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Residues of imazethapyr in field soil and plant samples following an application to soybean

Shobha Sondhia*
ICAR - Directorate of Weed Research, Jabalpur, Madhya Pradesh 482 004

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ABSTRACT

Imazethapyr is widely used in pulses and leguminous crops including soybean for control of a broad spectrum of weed species. This has often resulted in carryover effects on several sensitive rotational crops. Therefore field studies were conducted for two consecutive years to evaluate residues of imazethapyr in the soil and the soybean crop produce. Imazethapyr was applied at 100 and 200 g/ha as post-emergence herbicide in soybean field. Residues of imazethapyr were found in the range of 0.011 to 0.063 µg/g in the straw following an application in soybean field at 100 to 200 g/ha in both the years. However in the soil and soybean oil, residues were found below 0.01 µg/g in both the years at two levels of application of imazethapyr. The overall residues were less in the soil as compared to the plant samples. Terminal residues of imazethapyr in soybean plant and soil were found below maximum residue level (MRL) limits. This study demonstrated enrichment of imazethapyr residues in soybean plants after repeated application. Based on this study a pre-harvest interval of 80-90 days for soybean crop after imazethapyr application is suggested.

Key words: Imazethapyr, HPLC-PDA, Oil, Terminal Residues, Soil, Soybean plant

Soybean (*Glycene max*) is one of the most important crops in the world. Weeds impact soybean yields by competing for limited resources, primarily light, water, and nutrients. The yield of soybean crop in Asia is much lower than the potential yield. One of the major reasons for low yield is the severe crop – weed competition during critical crop growth period (Barnes and Lavy 1991) which necessitate the use of herbicides. As a consequence of herbicide use, the presence of residues in field crop may cause numerous environmental problems. Herbicides residues also remain on the soil surface due to the adsorption process which may potentially affect quality and yield of the next crop cultivated on the same field. Stable herbicides may be taken up by plants, which results in unwanted terminal residues (Barnes and Lavy 1991, Battaglin *et al.* 2000). Imazethapyr is used as a selective herbicide for the control of a broad spectrum of weed species (Sikkema *et al.* 2005, Sondhia 2013). Good crop tolerance and weed control in pulses and other leguminous crops have contributed to an increase in the popularity of this herbicide (Loux *et al.* 1989, Sondhia 2013).

Imazethapyr inhibits acetohydroxy acid synthase (AHAS), an enzyme common to the biosynthetic pathway for these amino acids. This inhibition causes a disruption in protein synthesis, which in turn, leads to interference in DNA synthesis and cell growth. Imazethapyr dissipates in soil by microbial degradation and photolysis under field conditions (Stougaard 1990, Sondhia 2013). Imidazolinone herbicides are generally weakly adsorbed to the soil (Gan *et al.* 1994). Organic matter and pH significantly affect imazethapyr behavior in the soil (Mangles 1991). Some authors reported leaching of imazethapyr below 25 cm in four months in acidic and sandy loam soils under laboratory and field studies (Battaglin *et al.* 2000, Sondhia 2013). Residues of imazethapyr were reported in stream and river water in Midwestern US at concentrations above the maximum residue limits in 71% of samples (Basham *et al.* 1987).

Knowledge herbicide to persist in soil and plant and injure rotational crops is important in weed management strategies. Soybean is commonly rotated with wheat in the tropical region. Residue of ALS inhibitors or their metabolites can persist into the following growing seasons and can potentially injure sensitive crops grown in rotation such as canola and lentils, mustard, or sugar beet (Moyer and Hamman 2001, Schoenau *et al.* 2005, Poienaru and Sarpe 2006). Since herbicides are necessary to manage prominent weeds, the presence of this residues in crop produce at harvest is of great concern. Therefore a two years field study was conducted to determine the terminal residues of imazethapyr in soil, soybean grain, oil, oilcake and straw.

***Corresponding author:** shobhasondia@yahoo.com

MATERIALS AND METHODS

Field experiments were conducted for two consecutive years during 2006-07 in a randomized block design with three replications. Soybean variety '*JS 335*' was sown and imazethapyr (10% SL) was sprayed as post-emergence *i.e.* 20 days (after sowing of soybean seeds) at rates of 100 (recommended dose) and 200 g/ha (double the recommended dose). Physico-chemical properties of imazethapyr are given in Table 1. A further three triplicate plots were sprayed with water without any herbicide and maintained as control. The crop was grown under irrigated conditions with recommended package of practices. During 2006 and 2007, the soybean field received approximately 890 and 995 mm rainfall, respectively (Fig. 1).

Soil samples were collected at harvest (110 days), which is equivalent to 90 days after spraying of the herbicide in soybean crop in both the years. Five-soil cores of each approximately 3 kg soil were randomly taken from untreated and treated plots avoiding 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, under shade, powdered and passed through a 3 mm sieve to achieve uniform mixing. The soil was clay loam in texture (clay 35.47%, silt 12.45%, and sand 52.09%), having nitrogen 300 kg/ha, phosphorus 40 kg/ha, and potassium 300 kg/ha, organic carbon 0.82 %, EC 0.35 mmhos/cm and pH 7.2.

At harvest, approximately 500 g of representative soybean grains and straw samples were collected from each imazethapyr treated and control plots. The straw samples were cut in small pieces and air-dried under shade. Soybean grains and straw samples were then ground in mechanical grinder. The imazethapyr reference analytical standard was obtained from AccuStandard, USA. All other chemicals and solvents used in the study were of analytical grade obtained from Merck, Germany. Imazethapyr residues in soil, and plant samples (oil cake, straw and pod) were determined as described by Sondhia (2013) using a Shimadzu HPLC coupled to diode array detector (DAD). Phenomenex C-18 (ODS) column (250 x 4.6 mm) and methanol: water (70:30 v/v) as mobile phase at a flow rate of 0.8 mL/min was used to separate imazethapyr residues. The LOD and the LOQ were found to be 0.001 and 0.01 µg/mL, respectively.

RESULTS AND DISCUSSION

In soil, and soybean oil, residues were found below 0.01 µg/mL in both the applied doses of imazethapyr *viz.* 100 and 200 g/ha, respectively in both the years. In contrast to the soil, residual concentration of 0.022 µg/g and 0.069 µg/g residues were detected in mature soybean pods where imazethapyr was applied at 100 and 200 g/ha doses in 2006. However, in 2007, 0.042 µg/g and 0.081 µg/g residues of imazethapyr were detected in the mature pod of soybean, following an application of imazethapyr at 100 and 200 g/ha, respectively (Table 2). This showed an enrichment of imazethapyr residues in soybean plant parts in second year of application. Residual concentration of 0.011 µg/g were detected in oil cake in 2007 under the lower dose however, 0.026 µg/g to 0.056 µg/g residues were detected in 2006 and 2007 at higher dose.

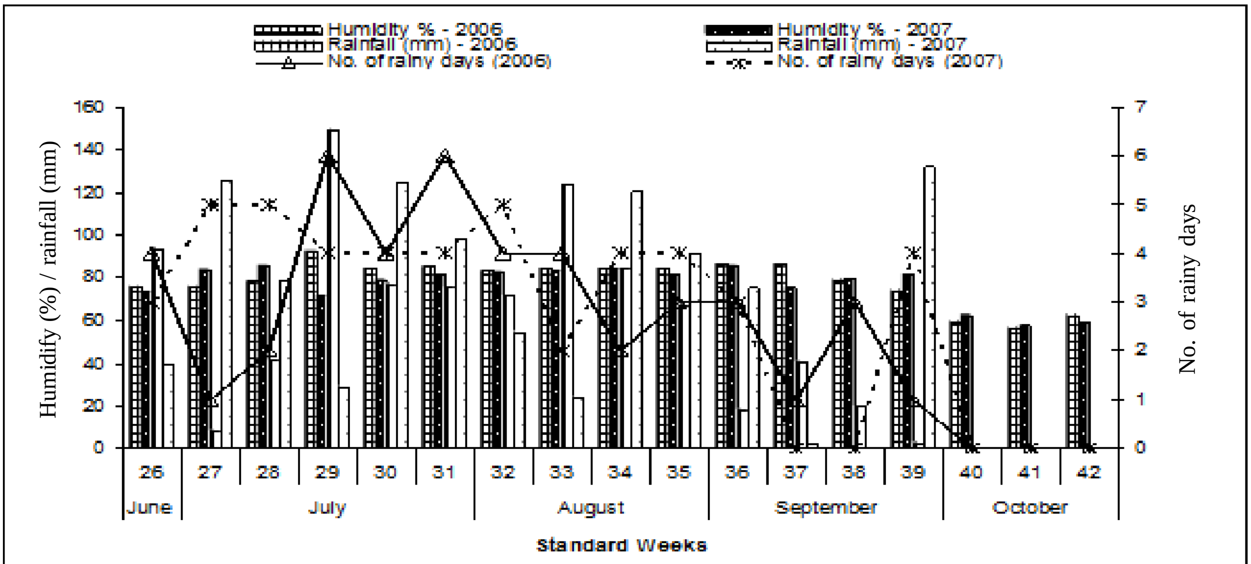


Fig. 1. Variation in humidify, rainfall and number of rainy days during 2006-07

Table 1. Some important physico-chemical properties of imazethapyr

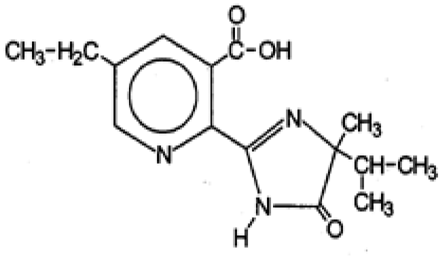
Chemical structure	IUPAC name	
	Molecular formula	$C_{15}H_{19}N_3O_3$
	Molecular weight	289.3
	Formulation	SL 10%
	Solubility in water	1400 mg/L
	Vapor pressure	<1x10^{-7} mmHg at 60 ^{0}C
	Henry's constant	1.30 X 10^{-02} Pa m^3/mol at 25^{o}C
	Partition coefficient Log P_{ow}	1.49

Table 2. Imazethapyr residues in soybean oil, oilcake, grains, straw and soil at harvest

Substrate	Imazethapyr residues (µg/g)*			
	100 g/ha		200 g/ha	
	2006	2007	2006	2007
Soybean straw	0.011 (+0.001)	0.049 (+0.004)	0.013 (+0.002)	0.063(+0.003)
Soil	<0.01	<0.01	<0.01	<0.01
Oil	<0.01	<0.01	<0.01	<0.01
Oil cake	<0.01	0.011	0.026 (+0.001)	0.056 (+0.009)
Mature pod	0.022 (+0.004)	0.042(+0.007)	0.069 (+ 0.008)	0.081(+0.006)

*mean of four replications

Sorption studies conducted in four soil types showed that imazethapyr had low Koc values (19.8-83.9), which suggested that little adsorption would be expected for any of soils and indicated that imazethapyr has high mobility and consequently a high potential to leach (Sondhia 2003). The amount of rainfall during the crop growing season when imazethapyr was applied might also have affected the persistence of the herbicide. During 2006 and 2007 the soybean field received approximately 890 and 995 mm rainfall, respectively. Due to higher solubility of imazethapyr in water (1.4-3.7 g/L), higher rainfall may have enhanced the leaching potential in soil in 2007 after the application of imazethapyr, this may have resulted in a reduced availability of imazethapyr in soil and hence less or no residues were found in soil and soybean plant produce at harvest (Barnes and Lavy 1991, Poienaru *et al.* 2006). However, reduced rainfall in 2006 means that there is increased herbicide adsorption making imazethapyr less available for plant uptake (Cantwell *et al.* 1989, Goetz *et al.* 1990) and less residues in soybean plants. This showed the fast degradation of imazethapyr residues in the soil and plants under reported agroclimatic conditions, although imazethapyr has a soil photolysis half-life of 33 months, and, in some field dissipation studies, the consistently persistence of imazethapyr was reported regardless of the soil type, agriculture practice and climatic effects (Imazethapyr 2015).

Marsh and Lloyd (1996) reported that imazethapyr persisted for longer period in Romanian soil and showed residual effect on succeeding barley and winter wheat even after 2-3 years. Cabbage was reported as the most sensitive to imazethapyr soil residues. Cabbage yields were reduced in 2 of 3 years while cabbage, tomato and cucumber showed visual injury symptoms after imazethapyr application in 2 of 3 years following post-emergence imazethapyr and imazamox application (Sullivan *et al.* 1998). Arora and Sondhia (2013) reported 0.082 and 0.023 µg/g residues in soybean grain and straw as a result of 200 g/ha application of imazethapyr in soybean crop. Low detections of residues was also indicative of low uptake, low translocation, or rapid degradation within the plant (Sidhu and Feng 1993).

Soil type, soil pH and Koc play an important role in the degradation and bioavailability of herbicides (Sullivan *et al.* 1988, Poienaru and Sarpe 2006, Sondhia 2013). Dissipation of imazethapyr is faster in soils with high pH and low adsorption since the amount available in the soil solution for microbial transformation is greater. The soil of experimental field in this study was almost neutral (pH 7.2) so that due to the small adsorption imazethapyr residues were not available in surface soil (0-20 cm) and were consequently not detected at harvest in soil. Sullian *et al.* (1998) reported imazethapyr residues mainly in the top 0-10 cm soil fraction but some imazethapyr was found in 10-20 cm and 20-40 cm depths. Besides the organic matter, the clay content can also play an important role in degradation of pesticides but Hollaway *et al.* (2006) reported persistence of imazethapyr residues for 24 and 5 months after treatment in clay soil and sandy soil respectively.

Some researchers recommend re-cropping periods of up to 6-34 months for imazethapyr due to leaching and persistence that may damage subsequent rotation crops and 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 (Bresnahan *et al.* 2000). Combination of chemical, biological, physical and environmental factors may operate at different level in influencing the degradation of herbicides (Sondhia 2013, 2013). Less persistence of imazethapyr was found in silty clay soil and high organic matter containing soils. Low concentration of the imazethapyr in soil is compensated by high microbial activity, which increased the rate of degradation (Sidhu and Feng 1993, Sondhia 2013).

In the soil almost neutral pH, high organic matter, soil clay content and sufficient rains might be the reason for less terminal residues of imazethapyr. The terminal residues of imazethapyr in plant parts were found higher in 2007 in comparison to 2006, however residues were below the maximum residue limits in soybean plants (0.1 mg/kg) set by some European countries (Canada Gazette 2006). This study demonstrated enrichment of imazethapyr residues in soybean plants after repeated application. Based on this study a pre-harvest interval of 80-90 days for soybean crop after the herbicide application is suggested.

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