



Degradation dynamics of alachlor in maize ecosystem

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Alachlor (CAS No. 15972-60-8) was developed by Monsanto Company (USA) and introduced in 1967 for pre-emergence or pre-plant control of broad spectrum of grass, sedge and broad-leaf weeds in corn, soybeans, dry beans, cotton, grain sorghum, sunflowers, peanuts and other crops. Ramesh *et al.* (2002) investigated the dissipation of alachlor in soil and plant in field condition (cotton cropping system), and in soil, water and fish in simulated model ecosystem. At harvest, cotton lint and seed samples were found to contain alachlor well below the detectable level. However, trace amounts of residues were found in cotton oil. The green leafy vegetable samples did not show any toxic symptoms of alachlor residues. The objectives of this research are the concise enumeration of the gas-liquid chromatographic (GC-ECD) separation methods used for the determination of residual fate, dissipation pattern and degradation behaviour of alachlor in maize crop and in its soil substrates.

The field experiment was conducted at Bidhan Chandra Krishi Viswavidyalaya, Mohanpur, Nadia, West Bengal during 2011-12. As alachlor 50% EC is a viscous liquid formulation, the mode of application is spraying in soil at 2500 and 5000 g/ha. Only one spraying was done on 38 days after sowing by knapsack sprayer. Samples of maize leaf and soil were collected from 8-10 places of each replicated plots in each sampling dates. Samples from each untreated control plots were also collected in the similar way. Samples were collected following standard sampling procedure. No trimming/washing was done. After following quartering method, 100 gm of valid representative maize leaf, maize grain and soil samples were collected. Maize leaf and soil samples were collected at 0 (2 hr), 1, 3, 5, 7, 10, and 15 day after the application of the herbicide from each treatment and control plots. Soil samples were also collected at harvest. Maize grain samples were collected only at harvest. Extracts were cleaned up immediately after extraction as far as possible but in

some cases the extracts were also stored in the cold chamber (-20°C) for a minimum period of time. Technical grade alachlor of 98% purity was obtained from Sinochem India Company Pvt. Ltd., India. All the reagents used were of analytical reagent (AR) and the solvent used was J T Baker grade. Working solution was prepared in ethyl acetate. GC model Chemito GC 1000 equipped with an Electron Capture Detector coupled with C-2000, 1.7 software was used for quantification of alachlor in different substrate.

The recoveries of alachlor from maize and soil samples were fortified at 0.01–0.10 µg/g levels using the present method of extraction and cleanup (Table 1). The limit of detection (LOD) and limit of quantification (LOQ) were 0.003 µg/g and 0.01 µg/g, respectively. For residue analysis, the samples of maize leaf (0.50 kg) and soil (1 kg) were drawn on 0 (2 h), 1, 3, 5, 7, 10, 15 days after spraying.

The extraction and cleanup of the samples were done according to the QuEChERS method. A representative sub-sample of 100 g was cut into small pieces and macerated at high speed blender for 2 min. 10g (5g for leaf sample) representative sample each of soil/Leaf/grain was taken in a 50 ml polypropylene centrifuge tube with a teflon lined cap and then 10 ml of J T Baker grade ethyl acetate was

Table 1. Recoveries of alachlor from maize field soil, maize leaf and maize grain

Substrate	Amount fortified (µg/g)	Amount recovered* (µg/g)	Recovery (%)	Average recovery (%)
Maize Soil	0.01	0.0094	94	94.67
	0.05	0.05	100	
	0.10	0.09	90	
Maize Leaf	0.01	0.0093	93	96.33
	0.05	0.043	86	
	0.10	0.11	110	
Maize Grain	0.01	0.0091	91	91
	0.05	0.044	88	
	0.10	0.094	94	

*Average of three replicates

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added to it, vortexes for 1 minute. After that 4 g of activated MgSO₄ and 1 g of NaCl was added, the sample was again vortexes for 1 min and then centrifuged for 10 minutes at 10000 rpm at 25 °C. After centrifugation, 4 ml supernatant ethyl acetate phase was collected with the help of micropipette in a 15 ml polypropylene centrifuge tube containing 50 mg of PSA and 150 mg of anhydrous MgSO₄. The tube was tightly sealed and vortexes for 1 min. and then centrifuged again for 10 min at 10000 rpm. 2 ml clean supernatant was transferred to an appropriate vial, then filtered through 0.2µ membrane filter and was ready for the final analysis in GC-ECD. A stock solution of 1 µg/g analytical grade alachlor (purity 98.1%) was used as an external standard. 1 µl of each cleaned up sample extracts obtained from different substrates were injected into GC-ECD under the operation parameters listed above. The alachlor residues were identified by comparing the retention time of sample peak with that of analytical standard.

Persistence of alachlor in Soil

The initial deposit of alachlor in maize field soil was found in the range of 1.15 to 2.50 µg/g for single (2500 g/ha) and the double (5000 g/ha) doses, respectively (**Table 2**). Around 90% of the initial residue was dissipated by 10th day, irrespective of dose. On and from 15th day onwards, no residue was detected in recommended dose, while 95.92% of the initial deposit got dissipated at 15th day at double the recommended dose. The residues of alachlor were below the quantification level of 0.01 µg/g at harvest at both the doses. The dissipation pattern of alachlor in soil followed first order kinetics for both single and double doses (**Figure 1a**). The residual half- life was found to be 3.24 days for single dose and 3.17 days for double dose. A half-life value of 3 days for alachlor in mix load site soil was reported (Anastasia

E.M. Chirnside *et al.* 2011). The persistence of acetanilide herbicides in soil is dependent on many factors such as temperature, soil moisture, pH and nutrient level (Beetsman and Deming 1974 and Taiwo and Oso 1997). Herbicide dissipation in cover crop residues was often more rapid than in soil with half-lives from 3 to 11 days (Locke *et al.* 2005).

Persistence of alachlor in maize leaf

The initial deposit of alachlor in maize leaf was detected to be 0.35 µg/g in case of recommended dose and for double dose the corresponding value was 0.66 µg/g (**Table 2**). Around 80 % of the initial residue was dissipated by 7th day at the recommended dose, while the value was 71% for the double dose. On and from 10th day onwards, no residue was detected at the recommended dose, while for the double dose, around 86.36% of the initial deposit got dissipated at 10th day, and the compound was below quantification limit of 0.01 µg/g on 15th day. No residues of alachlor were detected at harvest. The dissipation behavior of alachlor in maize leaf followed first order kinetics for both single and double doses (**Figure 1b**). The residual half- life was found to be 3.20 days for single dose and 3.62 days for double dose.

Persistence in maize grain and soil at harvest

No residue of alachlor was detected in maize grain and cropped soil at harvest. The residues were below the LOQ (0.01 µg/g) in both the treatments and the commodities. Similar observation in cotton lint and seed sample was reported (Ramesh and Maheshwari 2002).

The initial deposits (2 h after spraying) of alachlor in maize leaf ranged from 0.30 - 0.39 µg/g at recommended dose and 0.61 - 0.65 µg/g at double dose but in maize field soil, the corresponding values

Table 2. Alachlor residue in maize leaf and soil under maize crop

Days (Sampling)	Residues in maize soil (µg/g)		Residues in maize leaf (µg/g)	
	T ₁ = 2500 g/ha Mean ± S.D. (% Dissipation)	T ₂ = 5000 g/ha Mean ± S.D. (% Dissipation)	T ₁ = 2500 g/ha Mean ± S.D. (% Dissipation)	T ₂ = 5000 g/ha Mean ± S.D. (% Dissipation)
0	1.30±0.07	2.45±0.08	0.35±0.05	0.66±0.06
1	1.04±0.05(20)	1.96±0.06(20)	0.28±0.03(20)	0.52±0.05(21.21)
3	0.78±0.05(40)	1.47±0.05(40)	0.21±0.03(40)	0.39±0.04(40.91)
5	0.58±0.04(55.38)	1.10±0.06(55.10)	0.14±0.03(60)	0.28±0.04(57.58)
7	0.39±0.04(70)	0.72±0.04(70.61)	0.07±0.02(80)	0.19±0.03(71.21)
10	0.13±0.03(90)	0.24±0.05(90.20)	0	0.09±0.02(86.36)
15	0	0.10±0.03(95.92)	-	0
Regression equation	y = -0.093x + 3.153	y = -0.095x + 3.433	y = -0.094x + 2.562	y = -0.083x + 2.831
Half-life (T _{1/2}) in days	3.24	3.17	3.20	3.62

LOQ= Limit of quantification (0.01 µg/g); LOD= Limit of detection (0.003 µg/g)

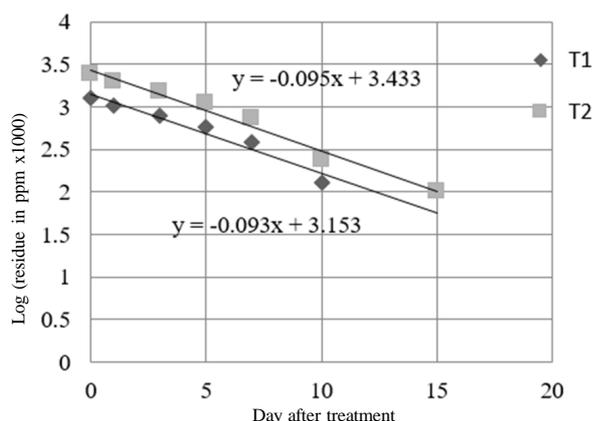


Figure 1a. Linear plot of dissipation of alachlor in soil under crop

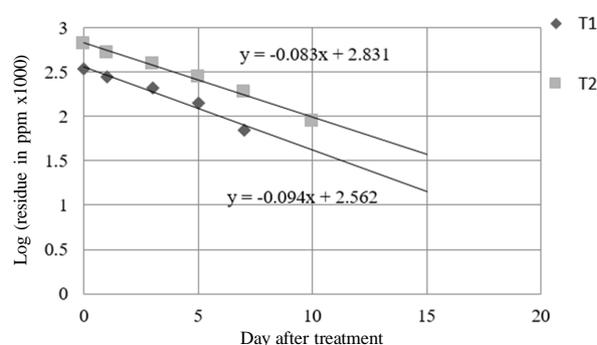


Figure 1b. Linear plot of dissipation of alachlor in maize leaf

were 1.23 - 1.31 $\mu\text{g/g}$ and 2.46 to 2.52 $\mu\text{g/g}$ for recommended and double dose respectively. The alachlor residues in maize leaf were below the quantification level of 0.01 $\mu\text{g/g}$ at 15th day after the application whereas, it still persisted up to 15th day in soil samples irrespective of doses. No residue of alachlor was observed at harvest in mature maize grain and soil. The half-life values were in the range 3.17–3.62 days irrespective of dose and substrate. As the alachlor residues were found below the quantification level in all the harvested maize substrates, it may be concluded that the herbicide will not pose any residual problem and may be safely recommended for use in maize ecosystem.

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SUMMARY

A field experiment was conducted at Bidhan Chandra Krishi Viswavidyalaya, Mohanpur, Nadia, West Bengal during 2011-12 to study the degradation dynamics of an acetanilide herbicide alachlor in maize ecosystem. Alachlor (50% EC) was applied to the cropped soil at 2500 and 5000 g/ha at 38 days after the sowing of seed. The initial deposits of alachlor in maize leaf ranged from 0.30 - 0.39 $\mu\text{g/g}$ at recommended dose and 0.61-0.65 $\mu\text{g/g}$ at double dose but in maize field soil, the corresponding values were 1.23-1.31 $\mu\text{g/g}$ and 2.46 to 2.52 $\mu\text{g/g}$ for recommended and double dose, respectively. The alachlor residues in maize leaf were below the limit of quantification LOQ of 0.01 $\mu\text{g/g}$ on 15th day after application whereas, it still persisted up to 15th day in soil samples irrespective of dose. At harvest, residues of alachlor were not observed in maize grains and soil samples. The half-life values of alachlor in maize ranged from 3.17-3.62 days irrespective of dose and substrate.

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