

Residues of imazethapyr in soybean grain, straw and soil under application of long term fertilizers in Typic Haplustert

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ABSTRACT

Experiment was conducted to see the effect of balance and imbalance level of nutrients on residue of imazethapyr. Imazethapyr was applied at 100 g/ha 25 days after sowing of soybean crop for managing annual and perennial broad-leaf weeds and sedges. Soil and crop samples were collected at harvest and analyzed by HPLC using photo diode array detector. Retention time of imazethapyr was found to be approximately 3.5 minutes. Residues of imazethapyr in soil at various treatments were found 0.0124 to 0.0121 µg/g. Concentration of imazethapyr in soil was maximum in 100% N + imazethapyr (T₇) and minimum in (100% NPK + imazethapyr (T₈)) treatments respectively. At harvest residue of imazethapyr was not found in soybean grains and straw in any set of treatments except T₇ (100% N).

Key word: Soil, Soybean grain, Straw, Residue, Imazethapyr, HPLC analysis

Imazethapyr belongs to imidazole group with a toxicity class of III and used as a selective herbicide. It is applied as pre-plant incorporated, pre-emergence, and post-emergence to control grasses and broadleaved weeds in pulse crops (Herbicide handbook 1989, Thomson 1993 and Anon. 2006).

Chemically, imazethapyr is a weak acid (like amphoteric) having both carboxylic acid and pyridine functional groups thus dissociation to the anion resulting very low sorption at high pH levels. Imazethapyr absorption varies with a number of soil properties but the soil organic carbon content has been considered the single best predictor of sorption for non-polar, non-ionizable organic chemicals.

Microbial degradation and photodecomposition have been suggested as major dissipation mechanism for imazethapyr. Imazethapyr incorporated in the soil persisted longer than imazethapyr applied to the soil surface.

Vertisols are dark-coloured Montmorillonitic, Hyperthermic and Typic haplustert clay that develop cracks when expanding and contracting with changes in moisture content. A few reports showed imazethapyr residues in soil in India (Sondhia 2008) and stream and river water in Midwestern US at concentrations above the maximum residue limits in 71% of samples (Battaglin *et al.* 2000). Hollaway *et al.* 2004 reported that 10% residues of applied imazethapyr persisted for 24 and 5 months after treatment in clay soil and sandy soil respectively. Soil pH affects imazethapyr sorption-desorption, which in turn

can affect persistence and bioavailability (Bresnahan *et al.* 2000).

Different nutrient level may also affect persistence of herbicides in crop and soil. But hitherto no work has been reported on the behaviors of herbicide persistence under balanced and imbalanced level of nutrients under field conditions in soybean crop. Thus experiment was conducted to examine the residue of imazethapyr in Typic haplustert under application balance and imbalance level of nutrients.

A field experiment was conducted on Vertisol in *kharif* 2007-08 at research farm of Jawahar Lal Nehru Krishi Vishva Vidyalaya, Jabalpur. The soil of the experimental field was medium black belonging to Kheri series of fine montmorillonitic hyperthermic family of Typic Haplustert. The soil was clay loam in texture. The experiment consists of 10 treatments replicated four times in a randomized block design. The gross plot size being 17 x 10.8 m with 1 m spacing in between the plots and 2 m spacing between the replications. The crop was raised under irrigated conditions with recommended package of practices. The recommended fertilizer dose (100% NPK) for soybean crop was applied on soil at the rate, of 20:80:20 NPK respectively through Urea, SSP, DAP, and MOP.

Treatments include T₁ (50% NPK), T₂ (100% NPK), T₃ (150%NPK), T₄ (100% NPK+Hand Weeding), T₅ (100% NPK+Zn), T₆ (100% NP), T₇ (100%N), T₈ (100% NPK+FYM), T₉ (100% NPK – S), T₁₀ (Control) and

imazethapyr was applied in all treatments at the rate of 100g/ha except T₄.

The soil was clay loam in texture (clay 56.82%, silt 17.91% and sand 25.27%) with available nitrogen (261 kg/ha), phosphorus (23.25 kg/ha), potassium (243 kg/ha), organic carbon (7.4 g/kg), EC (0.14 - 0.17 d/Sm) and pH (7.56).

Soil samples at 0-20 cm depth, were drawn at least from eight places from each plot to measure terminal residue of imazethapyr at harvest. Five-soil cores each comprised of approximately 3 kg soil were randomly collected from each plot avoiding the outer 20 cm fringes of the plots by using a soil auger up to a depth of 20 cm from the surface. Pebbles and other unwanted materials were removed manually. The soil samples were air-dried, powdered and passed through a 3 mm sieve to achieve uniform mixing. At harvest approximately 500 g of representative samples of soybean grains and straw were collected. The straw samples were cut in small pieces and air-dried. Soybean grain and straw samples were then ground in mechanical grinder.

Imazethapyr reference analytical standard was obtained from ACCU standard, USA. All other chemicals and solvents used in the study were analytical grade obtained from E. Merck and all the solvents were glass distilled prior to use.

Imazethapyr residues were determined as per literature reported method (Sondhia 2008). A portion of 20 g of representative samples (soil, grain and straw) in was taken a 250 ml Erlenmeyer flask, to which 40 ml 0.5 N NaOH solution was added the flasks were then shaken on a horizontal shaker for 1-2 hrs. After shaking samples were filtered and filtrates were collected. pHs of extracts were made up to 2 through the addition of 6N HCl and shaking vigorously. The content was transferred to a 250 ml separating funnel and partitioned in dichloromethane. The organic layer was dried on anhydrous sodium sulfate and passed through activated charcoal. The solvent evaporated to dryness on rotary evaporator.

Soybean grains and straw sample extracts were cleaned on a glass adsorption column (10 cm x 2 cm id) packed with celite (1 g) and activated charcoal (0.25 g) between anhydrous sodium sulphate (2 g) at each end. The concentrated extract was added at the top after pre-washing with methanol and eluted with methanol and water (60: 40). Eluates were collected and solvent was the flasks were then shaken evaporated on a rotary vacuum evaporator.

Imazethapyr residues were analyzed on a Shimadzu high-performance liquid chromatograph equipped with

diode array detector at 250 nm, Phenomenex C-18 (ODS) column (250 x 4.6 mm). The mobile phase used for the analysis of imazethapyr in soil, grain and straw was mixture of methanol and water (70:30) at a flow rate of 0.8 ml/min. Finally, residues were dissolved in 2 ml of methanol and filtered through pall Nylon 0.45mm filter paper. 20 ml of the aliquot of samples and standard was injected using micro syringe. Using these condition imazethapyr was eluted at 3.5 minutes. At these conditions retention time was found approximately 3.5 minutes.

Residues of imazethapyr as detected by HPLC in soil, soybean grain and straw are presented in (Table 1). The concentration of imazethapyr residues was found 0.0221 and 0.0190 µg/g at harvest in soybean grain and straw, respectively in treatments which consisted of 100% N application. However, in the other treatments residues of imazethapyr were found below detectable limits. Residues of imazethapyr in soil at various treatments were found 0.0124, 0.0119, 0.0106, 0.011, 0.0114, 0.0134, 0.0121 mg/g in T₁, T₂, T₃, T₅, T₆, T₇, T₈ and T₉, respectively. Concentration of imazethapyr in soil was maximum (0.0134 µg/g) and minimum (0.0093 µg/g) in T₇ (100% N + imazethapyr) and T₈ (100% NPK + imazethapyr), respectively.

The lowest concentration of imazethapyr in soil T₈ (100% NPK+ FYM+ imazethapyr) could be due to application of FYM along with 100% NPK. Low concentration of the imazethapyr in soil is also compensated by the increased microbial activity, which increased the rate of degradation (Shelton 1991, Sondhia 2005). Besides the organic matter, the clay content can also play an important role in degradation rate of pesticides.

Some author reported that imazethapyr has a rapid initial phase of degradation, followed by a slower second phase leading to long term persistence especially in clay soil (Hollaway *et al.* 2004, Sondhia 2008). Thus a few authors recommends re-cropping periods of up to 34 months for imazethapyr due to leaching and persistence that may damage subsequent rotation crops.

Several researchers have shown that imazethapyr adsorption increases as soil clay and organic matter content increase, however soil pH is the dominant factor determining imazethapyr adsorption to soil (Sondhia 2005, Stougaard *et al.* 1990). The experimental soil was rich in clay content (56.82%) that might be the reason that considerable amount of imazethapyr residues was detected from soil and crop produce at harvest but amount of residues was low in soil as compare to crop produce at harvest.

Table 1. Residues of imazethapyr at harvest time in soil and soybean grains and straw

Treatments	Residues* ($\mu\text{g/g}$)		
	Soil	soybean grain	straw
50% NPK+ imazethapyr (100 g/ha)	0.0124	ND	ND
100% NPK+ imazethapyr (100 g/ha)	0.0119	ND	ND
150% NPK+ imazethapyr (100 g/ha)	0.0106	ND	ND
100% NPK+ hand weeding	<0.001	ND	ND
100% NPK+Zn+ imazethapyr (100 g/ha)	0.011	ND	ND
100%NP+ imazethapyr (100 g/ha)	0.0114	ND	ND
100% N+ imazethapyr (100 g/ha)	0.0134	0.0221	0.0190
100% NPK+FYM+imazethapyr (100 g/ha)	0.0093	ND	ND
100% NPK S + imazethapyr (100 g/ha)	0.0121	ND	ND
Control + imazethapyr (100 g/ha)	0.0124	ND	ND

Detection limit – 0.001 mg/g, *Average of four replications

Though in the present study residues were found below the maximum residue limits in soil and grains set by some European countries (0.1 mg/kg), there is still concern for use of imazethapyr because of its persistence in soil and crop produce.

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