest factor in explaining the variation of %HB among the treatments. The type of tillage practice did not significantly influence herbicide persistence for any of the treatment soils with the exception of Odyssey® applied to the Fairland reduced tillage clay soil and the Newdale conventional tillage clay loam soil.

Table 3.1 Soil properties for the Fairland and Hallboro soils under reduced tillage (RT) and conventional tillage (CT).

Site	Texture	Clay Fraction	Organic Carbon	pH
		%		
Fairland CT	Clay Loam	27.3±0.8	3.05	5.89±0.09
Fairland RT	Clay	40.7±0.4	3.27	6.45±0.17
Hallboro CT	Sandy Loam	14.2±0.7	1.13	6.36±0.13
Hallboro RT	Sandy Loam	10.0±0.6	0.68	6.98±0.11

However, the increased organic carbon content in the Fairland reduced tillage clay soil (3.27%) and Newdale conventional tillage clay loam soil (3.62%) could account for the prolonged persistence of the herbicide in comparison to the Fairland conventional tillage clay loam soil (3.05%) and the Newdale reduced tillage clay loam soil (2.87%), respectively. Other studies have also found that imazethapyr persistence increases with increasing soil organic carbon content (Loux and Reese 1993).

In the 1999 field study, imazethapyr persistence was greater in the Fairland clay and clay loam soils than the Hallboro sandy loam soils regardless of tillage system. The soil organic carbon content was greater for the FRT (3.27%) and FCT (3.05%) than HCT (1.13%) and HRT (0.68%). The soil texture, particularly the clay fraction, of the Fairland clay and clay loam soils also differed from the Hallboro sandy loam soils (Table 3.1).

Table 3.2 Soil properties for the Newdale and Varcoe soils under reduced tillage (RT) and conventional tillage (CT).

Site	Texture	Clay Fraction	Organic Carbon	pH
		%		
Newdale CT	Clay Loam	27.1±0.4	3.62	7.74±0.11
Newdale RT	Clay Loam	27.2±0.7	2.87	7.51±0.22
Varcoe CT	Clay Loam	27.2±0.8	3.19	7.60±0.13
Varcoe RT	Clay Loam	29.1±0.5	4.23	7.42±0.18

The clay content of the FRT (41%) and FCT (27%) was much greater than HCT (14%) and HRT (10%). As such, there was a relation between herbicide dissipation and the amount of organic carbon and clay fraction when comparing the soil series. Other studies have shown that imazethapyr persistence is more predominant in fine-textured soils, particularly those with increased clay fraction (Stougaard et al. 1990), as well as soils with higher organic carbon contents (Curran et al. 1991; Loux and Reese 1993). Soil pH may also affect imazethapyr persistence, particularly at pH levels below 6 (Loux et al. 1989b; Renner et al. 1988), where the herbicide dissipation rate is slower due to increased sorption (Cantwell et al. 1989). However, there was no apparent relationship between imazethapyr persistence and soil pH in the 1999 field study. The discrepancy in the effect of soil pH on imazethapyr persistence among these various soil types may be due, in part, to differences in clay and organic matter contents, and the effect of these differences on sorption, and its subsequent effect on herbicide dissipation (Loux and Reese 1993).

Herbicide bioactivity of imazethapyr in the Newdale and Varcoe clay loam soils (2000) following application was significantly lower than in the 1999 field study. This difference may be due, in part, to frequent rainfall events before and shortly after herbicide application. The soil moisture contents 12 days following application in the 2000 field experiment ranged from 41.4% NCT to 48.7% VCT (Figure 3.14), approaching field capacity. Other studies have found that significant rainfall following application greatly reduced imazethapyr persistence, as the increased soil moisture resulted in increased microbial degradation (Goetz et al. 1990).

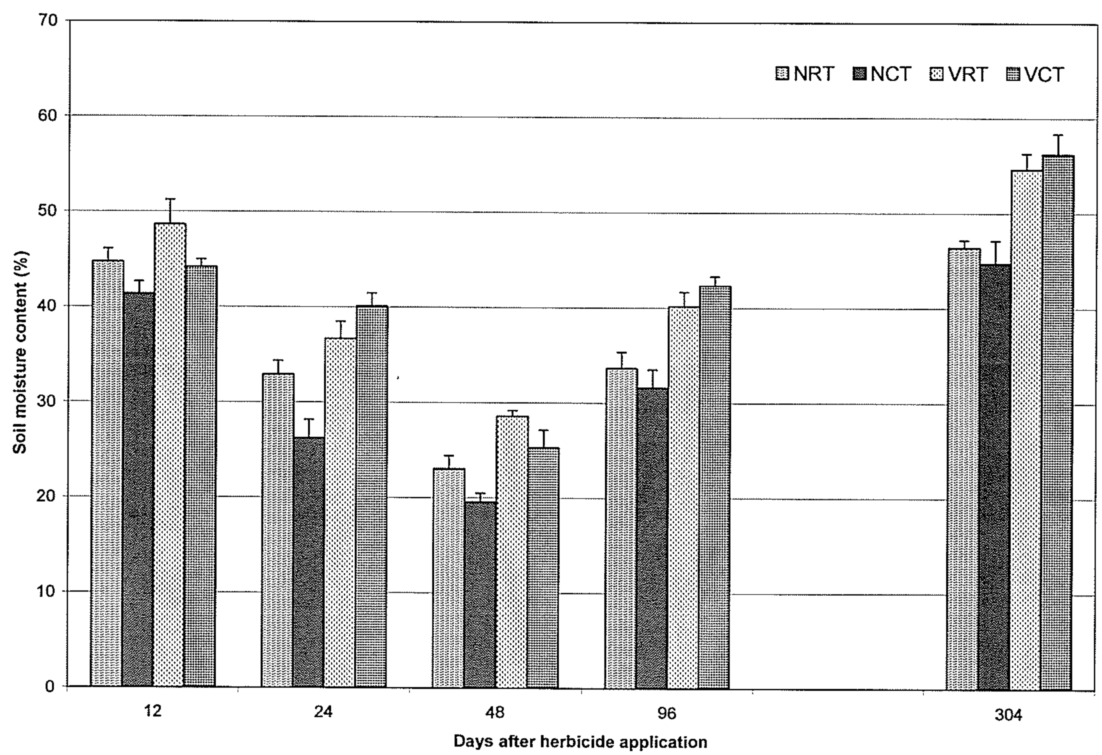

Figure 3.14 Soil moisture contents (%) for the Newdale reduced tillage (NRT), Newdale conventional tillage (NCT), Varcoe reduced tillage (VRT), and Varcoe conventional tillage (VCT) clay loam soils.

Biodegradation could have been an important herbicide dissipation process in the study. In laboratory incubations at 25°C, imazamox half-lives ranged from only 11 to 19 days when applied at recommended field rates to soils with moisture contents of 75% field capacity (Vischette et al. 2002). The optimum soil water content for imazethapyr biodegradation is also 75% of field capacity (Flint and Witt 1997). Leaching and surface runoff are two other processes that may have contributed to the decline in herbicide activity, particularly during the 2000 field study when rainfall events were above normal. Although imazethapyr is generally not mobile in soils (Mangels 2000), laboratory experiments using repeated 12.5 mm/h rainfall simulations detected imazethapyr residues at 30 cm depths (Jourdan et al. 1998). Also, herbicide loss by runoff is greatest when rainfall occurs shortly after herbicide application (Locke and Bryson 1997). In addition, imidazolinones were applied to the soil surface and volatilization and photodegradation processes may have contributed to losses in herbicide activity over time (Curran et al. 1992), but studies have suggested that imazethapyr volatilization and photodegradation losses are minimal (Goetz et al.1990).

The soil organic carbon content of the soils used in the 2000 field study varied from 2.87% and 4.23% and increased in the order of NRT < VCT < NCT < VRT (Table 3.2). As such, there was no obvious influence of slope position on the organic carbon content in soil. The treatment soils were similar in clay fraction and pH.

Herbicide bioactivity over the growing season was less persistent for the Newdale reduced tillage clay loam soil and the Varcoe conventional tillage clay loam soil than the Newdale conventional tillage and Varcoe reduced tillage clay loam soils. Soil organic carbon content was also smaller for NRT (2.87%) and VCT (3.19%) than for NCT (3.62%) and VRT (4.23%). The results indicate there was a relation between herbicide dissipation and the amount of organic carbon in the soil, similar to that in the 1999 field experiment, as well as other studies (Curran et al. 1991; Loux and Reese 1993), where increases in organic carbon content resulted in increased imazethapyr persistence.

3.6 CONCLUSION

The 1999 field study examined herbicide bioactivity of two imidazolinone-containing herbicide products applied to reduced and conventional tillage soils situated in a near-leveled terrain, while the 2000 field study examined imazethapyr bioactivity in upper – and lower slope position soils under reduced and conventional tillage. There were no obvious effects of either tillage or landscape position on soil properties that could influence imazethapyr persistence, particularly organic carbon, texture, and pH. As such, more pronounced relationships were observed between imazethapyr bioactivity and individual soil characteristics than with any particular treatment soil. Oat bioassays indicated that herbicide bioactivity (%HB) was most strongly influenced by herbicide product, organic carbon content, and clay fraction in the 1999 field study, and herbicide product, and organic carbon content in the 2000 field study. The %HB results from these experiments concur with those from other studies, which ascertained that imazethapyr persistence occurs more likely in medium to fine-textured soils with medium to high organic carbon contents than in coarse-textured soils with low organic carbon contents (Curran et al. 1991; Goetz et al. 1990; Loux and Reese 1993). Based on the bioassay results, the potential for crop injury by herbicide carry-over was limited, and there was no consistency in the effects of herbicide product, tillage system, and slope position on %HB in soil one year following herbicide application.

4. SORPTION OF IMAZETHAPYR ON CULTIVATED SOILS

4.1 Abstract

The sorption of imazethapyr by agricultural soils in western Manitoba was studied using a batch equilibrium method. The Freundlich constant (Kf) was determined for four series (Fairland clay and Fairland clay loam, Hallboro sandy loam, Newdale clay loam, and Varcoe clay loam) collected from fields under conventional and reduced tillage. For all soil series, sorption was greater in soils under conventional tillage than reduced tillage for all series. Kf values were also greater in the lower landscape positions (Varcoe clay loam) than upper slope positions (Newdale clay loam) in both conventional and reduced tillage fields. When using all data, no correlation was observed between Kf and either soil pH, organic carbon, or clay fraction. This was attributed to the limited number of acidic soils used in the study.

4.2 Introduction

Imazethapyr is the active ingredient in the herbicides Pursuit®, Odyssey®, and Absolute®. These herbicide products are capable of controlling multiple weed flushes during the growing season. Imazethapyr has been shown to persist in Canadian Prairie soils for more than one year, thus posing a threat to sensitive crops like wheat, barley, and canola when these crops are subsequently planted in rotation (Curran et al. 1992; Loux and Reese 1993; Stougaard et al. 1990).

Imazethapyr persistence and bioavailability to plants and microorganisms is influenced by the extent of imazethapyr sorption onto soil (Cantwell et al.1989; Loux and Reese 1993). Imazethapyr sorption increases with decreasing soil pH, particularly at pH levels below 6 (Loux et al. 1989a). Soil organic matter and clay surfaces have pH dependent charges, which play a significant role in herbicide sorption (Stevenson 1972). Previous studies concluded that organic matter played a greater role in imazethapyr sorption than soil clay particles (Che et al. 1992; Gennari et al. 1998; Goetz et al. 1986). The contribution of clays to sorption is largely influenced by the presence of iron and aluminum oxides that are capable of bonding with anionic herbicides at low pH levels (Gennari et al. 1998; Pusino et al. 1997). Consequently, soil organic matter and pH are the soil properties most commonly correlated with herbicide sorption.

Ionizable herbicide sorption occurs to the greatest extent nearest the chemicals' dissociation constants (pKa) (Best et al. 1974; Kells et al. 1980). Imazethapyr is an amphoteric molecule possessing both an acidic carboxyl, and basic imidazolinone and pyridine functional groups. The 3.9 acidic pK_a of the ionizable carboxyl group establishes that the imazethapyr molecule be in a primarily anionic state at near-neutral soil pH. As soil pH increases, the ionic character of the clay, organic matter, and Al/Fe - oxide surfaces become increasingly anionic, thus decreasing the capacity of imazethapyr to sorb to soil particles (Renner et al. 1988b). Alternatively, as soil pH decreases, protonation of the basic N – atoms (pK_a 2.1) results in a positive or neutral molecule capable of binding to soil colloids, and increasing sorption (Stougaard et al. 1990). Loux et al. (1989a) observed that sorption of imazethapyr by soil increased as soil pH decreased. In contrast, Gan et al. (1994) observed that pH did not greatly affect imazethapyr sorption in the pH range of 4.8 and 7.1.

Soil moisture content also influences sorption as herbicide and water molecules compete for sorption sites in ion-dipole reactions, or for ligand positions on the soil surfaces (Calvert 1980; Theung 1979). At low soil moisture levels, there is increased dissociation of water at the colloid surface, therefore a decreased competition with herbicides for binding sites (Goetz et al. 1986) allowing for increases in herbicide sorption by soil. Thus, the amount of moisture in soil can significantly affect the magnitude of imazethapyr sorption.

The type of cropping system, climatic conditions, and topography are factors that determine soil physical, chemical, and biological properties. Consequently, the potential for herbicide sorption can vary significantly within a field or region.

The objective of this study was to determine the impact of soil constituents on imazethapyr sorption under conventional and reduced tillage in varying soil landscapes.

4.3 MATERIALS AND METHODS

4.3.1 Site Description

The soils used in this experiment were collected from Fairland clay and clay loam, Hallboro sandy loam, Newdale clay loam, and Varcoe clay loam soils under reduced and conventional tillage. The specific location and characteristics of these soils is described in section 3.3.1.

4.3.2 Soil Sampling and Preparation

Soil samples were collected in May 2000 (Fairland clay and clay loam and Hallboro sandy loam series) and July 2000 (Newdale clay loam and Varcoe clay loam series) to examine the impact of soil characteristics, tillage system and landscape position on the sorption of imazethapyr. To obtain a representative clean sample for each soil series, four samples were collected randomly outside each plot using PVC pipe soil cores (5 cm radius and 10 cm depth). The samples were mixed in plastic freezer bags, and the composite samples were transported to the laboratory in coolers at approximately 4°C, and placed in a freezer. Prior to analysis, the samples were thawed at room temperature, and sieved through a 4 mm sieve. Following 48 hours of air-drying, the soil was passed through a 2 mm sieve to remove plant residues.

4.3.3 Imazethapyr Solutions

The herbicide stock solution (100ug/mL) for this study was prepared by dissolving analytical imazethapyr (99% purity; SUPELCO; Bellefonte, PA) in 0.01 M $CaCl_2$ (aq) solution. Imazethapyr solutions were prepared at 1, 2, 4, 8, and 16 ug/ml by diluting the 100 ug/ml stock solution with 0.01 M $CaCl_2$ (aq).

4.3.4 Sorption Procedures

Sorption studies were performed using the batch equilibrium method to determine the sorption of imazethapyr on soils. Sorption isotherms were determined in triplicate using a 1:3 absorbent / solution ratio. The batch equilibrium procedure was as follows: For each of the five herbicide concentrations (1, 2, 4, 8, and 16 ug/ml) 5 grams of soil was mixed with 15 ml of imazethapyr solution in 50 ml Teflon tubes. The suspensions were rotated for 24 hours at room temperature with equilibrium being reached after 8 hours

(Gennari et al. 1998). Sorbent and solution phases were separated by centrifugation at 10,000 RPM for ten minutes to obtain a clear supernatant. A liquid-liquid extraction was performed using 10 ml of supernatant and 5 ml of methylene chloride. Exactly 2 ml of the methylene chloride solution was removed, and the methylene chloride was dried-down with nitrogen gas, leaving the extracted imazethapyr residues. The samples were reconstituted in1 ml of methanol and 100 μl of phenyltrimethylammonium hydroxide, a derivitizing agent, and then placed in a water bath at 50°C for 20 minutes. The samples were analyzed using the NPD detector of a Hewlett Packard 6890 gas chromatograph (GC) with a 7683 autosampler and Hewlett Packard Chemstation software. The GC column used was a J&W DB-5MS with a length of 30 m, an inside diameter of 0.32 mm, and a film thickness of 0.25 μm. The carrier gas was helium, and the detector gases were hydrogen and air. The amount of herbicides sorbed onto the soil was calculated by the difference between the initial and the equilibrium herbicide concentrations. The Freundlich distribution coefficient, K_f [$\mu g^{1-1/n}\ g^{-1}\ mL^{1/n}$], was quantified by nonlinear regression using the empirical Freundlich equation in the log transformation form:

$$\log C_s = \log K_f + {}^{1/n} \log C_e \qquad (2)$$

where C_s = the concentration of imazethapyr sorbed by soil at equilibrium [$\mu g\ g^{-1}$], C_e = the concentration of imazethapyr in solution at equilibrium [$\mu g\ mL^{-1}$] and 1/n = dimensionless Freundlich constant.

4.3.5 Statistical Analysis

The data generated from the batch equilibrium experiments were subjected to analysis of variance (ANOVA) using SigmaStat® 2.03 software (1995 SPSS Inc). The linear regression analysis on the data used the SigmaPlot® 2000 software (1986-2000 SPSS Inc).

4.4 RESULTS

The Freundlich sorption isotherms showed a good fit to the measured data for all soils (r^2 >0.90), with the exception of the Hallboro reduced tillage sandy loam soil (0.72 $\mu g^{1-1/n}$ g^{-1} $mL^{1/n}$) and Hallboro conventional tillage sandy loam soil (0.68 $\mu g^{1-1/n}$ g^{-1} $mL^{1/n}$) (Table 4.1). The order of Kf values, from highest to lowest, for imazethapyr sorption was: Varcoe conventional tillage clay loam soil > Newdale conventional tillage clay loam soil > Varcoe reduced tillage clay loam soil > Fairland conventional tillage clay loam soil > Hallboro conventional tillage sandy loam soil > Hallboro reduced tillage sandy loam soil > Newdale reduced tillage clay loam soil > Fairland reduced tillage clay soil. The magnitude of the Kf values is indicative of weak to moderate binding of imazethapyr to soils. The slopes of isotherms (1/n) generally indicate nonlinear relationships between herbicide concentration and sorption ($1/n < 1$ and $1/n > 1$), and ranged between 0.78 $\mu g^{1-1/n}$ g^{-1} $mL^{1/n}$ and 3.15 $\mu g^{1-1/n}$ g^{-1} $mL^{1/n}$ (Table 4.1).

For each of the four soil series, sorption of imazethapyr was greater in soils under conventional tillage than in soils under reduced tillage. The differences in sorption were significant for Fairland reduced tillage clay soil and Fairland conventional tillage clay loam soil ($p < 0.001$), Hallboro reduced tillage sandy loam soil and Hallboro conventional tillage sandy loam soil ($p < 0.05$), Newdale reduced tillage clay loam soil and Newdale conventional tillage clay loam soil ($p < 0.001$), and Varcoe reduced tillage clay loam soil and Varcoe conventional tillage clay loam soil ($p < 0.05$) (Table 4.1).

Table 4.1 Freundlich sorption constants (Kf), slope of the isotherm (1/n) values, and isotherm coefficients of determination (r^2) for the sorption of imazethapyr on various conventional tillage (CT) and reduced tillage (RT) soils.

Soil	Kf ($ug^{1-1/n}g^{-1}ml^{1/n}$)	1/n	r^2	p-value
Fairland				
CT clay loam	1.28±0.10	1.87±0.11	0.91±0.01	p <0.001
RT clay	0.74±0.01	3.15±0.66	0.90±0.10	
Hallboro				
CT sandy loam	0.99±0.02	1.43±0.03	0.68±0.05	p <0.05
RT sandy loam	0.87±0.03	1.42±0.11	0.72±0.11	
Newdale				
CT clay loam	1.77±0.07	0.98±0.05	0.99±0.01	p <0.001
RT clay loam	0.78±0.04	1.33±0.01	0.99±0.01	
Varcoe				
CT clay loam	2.55±0.45	0.78±0.02	0.97±0.03	p <0.05
RT clay loam	1.55±0.02	1.00±0.90	0.90±0.06	

Imazethapyr also had a greater affinity for the Varcoe clay loam soils (lower slope position) than the Newdale clay loam soils (upper slope position). The difference in sorption in the two slope positions was significant in the reduced tillage plots, 1.55 $\mu g^{1-1/n}$ g^{-1} $mL^{1/n}$ (Varcoe clay loam) and 0.78 $\mu g^{1-1/n}$ g^{-1} $mL^{1/n}$ (Newdale clay loam) ($p < 0.001$), but not the conventional tillage plots, 2.55 $\mu g^{1-1/n}$ g^{-1} $mL^{1/n}$ (Varcoe clay loam) and 1.77 $\mu g^{1-1/n}$ g^{-1} $mL^{1/n}$ (Newdale clay loam) ($p = 0.1$).

Linear correlations were applied between Kf values and soil components to discern their importance to imazethapyr sorption by soil. No correlation was found between Kf and organic carbon ($r^2 = 0.20$), clay fraction ($r^2 = 0.01$), or pH ($r^2 = 0.24$), despite the fact this study included soils with a wide range of organic carbon contents (0.68% - 4.23%) and clay fractions (10% - 41%), and a moderate pH range (5.89 to 7.74).

4.5 DISCUSSION

Imazethapyr sorption has been widely studied in many different soils and humic acids with comparatively similar results. The results from this adsorption experiment differ considerably from previous studies, which found correlations between sorption and various soil factors like pH, organic matter and clay fraction.

Soil behavior of ionic herbicides, like imazethapyr, is affected by pH, clay fraction and organic matter (Stougaard et al. 1990). Soil solution pH influences sorption by affecting the ionic character of organic matter and mineral surfaces, and the molecular structure of the herbicide. Sorption of imazethapyr should occur primarily at lower pH levels since the acidic and basic pKa's of the herbicide are 3.9 and 2.1, respectively, and sorption of ionizable herbicides is greatest near the pKa (Best and Weber 1974; Kells et al. 1980). This occurs due to the protonation of the functional groups, which results in a positively charged molecule, and the decrease of repulsive forces between soil constituents and the herbicide allowing for increased molecular sorption. At higher pH levels, imazethapyr would be largely dissociated and significant anion sorption would not be expected (Stougaard et al. 1990).

The regression analysis showed a very weak correlation ($r^2 = 0.24$) between Kf and pH. These results differ from other studies that found a strong correlation between Kf and soil solution pH (Che et al. 1992; Gennari et al. 1998; Loux et al. 1989a); however, these studies used soils with a wider range of pH levels. Che et al. (1992) studied imazethapyr sorption at soil pH levels of 3.0, 5.0, and 8.0, while Gennari et al. (1998) conducted experiments on soils and humic acids with pH levels ranging from 4.5 to 7.5. Loux et al. (1989a) studied sorption of imazethapyr on various soils, sediments and exchange resins with soil solution pH values between 4.2 and 8.3, and found that sorption was greatest at pH levels below 6.0. The limited range of soil pH in this experiment, 5.89 to 7.60, particularly the lack of range of soils with pH levels below 6.0 where greater sorption is expected, may be a reason for the lack of correlation between pH and imazethapyr sorption.

Table 4.2 Freundlich sorption constants (Kf), soil pH, organic carbon content (OC), and clay fraction of Fairland, Hallboro, Newdale, and Varcoe conventional tillage (CT) and reduced tillage (RT) soils.

Soil	Kf ($ug^{1-1/n}g^{-1}ml^{1/n}$)	pH	OC %	Clay Fraction %
Fairland				
CT clay loam	1.28±0.10	5.89±0.09	3.05	27.3±0.8
RT clay	0.74±0.01	6.45±0.17	3.27	41.7±0.4
Hallboro				
CT sandy loam	0.99±0.02	6.36±0.13	1.13	14.2±0.7
RT sandy loam	0.87±0.03	6.98±0.11	0.68	10.0±0.6
Newdale				
CT clay loam	1.77±0.07	7.74±0.11	3.62	27.1±0.4
RT clay loam	0.78±0.04	7.51±0.22	2.87	27.2±0.7
Varcoe				
CT clay loam	2.55±0.45	7.60±0.13	3.19	27.2±0.8
RT clay loam	1.55±0.02	7.42±0.18	4.23	29.1±0.5

The effects of pH on sorption in this experiment may also be restricted because of the nature of the soil textures. Loux et al. (1993) found that pH had a greater influence on sorption in soils with relatively low clay contents. Six of the eight soils in this study are classified as clay or clay loam (Table 4.2), possibly narrowing the potential effects of pH on imazethapyr sorption by soil.

Though the soils used in this study had a broad range of organic carbon content (OC), a very weak correlation ($r^2 = 0.20$) was found between Kf and OC. Previous studies on imazethapyr sorption found Kf positively correlated with organic matter (Che et al. 1992; Gennari et al. 1998; Loux et al. 1989a; Oliviera et al. 1999). However, the correlations were largely influenced by data derived from experiments on soils with lower pH values. Gennari et al. (1998) found positive correlations between Kf and organic matter, especially when soils were divided according to soil pH >6.5 and pH <6.5. An experiment conducted by Loux et al. (1989a) yielded similar results with imazethapyr sorption increasing with increasing organic carbon content, especially at low soil pH. Oliviera et al. (1999) attributed increases in Kd values for soils with pH <6.25 to organic carbon content. Unfortunately, the limited number of acidic soils in this study disallows any type of data separation according to soil pH to determine if a stronger correlation is possible.

Soil texture, clay fraction in particular, can influence imazethapyr sorption. Stougaard et al. (1990) found that sorption was greater in fine-textured soils than coarse-textured soils. Loux et al. (1989a) studied imazethapyr sorption on numerous soils and found a positive correlation between Kf and clay content. However, other studies on imazethapyr sorption yielded no correlation between Kf and clay fraction (Gennari et al. 1998; Oliviera et al.

1999). In accordance with these studies, this sorption experiment also found no correlation between Kf and clay content ($r^2 = 0.01$), though the clay fractions of the soils ranged from 10 to 41%.

Greater sorption of imazethapyr occurred in the conventional tillage soils for all soil series, but this could not be fully explained based on the observed differences in soil characteristics between conventional and reduced tillage soils. In the Fairland series, the conventional tillage clay loam soil had a slightly acidic pH that may have allowed for greater sorption. The Hallboro sandy loam conventional tillage soil had higher organic carbon and clay contents providing more sorption sites for the imazethapyr molecule. This was also the case in the Newdale clay loam series as the conventional tillage soil had increased organic compared with the reduced tillage soil. However, the Varcoe clay loam conventional tillage soil showed greater sorption than the reduced tillage soil despite Varcoe clay loam reduced tillage soil having a lower pH, and higher organic carbon and clay contents, factors that promote increased imazethapyr sorption. It is possible that other properties such as the quality of soil organic matter could have influenced imazethapyr sorption.

The Varcoe clay loam series in the lower slope showed greater imazethapyr sorption than the Newdale clay loam soils in the upper slope. In the conventional tillage soils, the Varcoe and Newdale clay loam soils had similar pH and clay fraction, but the Varcoe clay loam soil had a slightly higher organic carbon content, which may account for the greater sorption on this soil. The Varcoe clay loam soil from the reduced tillage area had both higher organic and clay contents than the Newdale clay loam soil, which could have contributed to the greater Kf value for this soil.

In summary, there is a lack of correlation between the Kf values and pH, organic carbon, and clay fraction for the entire data set. However, when examining individual series, the influence of these parameters on imazethapyr sorption is visible to a small extent.

4.6 CONCLUSION

Imazethapyr sorption was greater in the conventional tillage soils than reduced tillage soils. Higher Freundlich constants, Kf, were also obtained for the lower slope soils (Varcoe clay loam) in comparison to the upper slope soils (Newdale clay loam) in both the conventional and reduced tillage plots. Results from the batch equilibrium experiments in this study could not be well correlated with the measured soil parameters pH, organic carbon, and clay fraction. The soils used in this study had a wide range of organic matter and clay contents, but there was a limited number of soils with a pH <6.0. This may have contributed to the lack of correlation over the entire data set as the strong influence of pH on the ionic character of organic matter, clay surfaces, and the imazethapyr molecule, and the subsequent effects on sorption could not be adequately observed in this study.

5.0 GENERAL DISCUSSION

5.1 Impact of tillage on imazethapyr efficacy and carryover

Organic matter, texture, pH, moisture, and microbial populations are all important soil factors that help predict herbicide carryover. These properties are influenced by management practices like tillage (Locke and Bryson 1997). The influence of management practices on soil properties is not always consistent, and can vary considerably within fields and regions making it difficult to predict potential carryover problems.

Soils under reduced tillage generally have greater organic matter content (Andruilo et al. 1987; Doran 1980; Carter and Mele 1992), higher microbial populations (Angers et al. 1993; Linn and Doran 1984), improved soil moisture conditions (Wagger and Denton 1992), and increased soil acidity (Dick 1983; Weill et al. 1989) in comparison to soils under conventional tillage. The physical-chemical soil properties measured for this study indicate that the four soil series were not strongly influenced by tillage practice. For example, reduced tillage soils had greater organic matter contents than conventional tillage soils, but only in the Fairland clay and Fairland and Varcoe clay loam soils. The reduced tillage Newdale and Varcoe clay loam soils had a lower soil pH, but the difference in pH between the conventional and reduced tillage soils for these two series was actually quite negligible. Soil moisture content in the reduced tillage soils was significantly greater than the conventional tillage soils, but on average across the four soil series only 50% of the time.

As outlined above, the differences in soil properties between conventional and reduced tillage fields in this study were quite variable indicating that general trends in soil conditions observed in other studies should not be relied upon as an absolute, and that actual physical measurements of soil properties in individual fields might be necessary to evaluate carryover problems.

The results from this study ascertained that imazethapyr persistence would occur to greater extent in medium to fine-textured soil with moderate to high organic matter content, and to a lesser extent in soils with little to no organic matter. These findings are in general agreement with other studies that examined imazethapyr persistence. Loux et al. (1993) found imazethapyr appeared to be more persistent in clay soil with 3.3% organic matter than a silt loam soil with 1.6% organic matter content. Stougaard et al. (1990) discovered imazethapyr was less persistent and more efficacious and mobile in coarse-textured soils. Goetz et al. (1990) concluded imazethapyr was more persistent in soil with higher clay and organic matter contents.

This study also concluded there was limited imazethapyr bioactivity one year following application, and any residual herbicide in the soil would not injure subsequent crops in rotation. Similarly, Jensen et al. (1995) found that sensitive crops were not affected by imazethapyr one year following application. Loux et al. (1993) also observed that imazethapyr residues remaining in silt loam and clay soils 52 weeks after application were not sufficient to reduce corn yields the following year.

Based on this study, it was determined that imazethapyr persistence is not strongly influenced by tillage system or landscape position. When examining the possibility of herbicide persistence, factors like soil texture and organic matter content would be better indicators of potential imazethapyr carryover. Producers worried about herbicide carryover would be best to determine the texture and organic matter content of their fields before applying herbicides. Imazethapyr applied to fields with coarse textures and lower organic matter contents are unlikely to have a carryover problem. Soils with finer textures and higher organic matter have a greater potential for imazethapyr carryover, but residual activity is still expected to be minimal. The Fairland soil with a clay fraction of 41% and organic matter content of 3.27% used in this study had very little herbicide bioactivity one year following application indicating imazethapyr applied at recommended rates is not a threat to subsequent crops, and may be used in herbicide rotations.

5.2 Improvement of experimental design for future studies

Though the experimental design used in this study was effective, some changes could be made to improve future research. One improvement would be to increase the number of replicates used in both the bioassay and batch equilibrium experiments to reduce the large variability in the results. Another improvement to the bioassay experiment would be to increase the initial number of oat seeds planted in each pot to 15 then thin the number of shoots to 10 after emergence. The damage and effects caused by implanting pregerminated seeds into the soil would no longer sway the bioassay results, allowing for reductions in biomass and shoot growth to be solely attributed to residual herbicide in the soil. Another possible change would be to combine both the field and laboratory experiments by extracting imazethapyr directly from the treated soils in the field study. This would allow an examination of imazethapyr adsorption as it would occur under 'real' soil conditions. Future research using field studies would also benefit by using a wider range of soils. Including soils with lower pH values or medium textures would have contributed to the results of this study. Ultimately the field and laboratory experiments were conducted under the best possible circumstances, and yielded some valuable insight into imazethapyr persistence.

6.0 CONCLUSION

Bioassay results from the 1999 and 2000 field studies indicated that imazethapyr is more persistent in medium and fine-textured soils with higher levels of organic matter. Results from the batch equilibrium experiments could not be well correlated to measured soil parameters pH, organic carbon, and clay fraction, but previous studies concluded that imazethapyr sorption was influenced by these parameters, particularly soil pH. The effects of tillage practice on soil properties were not consistent throughout this study, and did not regularly conform to results from previous studies, indicating that generalizations regarding soil parameters affected by tillage are ambiguous and should not be relied on as absolutes. Producers including imazethapyr-sensitive crops in their rotation would be best to test their soil to determine texture, organic matter content, and pH prior to using herbicide products containing imazethapyr on their fields. However, herbicide bioactivity one year following application on clay soils with increased organic carbon content under reduced and conventional tillage used in this study was low, indicating the risk of crop injury due to imazethapyr carryover is small, regardless of tillage practice or landscape position.

7.0 CONTRIBUTION TO KNOWLEDGE

Tillage and landscape position did not strongly influence imazethapyr persistence in soil. Producers concerned with herbicide carryover should determine the texture and organic matter content of their fields as these two soil properties are better indicators of potential imazethapyr persistence. Herbicide applications to coarse-textured soils with low organic matter contents are unlikely to have a carryover problem. Soils with finer textures and higher organic matter contents have greater potential for imazethapyr persistence. Limited imazethapyr bioactivity measured one year following application indicates that significant injury to subsequent crops is unlikely, and herbicides containing imazethapyr can safely be used in an herbicide rotation.

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