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Degradation of Alachlor in Water and Tropical Soils of India

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Alachlor [2-Chloro-N-(2,6-diethylphenyl)-N-(methoxy methyl) acetamide] is a selective pre-emergence aniline herbicide control weeds in groundnut, sugarcane, soybean, maize, brassicas, cotton, radish, oilseed rape and transplanted crops like onion, chillies, cabbage and cauliflower. It controls a wide range of grasses and broadleaved weeds like barnyard grass and certain species of *Digitraia, Setaria, Amaranthus, Protulaca* and *Trianthema* when used at 1.68 to 4.48 kg a.i./ha. Being a selective systemic herbicide, it is absorbed principally by germinating shoots and roots with translocation throughout the plant. At ambient temperature, pure alachlor is a colourless soild and slightly soluble in water (Table-1, Chesters et al. 1989, Pesticide Manual, 1997). Alachlor is degraded in soil by microbial action to 2-chloro-2',6'-diethylacetamide and finally to aniline derivatives. The half-life of alachlor in soil varies from 1-30 days which is depending on soil type and climate. About 90% of the alachlor applied in soil degraded in 4 weeks in no-till and in 8 weeks in convention tillage (Sadeghi et al. 1997). Half-life of alachlor in soil at 80, 50 and 20 % field capacity were found to be 8, 10 and 20 days (Shaid et al.,1993). At 80% field capacity, the half-life of alachlor in sandy loam and clay loam soil were 18.8 days and 11.1 days respectively (Zimdahl et al. 1982). Half-life of two alachlor formulations namely emulsifiable concentrate (EC) and microencapsulated (ME) in a clay loam soil cultivated with maize crop were found to be 10 and 16 days respectively (Trevisan et al. 1997). Weed et al. (1998 and 1999) reported 50% dissipation in less than 3 days and 90% dissipation in 15 to 23 days using M2CM hybrid model. In surface water, 55% of the applied alachlor degraded within 28 days (Pesticide Manual, 1997)

The influence of different physico-chemical properties on the persistence of alachlor in soil and water is not very well understood. The present investigation was therefore, carried out to study the persistence of alachlor in inceptisol (alluvial soil), ultisol (red soil), vertisol (black soil) and oxisol (laterite soil) which vary widely in physico chemical properties. Degradation studies have also been carried out in acidic (pH-5.0), neutral (pH-7.1) and basic (pH-8.4) buffer solution.

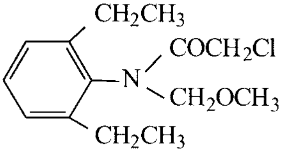

Figure 1. Chemical structure of alachlor

Table 1. Physico-chemical properties and environmental fate of alachlor

Molecular formula	$C_{14}H_{20}ClNO_2$
Molecular weight	269.8
CAS RN	15972-60-8
Colour	White
Physical state	Solid
Odor	None
Melting point	40-41° C
Boiling point	100° C at 0.003 kPa
Vapour pressure	$2.9X10^{-6}$ kPa at 25° C
Vapour density	$3.2X10^{-4}$ mg L^{-1}
Octanol-water partition coefficient (K_{ow})	430
Water solubility	240 mgL^{-1}
Specific gravity	1.133 at 25° C
Soil persistence $T_{1/2}$(days)	1-30
Water persistence $T_{1/2}$(days)	28

MATERIALS AND METHODS

Analytical grade (98.8%) alachlor and its EC formulation Lasso (50EC) were provided by Monsanto Company, St. Louis, U.S.A. and Monsanto Chemical of India Limited respectively.

Common chemicals and reagents of LR grade procured from the local market were used as received except anhydrous sodium sulphate which was pre-washed in glass column with acetone and dried in an oven at 110° C for 2 hours. Solvents dichloromethane, hexane and acetone of LR grade were obtained from E. Merck (India) and were glass distilled before use. Florisil (for residue analysis) and charcoal (activated) were obtained from Merck (Germany) and E. Merck (India) respectively. Charcoal powder and Florisil were also activated in oven for 1 hour at 110° C before use.

Stock solution of alachlor for fortification of soils and water samples were prepared from its Lasso (50EC) formulation. The standard solution for GC analysis was prepared using analytical grade material (98.8%). Series of dilutions were made to get the required working solutions.

Four type of fresh soil samples i.e. inceptisol, ultisol, vertisol and oxisol were collected from the agricultural fields at 0-15 cm depth to study the persistence and dissipation of alachlor formulation under aerobic conditions. The inceptisol sample was collected from New Delhi; vertisol from Bangalore (Karnataka); ultisol from Ranchi (Jharkhand) and oxisol from Bhubaneswar (Orissa). Soil samples were dried at room temperature and sieved through 100 mesh size. Soil properties of each soil were determined using standard methods (Table-2).

Table 2. Physico-Chemical properties of test soils.

Soil type	pH	Organic matter	CEC	Particle size (%)		
				Sand	Silt	Clay
Inceptiol	8.1	0.23	2.10	73.2	7.8	19.0
Ultisol	6.5	0.59	13.0	54.2	27.2	18.6
Vertisol	7.6	1.10	45.6	8.2	39.0	52.8
Oxisol	5.2	0.36	3.50	77.1	10.90	12.0

Dried and sieved soil (800 g) samples were fortified with alachlor formulation at 0.5 μg/g and 1.0 μg/g concentration, mixed thoroughly and transferred into series of 100 mL glass beaker (50 g each). Control experiment was conducted side-by-side in all the four types of soils. Required quantity of distilled water was added to bring the soil to 50% field capacity moisture level and weight of the beaker noted.

Persistence of alachlor in water was studied in buffer solutions adjusted at pH 5.0, 7.1 and 8.4. Acidic buffer solution (pH-5.0) was prepared by dissolving 87 g of sodium acetate in 400 mL of distilled water, 10.5 mL of glacial acetic acid and diluted to one liter. The pH of the solution was adjusted at 5 by addition of acetic acid. Neutral buffer solution (pH-7.1) was prepared by dissolving 9.8 g of potassium acetate in water and diluted to one liter. The pH of 7.1 was adjusted by addition of either acetic acid or potassium hydroxide solution. Basic buffer solution (pH 8.4) was prepared by dissolving 16.7 g of anhydrous potassium dihydrogen phosphate in distilled water and diluted to one liter. The pH was adjusted at 8.4 by the addition of sodium hydroxide solution.

Soil sample (50 g) was taken in a glass petri dish and dried for 24 hr at room temperature to remove moisture. The dried powder soil mixed with 0.25 g of activated charcoal, 0.25 g of Florisil and 10 g of anhydrous sodium sulphate was packed dry in glass column (25 mm id) over a cotton plug. The column was eluted with 100 ml of hexane: acetone mixture (9:1, v/v) and the elute concentrated in a vacuum rotary evaporator at 40^0 C. The residue was finally dissolved in hexane for GC analysis.

Water samples (50 mL) were taken in a separating funnel (250 mL) and saturated sodium chloride solution (25 mL) and of 15% dichloromethane (DCM) in hexane (25 mL) was added to it. The funnel was shaken vigorously for 2-3 minutes and the lower aqueous layer drawn into a fresh 250 mL separating funnel. The

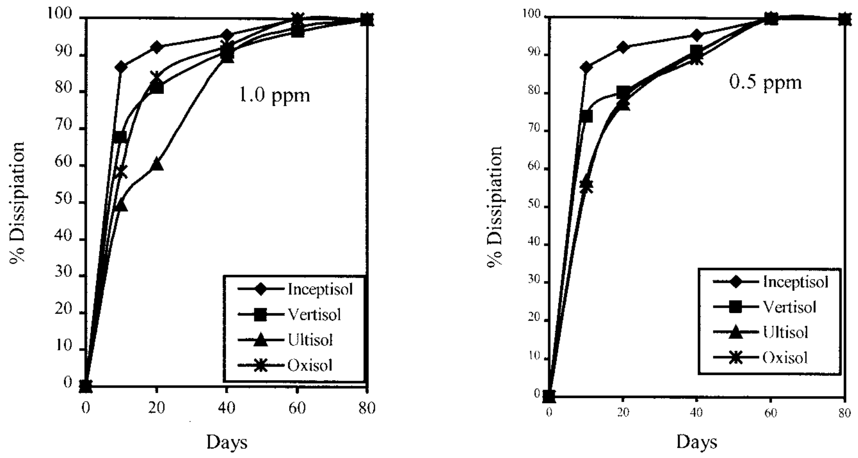

Figure 2. Percent dissipation of alachlor in different soil types

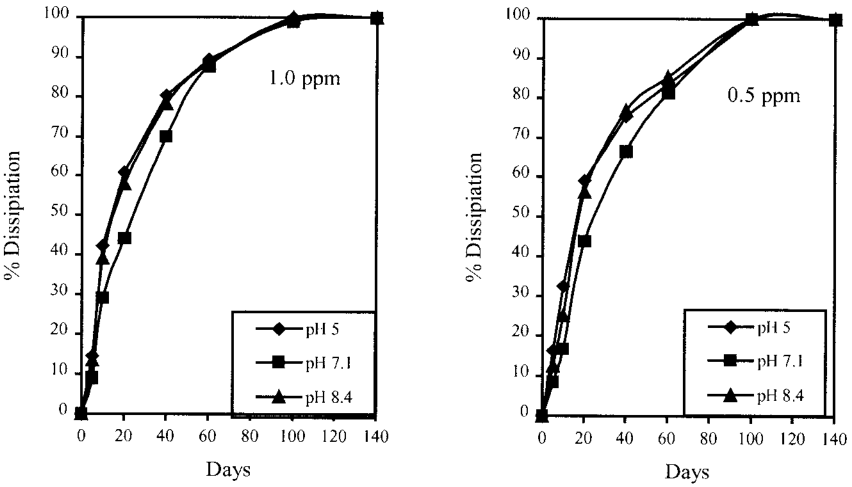

Figure 3. Percent dissipation of alachlor in water at different pH

partitioning was repeated two more times using fresh portions of 25 mL of dichloromethane-hexane mixture. The three extracts were combined and dried by passing through anhydrous sodium sulphate over a small pad of cotton. The extracts were concentrated to about 1-2 mL in a rotary vacuum evaporator at 40^0 C. About 6-8 mL of hexane was added and the mixture evaporated under reduced pressure. The process was repeated once more to remove the last traces of DCM. The residue was finally dissolved in hexane for GC analysis.

The residues of alachlor were estimated on Hewlett Packard 5890 series-II gas chromatograph coupled with 3390 series integrator and fitted with 30 meter capillary fused silica column, 0.53 mm id, stationary phase, BPX-50, film thickness - 0.5 micron and electron capture detector (ECD). High purity nitrogen gas (IOLAR grade-1) with the flow rate 20 ml/min was used. GC operating temperatures (^{o}C) were oven: 185, injector: 260 and detector: 300. Under these conditions, the retention time of alachlor was found to be 3.80 min.

The method followed for the extraction of alachlor from soil and water samples was validated by fortifying at five different concentrations ranging from 0.1 µg/g to 1.0 µg/g level. The recoveries of alachlor in soil samples ranged from 87 to 94% and 93 to 98 % in water samples.

RESULTS AND DISCUSSION

During GC analysis, no degradation of alachlor was observed. Good resolution and straight baseline was observed in the GC analysis. The retention time, peak area and peak height were observed for the estimation of alachlor residues. Residues of alachlor as detected upto 40 days in different soil samples fortified at 0.5 and 1.0 µg/g concentraton. No residues were detected after 80 days. Its half-life ($T_{1/2,}$ days) in inceptisol, vertisol, ultisol and oxisol soil spiked at 0.5 and 1.0 µg/g were found to be 9.7, 9.8; 12.4,13.2; and 11.8,11.02 and 12.6,10.76 days respectively. The rate of dissipation of alachlor was lowest in vertisol and highest in inceptisol (Figure-2). Residues of alachlor was below detectable limit in all the four type of soils after 80 days. Residues of alachlor in acidic, neutral and basic buffer solution fortified at 0.5 and 1.0 µg/g level persisted upto 60 days. Degradation of alachlor was faster in acidic and basic water as compared to neutral water (Figure-3). No residues of alachlor were detected in acidic and basic water after 100 days. Further, the residues of alachlor were below detectable limit after 140 days in all the water samples fortified at 0.5 and 1.0 µg/g level. The half-life of alachlor in acidic, neutral and basic buffer solutions spiked at 0.5 and 1.0 µg/g was 22.65, 18.24; 24.23, 15.44 and 20.75, 19.04 days respectively.

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