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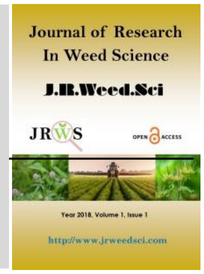

Bioefficacy and Fate of Pendimethalin Residues in Soil and Mature Plants in Chickpea Field

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Abstract

In modern agriculture, use of herbicides has become indispensable for managing weeds in cultivated lands, due to the scarcity of labors in almost all parts of the globe. Herbicides provide effective weed control in agriculture, yet their continuous and excessive use may pose serious health and environment threats. Pendimethalin is proved to be a very effective pre-emergence herbicide for the control of annual weeds in leguminous and other vegetable crops. Therefore bio-efficacy and residues of pendimethalin were determined in chickpea (*Cicer arietinum* L.) crop at farmers' field. Pendimethalin was applied at 796 g ha^{-1} as a pre-emergence herbicide to control weeds in chickpea fields. Pendimethalin was found effective against weeds *viz. Vicia sativa*, *Convolvulus arvensis*, *Chenopodium album* and *Lathyrus sativa*. Terminal residues of pendimethalin were found below <0.001 µg g^{-1} in the chickpea grains and straw samples. However, in the soil, residues were found to be between 0.001 to 0.0029 µg g^{-1}. The study showed degradation of pendimethalin to the legal maximum residues limit (MRL, 0.05 mg kg^{-1}) in the chickpea grains. Based on this study a pre-harvest interval of 131 days was suggested for chickpea crops after pendimethalin application.

Keywords: Bioefficacy; Chickpea; Herbicide; Pendimethalin; Soil; Terminal residues

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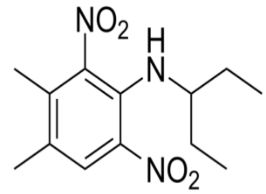
1. Introduction

Herbicides play an important role in higher crop yields by controlling a variety of weeds; however, at the same time their longer persistence in the soil at various surfaces may potentially affect the quality and yield of the next crop cultivated on the same field and may cause several environmental problems. Longer persistence also increases the chance of contaminating surface and groundwater through leaching and runoff processes (Sondhia, 2009a, b; Khare and Sondhia, 2017).

Pendimethalin is rapidly lost by biochemical processes such as photodecomposition, microbial degradation and volatilization (Tsiropoulos and Miliadis, 1998; Sondhia, 2013). Pendimethalin inhibits growth of weeds by disrupting the mitotic sequence through inhibiting production of the microtubule protein, tubulin (Appleby and Valverde, 1989). Adsorption and dissipation of pendimethalin and other similar class of herbicide in the soil has also been demonstrated by Zheng (1993), Sondhia and Dubey (2006), Schleicher et al. (1995), etc. It has been observed that pendimethalin adsorbs rapidly and strongly in the soil due to its high potential for hydrogen bonding. Its persistence in the soil is affected greatly by cultivation, soil temperature, and moisture conditions (Schleicher et al. 1995). Though pendimethalin is categorized as a low-volatile and low leaching compound (Kaleem et al. 2006; Sondhia 2013) (Table 1), however, field dissipation studies demonstrated that pendimethalin is persistent in silty flay soil, and its persistence is longer when it is incorporated into the soil rather than applied to the surface (Zimdahl, 1984, Lee et al. 1997, Sondhia, 2013). Field dissipation studies demonstrated its half-life up to 98 days at 30°C (Kol et al. 2002). Pendimethalin degrades rapidly in anaerobic soil and slowly in aerobic soil conditions. Recent studies demonstrated a potential risk of environmental contamination due to the use of sulfonylurea and imidazoline group of herbicides, which are active at very low rate of application (Fletcher et al. 1993; Peterson et al. 1994; Sondhia, 2013).

Bioefficacy of pendimethalin and other dinitroaniline herbicides in the chickpea crop has also been reported (Chaudhary et al. 2005; Sondhia, 2012; Butter et al. 2015) but not much work has been done to study the bioefficacy of pendimethalin against weeds or their fate in the chickpea crop under actual farmers' field at tropical conditions. Reports have shown that residues of some herbicides and/or their metabolites can persist into the following growing seasons (Peterson et al. 1994; Sondhia, 2008, 2010) and caused phytotoxicity to sensitive crops grown in rotation such as canola and lentils, mustard, or sugar beets (Moyer and Hamman, 2001; Sondhia 2013) is reported as a result of prolonged persistence of some herbicides in the soil.

Table 1- Important physico-chemical properties of pendimethalion.

Chemical structure	
IUPAC name	((N-(1-ethylpropyl)-2, 6-dinitro-3, 4-xylidine)
Molecular formula	$C_{13}H_{19}N_3O_4$
Molecular weight	281.3
Formulation	SL 38.6 %
Solubility in water	1400 mg/L
Vapor pressure	1.94 mPa at 25 ^{0}C
Henry's constant	2.73 x 10^{-02} Pa m^3/mol at 25^{o}C
Partition coefficient Log P_{ow}	5.81

Chickpea (Cicer arietinum L.) is one of the important vegetable crops of winter season mainly sown in September-November and harvested in February-March in this state, under tropical conditions. Madhya Pradesh is a major chickpea producing state contributing 30 to 35% to the national production and production and productivity of chickpea have registered positive growth rate, with the area increased from 19.17 to 25.61 lakh ha and production doubled from 12.26 to 23.71 lakh tones. Productivity has increased from 640 to 925 kg ha-1 during the period (http://www.icar.org.in/files/state-specific/chapter/76.htm). Chickpea is a rich source of protein and form an important part of vegetarian diet. Chickpea contains about 17-20% of protein. Pendimethalin is commonly used as a pre-emergence herbicide and found effective for the control of annual weeds in leguminous and other field crops (Sondhia and Dubey 2006; Lin et al. 2007). Behavior of pendimethalin residues in soils of cotton fields indicated that it is more persistent than trifluralin (Tsiropoulos and Miliadis, 1998). The experimental results of Jaźwa1 et al. (2009) demonstrated that pendimethalin is relatively stable compound in comparison to other dinitroanilines and may cause problems with follow-up crops. Too much reliance on chemical weed control enhances the risk of residues in the soil and crop plants. Consequently the presence of terminal residues in plants or/crop produced at harvest is a matter of great concern. Presently, research on the bioefficacy and terminal residues of pendimethalin in chickpea crop especially in the large vegetables/pulses growing area under sub-tropical climate conditions are very infrequent. The wide range of crops that are treated with this herbicide can result in repeated applications of pendimethalin on the same land. The chickpea farmers considered in this study have been applying pendimethalin herbicides continuously for the last 6-7 years. Thus, current study was undertaken to establish bioefficacy of pendimethalin against weeds and accumulation of the pendimethalin residues in chickpea plants (grains and straw) and soil of chickpea growing fields.

2. Materials and Methods

This study was scrupulously planned to determine terminal residues of pendimethalin in the soil, chickpea grain, and straw at farmers' fields in the chickpea growing area. Field experiments were conducted at five different locations in the village Pola of Majholi district near Jabalpur, Madhya Pradesh in Rabi 2013-14 (Table 2). Farmers' fields were located at 23.130N latitude, 79.580E longitude at an altitude of approximately 390 m above mean sea level. Jabalpur is located in the center of India and fall in the agro-climatic region of Kymore plateau and Satpura Hills agroclimatic region. The climate of the region is subtropical, with an average rainfall of 1500 mm. The soils in the experimental area are mostly black, known as vertisols, and belongs to kheri-series of fine montmorillonite, Hyperthermic family of Typic Haplusterts. Rice and soybean are the major crops grown in the rainy season and chickpea in winter season.

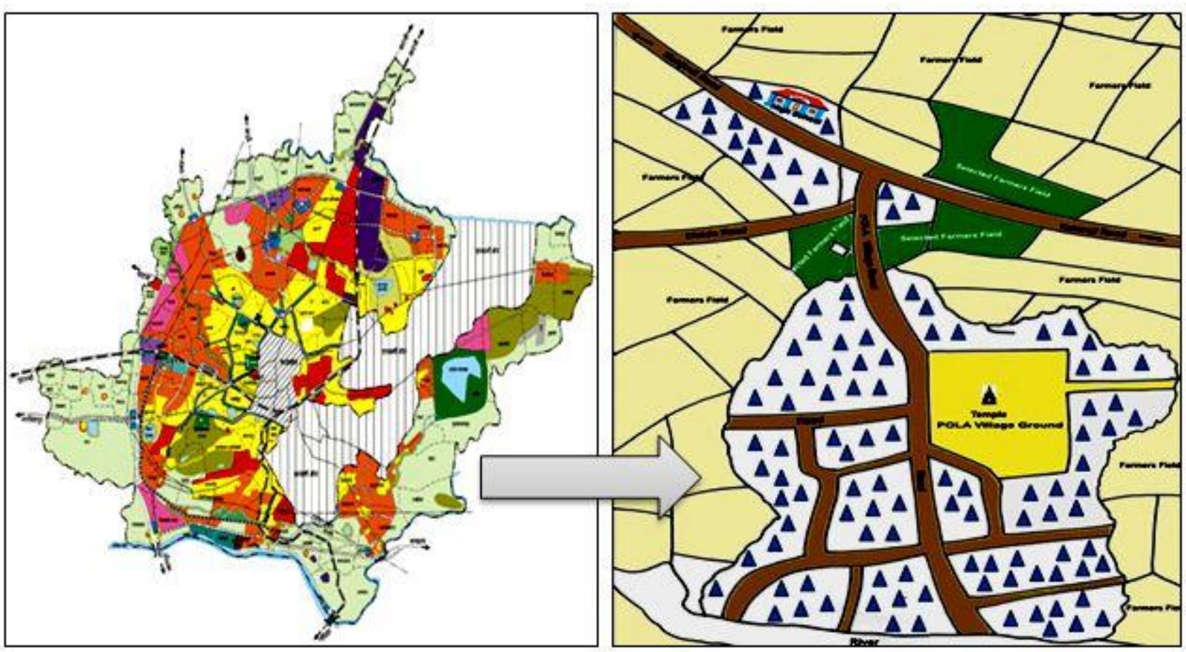


Figure 1- Chickpea growing area under the study area near Jabalpur, Madhya Pradesh

The soils taken from farmers' field in different locations have diverse physico-chemical characteristic. Local chickpea variety JG 130 was sown, and commercial available formulation of pendimethalin (Stomp Xtra 30 EC) was applied at 796 g ha^{-1} in chickpea crop as pre-emergence herbicide i.e., 3 days after sowing of chickpea seeds at rates of 70 g ha^{-1} in each location as per the recommended guidelines. The crop was grown under irrigated conditions with the recommended package of practices of Madhya Pradesh State.

Table 2- Soil physicochemical characteristic at five locations of study area.

Soil characteristics	Field location				
	Location 1	Location 2	Location 3	Location 4	Location 5
pH	8.04	8.06	8.11	8.09	8.14
EC(dS m^{-2})	0.12	0.110	0.16	0.15	0.12
N (kg ha^{-1})	156.80	146.30	219.5	167.25	203.80
K (kg ha^{-1})	11.59	16.762	19.72	24.86	25.09
Organic Carbon (%)	0.377	0.405	0.418	0.423	0.29

2.1. Collection of samples for terminal residues in soil and plant: For the determination of terminal residues

Plant and soil samples were collected at harvest (approximately 133 days, which is equivalent to 131 days after spraying the herbicide on chickpea crop) randomly from ten to fifteen different locations, representing an area of 500 M^2 areas in each location, so that the sample areas covered a wide range of chickpea-growing areas under various farming practices. Five to six soil cores of each having approximately 3 kg of soil were taken from each location for residue studies from each untreated and treated plots with a soil auger up to a depth of 15 cm from the surface. Bulk soil samples from each location were then air dried, powdered, and passed through a 3-mm sieve. Approximately 3 kg of representative chickpea plant sample was collected randomly from the pendimethalin treated and control plots (no pendimethalin application). Grains were separated from the plants samples. The plant straw samples were cut in small pieces and air-dried. Chickpea grains and straw samples were then ground in a mechanical grinder.

Pendimethalin residues analysis from plants and soil were done by HPLC following the method of Sondhia (2013). Pendimethalin reference analytical standard of 99.9 % purity were purchased from ACCU standard, USA. Chemicals and solvents used in the study were of analytical grade (E Merck). The spiked samples (chickpea straw, grains, and soil) were fortified separately with standard solutions to obtain 0.01 and 0.5 μg g^{-1} concentrations, and the extraction and cleanup processes were described above as the percentage of the recovery of pendimethalin (Table 3). The pendimethalin residue was determined by comparing the peak area of the samples and calibration curves of five levels of standards. The limit of determination (LOD) and the limit of quantification (LOQ) were found to be 0.001 and 0.01 μg mL^{-1}, respectively (Sondhia, 2008).

2.2. Bio-efficacy of pendimethalin against weeds

Weeds emerged and weed dry weight after application of pendimethalin as a pre-emergence herbicides was determined at 60 days to evaluate bioefficacy of pendimethalin against weeds and effect on chickpea grain yield and benefit cost ratio (B:C) as a result of pendimethalin application in chickpea fields.

3. Results and Discussion

3.1. Recoveries and detection limit:

Following the method of Sondhia (2013), the retention time of pendimethalin was found to be approximately 6.4 minutes. Pendimethalin recoveries from plant and soil samples were varied from 92 to 80% with a recovery of 86-85 for straw, 80 to 88 for chickpea grains, and 92-83 for soil at two levels of fortification, 0.5 and 1.0 µg g^{-1}. The limit of detection of the method was 0.001 µg ml^{-1}. At this detection level the signal to noise ratio was 3:1. Due to low organic matter content of the soil, soil blanks did not exhibit any peak interfering with the retention time of pendimethalin; therefore, soil samples were not subjected to a cleanup step. The recovery for pendimethalin grain and straw was acceptable up to fortification level of 0.01 µg g^{-1} (Table 3). Recoveries of pendimethalin from various matrixes at different concentration levels were satisfactory.

Table 3- Recovery of the pendimethalin from soil, grain and straw at 0.5 to 1.00 µg g^{-1} fortification level.

Samples	Fortification (µg g^{-1})*	Amount recovered (µg g^{-1})*	Recovery (%)
Chickpea straw	0.50	0.43$\pm$0.021	86
	1.00	0.85$\pm$0.012	85
Chickpea grain	0.50	0.40$\pm$0.016	80
	1.00	0.88$\pm$0.013	88
Soil	0.50	0.46$\pm$0.011	92
	1.00	0.83$\pm$0.015	83

3.2. Bio-efficacy of pendimethalin against weeds:

The bio-efficacy of pendimethalin was determined against the weed flora in the chickpea growing fields. The farmers' field was infested with mainly broadleaved weeds. Some sedges such as *Cyperus rotundus* were also present in low density. Fields were infested heavily with *Vicia sativa*, *Convolvulus arvensis*, *Chenopodium album* and *Lathyrus sativa*. Weed population per square meter showed an effective control of these weeds in chickpea fields in all the five locations as compared to farmers' practice where no chemical weed management was made. Weed count was found to be 32 to 92 no m-2 in various fields in comparison to the farmers practice. No weeding resulted in 106 to 236 no m-2 of weeds in chickpea fields. No weeding also caused higher weed dry weight (127 to 173.8 g m^{-2}) and reduced grains yield in the chickpea fields due to high growth of weeds. The bioefficacy of pendimethalin against weeds density is presented in Table 4. In case of weed population, significant difference was observed between the farmers practice and pendimethalin application (P=0.05). The weed population in the weedy check was significantly higher than other plots where pendimethalin was applied as pre-emergence herbicides to control annual weeds.

Higher yield of chickpea grains (2.10 to 1.14 t ha^{-1}) was found under pendimethalin application at 796 g ha^{-1} in the farmers yield as compared to no weeding practice opted by the farmers which resulted in low yield (0.84 to 1.23 t ha^{-1}). Results showed that pendimethalin effectively controlled all the weeds appeared in the field and gave higher weed control efficiency and overall average benefit of Rs. 17040 ha^{-1} with benefit: cost (B: C) ratio of 2.93 over farmers practice (FP) (Table 4).

Table 4- Effect of pendimethalin on weed control efficiency and yield of chickpea at Majholi locality of Jabalpur.

Farmer's names	Treatment	Weed count (no. m^{-2})	Dry weight (g m^{-2})	Grain yield (t ha^{-1})	Economic benefit over FP (Rs. ha^{-1})	B:C ratio
Lahori Patel	Pendimethalin 796 g ha^{-1}	92	35.4	1.19	10,500	2.04
	Farmers practice*	236	154.0	0.84	-	1.57
Lochan Patel	Pendimethalin 796 g ha^{-1}	67	32.6	1.89	22,500	3.25
	Farmers practice - no weeding	164	127.0	1.14	-	2.13
Sugreev Patel	Pendimethalin 796 g ha^{-1}	27	30.3	2.10	21,300	3.62
	Farmers practice - no weeding	106	135.6	1.39	-	2.03
Atal Patel	Pendimethalin 796 g ha^{-1}	32	32.6	1.73	19,800	2.98
	Farmers practice - no weeding	134	173.8	1.07	-	2.00
Chetu Lal	Pendimethalin 796 g ha^{-1}	32	15.6	1.60	11,100	2.76
	Farmers practice - no weeding	106	80.2	1.23	-	2.30

Farmers practice* No herbicide, only little manual weeding

The bioefficacy at 60 days after sowing revealed that application of pendimethalin at 796 g ha^{-1} reduced the weed population to a greater extent as compared to the farmers practice. The lowest weed population was found in the field of farmer Sugreev Patel. The applications of pendimethalin did not cause phytotoxicity to the chickpea plants. The maximum weed dry weight was recorded in farmers practice. The result indicated that pendimethalin 38.6 % EC at 796 g ha^{-1} was found effective against the grassy weeds as well as broad leaved weeds found in the chickpea fields in comparison to the weed management based on farmers practice.

The bioefficacy of pendimethalin (stomp-Xtra) 38.7% at 796 g ha^{-1} rate was significantly higher as compared to the farmers practice (P=0.05). The grain yield of the chickpea crop was highest under the pendimethalin treated fields. In general, pendimethalin application in the farmers' field enhanced the grain yield by approximately 29.4 to 39.7 % over farmers practice (no herbicide) by reducing the weed population. Butter et al. (2015) also reported that application of pendimethalin at 750 g/ha were effective in minimizing weed density and weed population as compared to other herbicidal treatments at all stages of observation. Butter et al. (2015) also reported high yield of chickpea using pendimethalin.

3.3. Terminal residues of pendimethalin in the soil chickpea field

Pendimethalin residues in different commodities are presented in Table 5. At harvest (approximately after 131 day), pendimethalin residues in the soil of chickpea field were found to be 0.001 to 0.0029 μg g^{-1} at 796 g ha^{-1} dose in three locations. However, pendimethalin residues were found below the detection limit in the grains and straw of chickpea field at harvest. This showed that with passage of time pendimethalin residues decreased in the soil of chickpea field successively.

It has been reported that pendimethalin is highly adsorbed onto the soil and organic matter (Sondhia and Dubey, 2006). Pendimethalin adsorbs strongly in the topsoil's and has reported soil-water partition coefficients (Kd values) varied from 99.8 (0.59 % organic carbon) to 1638 (16.9 % organic carbon) (Zheng and Cooper, 1993). Increasing soil organic matter and clay content is increased soil binding capacity of pendimethalin (Extoxnet, 1996). Because of the strong affinity for the soil, pendimethalin was expected to be less mobile. In spite of high adsorption and less mobility of pendimethalin to soil, several reports demonstrated longer persistence of pendimethalin in soil. Half-lives of 60 to 62 days were reported by Jaźwa1 et al. (2009) in the soil of funnel field. Jaźwa1 et al. (2009) reported 0.017 μg g^{-1} pendimethalin residues in funnel crop. They demonstrated that the degradation rate of pendimethalin was slow ($t_{1/2}$=60 days) and therefore presence of its residues in the soil can be locally toxic for the succeeding plants as residues in the soil was 0.221 μg g^{-1}, and were 3 times higher than in other samples of the soil taken in the period of crops.

Triantafyllidis et al. (2005) reported presence of pendimethalin residues up to 129 days after the treatment in the top soil of tobacco field with a half-life of 23-27 days. Lazic (1997) reported that 50 % of the herbicide degraded in an average of 50 days and resulted pendimethalin 0.239 μg g^{-1} residues in young onion and 0.113 μg g^{-1} in ripe onion (Lazic, 1997).

Table 5- Terminal residues of pendimethalin in chickpea grain, straw and soil at harvest.

Location	Farmer's names	Residues (μg g^{-1})		
		Soil	grain	Straw
1	Lahori Patel	0.0010	<0.001	<0.001
2	Lochan Patel	0.0029	<0.001	<0.001
3	Sugreev Patel	<0.001	<0.001	<0.001
4	Atal Patel	0.0012	<0.001	<0.001
5	Chetu Lal	<0.001	<0.001	<0.001

Sharma and Mehta (1989) reported 0.103 μg g^{-1} residues in onion at harvest when the pendimethalin treatment was 2.0 kg ha^{-1}. Tsiropoulos and Miliadis (1998) reported 0.054 μg g^{-1} residues in onions treated at 2.0 kg ha^{-1}. Several other factors such as, soil contact, microbial action, soil moisture, and photodecomposition can affect terminal pendimethalin residue levels in the harvested crop. In reported study, mature plant samples collected at harvest were analyzed for terminal residues of pendimethalin in the fields of various farmers. In the soil, the highest residual concentration of 0.0029 μg g^{-1} was found in field of Mr Atal Patel. However, the lowest residues concentration of pendimethalin residues was detected from location 1 of Lahori Patel (0.010 μg g^{-1}).

In all the locations, pendimethalin residues in grains and straw were found to be <0.001 μg g^{-1} (Table 4). This showed the fast degradation of pendimethalin residues in the soil and plants under reported agroclimatic conditions, although pendimethalin has a soil photolysis half-life of 98 days, and, in some field dissipation studies, the consistently persistence of pendimethalin was reported regardless of the soil type, agriculture practice and climatic effects (Lazic et al. 1997; Sondhia, 2012).

The fate and persistence or dissipation of a chemical is mainly controlled by its physico-chemical properties, crop management practices and environmental conditions that include climate, soil physico-chemistry and microbial activity in the soil. In contrast to the plant samples, at two locations in farmers' fields, pendimethalin residues were found to be below 0.001 μg g^{-1} in the top 15 cm of soil, however 0.0010, 0.0012 and 0.0029 μg g^{-1} residues in soil at 15 cm of soil depth was detected at 796 g ha^{-1} of the applied dose of pendimethalin. In the literature, some studies demonstrated longer pendimethalin in some soils (Lazic et al. 1997; Jaźwa1et al. 2009; Sondhia, 2012). Due to the longer crop growth period of chickpea crop, pendimethalin got sufficient time to degrade in the soil and plants following its application; this reduced bioavailability of pendimethalin in the chickpea grains and straw, causing there to be no residues in the grain and straw at harvest at all locations. Less adsorption and a slightly alkaline pH 8.04-8.14, also favored the fast dissipation of pendimethalin residue in the surface soil (0-15 cm); thus, low pendimethalin residue was consequently detected at harvest in the soil at three locations.

These results suggested that there is less risk of pendimethalin carryover to succeeding crops planted the year following pre-emergence application to chickpea in subtropical conditions. Prevailing soil properties of the experimental field lead to less terminal residues of pendimethalin in the soil and plants. The terminal pendimethalin residues in plant parts was found to be below the maximum residue limit in chickpea plants set by the EPA, USA (0.05 mg kg^{-1}). Based on this study, a pre-harvest interval of 131 days after herbicide application is suggested for chickpea crops. It can be concluded that pendimethalin application considerably increased chickpea yields compared with the unweeded control (farmers practice) under subtropical conditions. Further trials may be conducted involving more locations, soil types, climatic conditions and supplemental weed control methods using other pre emergence herbicides in conjunction with other post-emergence herbicides.

Conflict of interest

Authors declare no conflicts of interest for this study.

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