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Effect of Repeated and Long Term Application of Butachlor on its Dissipation Kinetics in Rice Soil

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Influence of repeated and long term application of butachlor in rice-rice cropping system on butachlor persistence was examined by applying at 750 g/ha continuously for 12 years. Its dissipation under two sources of nitrogen (N) nutrition and seasons was studied by gas chromatography. Build up of butachlor residue in soil and the rate of dissipation over the years was compared in a block of four years during both kharif and rabi seasons separately. Dissipation of butachlor in soil followed first order kinetics and the degradation rate constant was ranged from 0.052-0.076 $days^{-1}$ for kharif and 0.060-0.093 $days^{-1}$ for rabi seasons. The half-lives of butachlor calculated were 9.1-13.3 days under 100 % inorganic N applied plots and 8.5-10.8 days under 75 % inorganic N + 25 % organic N applied plots irrespective of seasons. Residue of butachlor in rice grain and straw at harvest from both the treatments and across seasons was below the detectable limit of 0.001 µg/g. Bioaccumulation of butachlor residue in the crop produce was not found and showed the safety of the herbicide for weed control in transplanted rice.

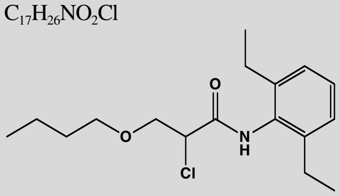
Keywords: Butachlor, Green manure, Nitrogen, Persistence, Continuous application, Puddled rice.

INTRODUCTION

In the present day agriculture where there is high demand and acute scarcity of farm labourers, the weed control with herbicides has become indispensable. Consequent to agronomic, sociologic and economic benefits derived through the chemical weed control strategy, the herbicide use in India has expanded widely. A number of herbicides have been registered and recommended for effective weed control in rice. Once the herbicides reach the soil environment, they undergo various reactions like photodecomposition, volatilization, leaching *etc.* depending on the soil and herbicide properties and climatic conditions [1,2]. Particularly the behaviour in the wetland rice field condition is generally complex and factors like temperature, pH, solar radiation, microbial activities, soil properties and agricultural practice might have influenced the pesticide dissipation [1,3,4]. The alternate oxidation and reduction processes in non-continuously flooded soils may favour degradation of pesticides [5]. Excessive, repeated and continuous use of the same herbicide may pose problems such as phytotoxicity to crop plants, residual effect on susceptible inter-crops, on succeeding crops or non target organisms and ultimately health hazards due to accumulation of residue into the soil, crop produce and ground water [6]. Repeated application of pesticide has also been reported to cause changes in the metabolic pattern of pesticide decomposition [7]. Residue buildup of pretilachlor and mefenacet due to their continuous use was not observed in Japan [8]. Butachlor (N-butoxymethyl-2-chloro-2,6-diethylacetanilide) is a pre-emergence herbicide, used extensively in puddled transplanted rice [1,9,10] for controlling the sedges and broad leaf weeds in transplanted and direct seeded paddy fields [11]. It belongs to chloracetamide group has the water solubility of 20 mg/L and Koc of 700 (WSSA, 2002) and other properties are given in Table-1. It quickly undergoes photo decomposition, microbial degradation and volatilization and did not pose serious problem of environmental pollution [1,12]. Under field conditions, it dissipates at faster rate and follows first order kinetics rate [13] and degrades quickly at pH 7.0 in 40 °C [14]. The half life of butachlor in rice fields was found to be about 0.8 to 5.5 days.

Integration of inorganic and organics source of nutrients for crop production is also a most significant factor influenced the herbicide behaviour in rice soils [15]. The residues of isoproturon, 2,4-D and butachlor in the soil under rice-wheat cropping was not build up when the organic matter was continuously applied for 5 years [16]. Mukherjee [17] used different organic amendments *viz.*, rice straw, farm yard manure, saw dust and charcoal and found farm yard manure was the most effective for the degradation of atrazine to the tune of 89.5 % within 60 days. Prakash *et al.* [18] reported that the adsorption of butachlor increased with increase in

TABLE-1
PHYSICO-CHEMICAL PROPERTIES OF THE HERBICIDE USED IN THE STUDY

Properties	Butachlor
Chemical name (IUPAC)	N-butoxymethyl-2-chloro-2',6'-diethylacetanilide
Empirical formula	$C_{17}H_{26}NO_2Cl$
Chemical structure	
Molecular weight	311.90
Water solubility	23 mg L^{-1} (24 °C)
Vapour pressure	2.4 $\times 10^{-1}$ mPa at 25 °C
Soil sorption coefficient (K_{oc})	700
pK_a	3.7
log K_{ow}	4.5
Henry's law constant at 20°C	1.54×10^{-6}

organic carbon content of soils with added animal manure. However, to our best of knowledge few informations on dissipation behaviour of butachlor in wetland ecosystem under puddled rice cultivation as influenced by continuous and repeated application and green manuring have been published in India and Asia. With this fact, the field experiment was conducted from kharif 2000 to 2012 to assess the dissipation kinetics and residue of butachlor in soil under rice-rice cropping system as influenced the source of nitrogen and seasons under continuous application.

EXPERIMENTAL

Field experiment: Field experiments with rice as test crop were conducted at the wet land farm of the Tamil Nadu Agricultural University, Coimbatore, India from 2000 to 2012. The experimental farm is located at 77°E 11°N latitude and 426 metre above mean sea level. Experimental field soil was fine clay loam in texture and belongs to Typic chromusterts group and noyyal soil series. The experimental field soil is medium in organic carbon status with the available nutrient status of low nitrogen, medium phosphorus and high potassium and has alkaline soil reaction and the electrical content of below 0.30 dS m^{-1}. The first crop of rice was planted during kharif season 2000 (sowing/planting from June to August months) and in rotation during rabi season (sowing/planting from September to November months) of every year. Field trials were carried out for 15 years with twenty eight rice crops in rice-rice cropping system during kharif and rabi seasons of 2000-12. The test chemical butachlor 50 % EC was purchased from the market and applied to the transplanted rice field of each crop as a pre emergence herbicide at the recommended level (0.75 kg/ha) under two sources of nitrogen (N) *viz.*, 100 % inorganic N through prilled urea and 75 % inorganic N as urea + 25 % organic N through green manuring with *Sesbania rostrata* for kharif season only. Control plot was sprayed with water without herbicide. Other agronomic and cultural practices were followed as recommended for the transplanted rice in the state crop production guide.

Climatic conditions: Weather parameters prevailed during the 13 years (kharif, 2000 to rabi, 2012) of experimentation were measured daily and the mean values during crop growing period was worked out and is given in Fig. 1.

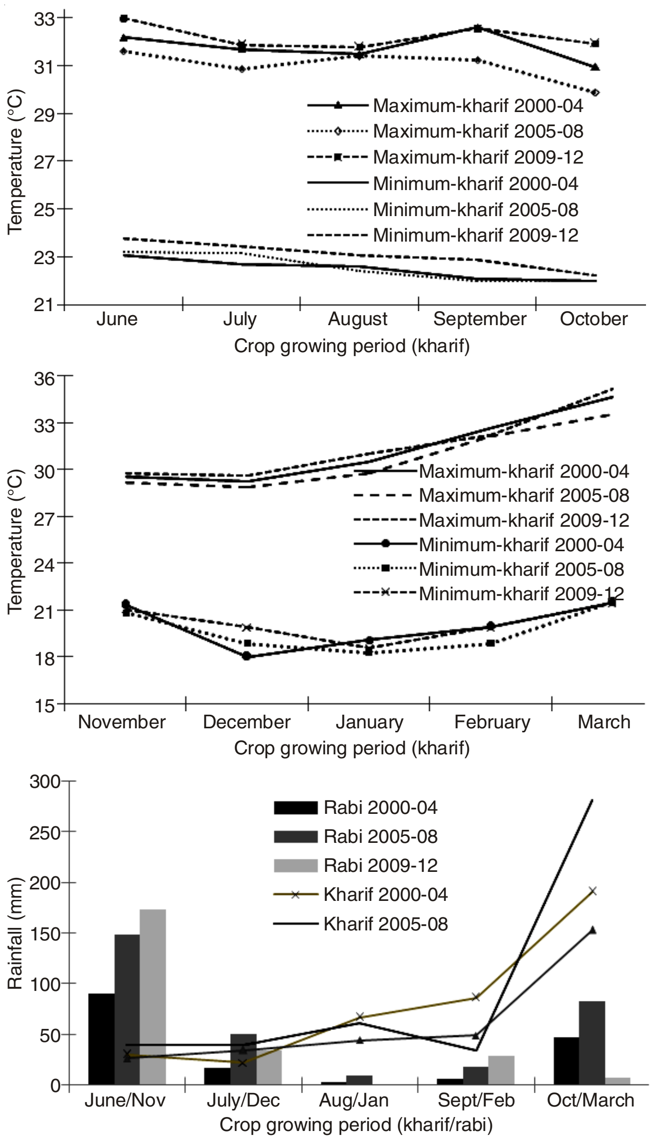


Fig 1. Weather parameters recorded during the crop growing period from 2000-2012

Collection of samples: Soil samples were collected periodically from 0 (2 h after herbicide application), 1, 7, 15, 30, 45, 60 days and at harvest. Two kilograms of five-soil cores each were randomly taken from all plots avoiding the outer 20 cm periphery of the plots using a soil auger up to a depth of 15 cm from the surface. Pebbles and other unwanted materials were removed manually. The cores from each plot were bulked together and reduced to 0.5 kg and stored in deep freezed condition until residue extraction. At harvest, 500 g of representative rice grains and straw samples were collected from herbicide-treated and untreated plots. The straw samples were cut into small pieces and air-dried. Rice grains and straw samples were then ground on a mechanical grinder and used for residue analysis.

Extraction, cleanup and instrumental determination: Butachlor was extracted from soil samples using acetone and the residue was eluted using dichloromethane. Clean up of herbicide residue was done using anhydrous sodium sulphate and florisil [19]. Dried residues were re-dissolved in known volume of hexane (HPLC grade) for injection into gas chromatograph. Recovery studies were carried out in order to establish the reliability of the analytical method and to know the efficiency of extraction and clean up steps employed for the present study before analyzing the unknown samples. The rice straw, grain and harvest soil samples from control treatment were fortified with the known concentration of butachlor analytical standard *viz.*, 0.001, 0.005, 0.01, 0.05 0.10, 0.5 and 1.00 ppm. After 2 h of fortification, different substrates were subjected to extraction and clean up to determine the recovery per cent of the applied butachlor from different matrices.

Data analysis: Rate of dissipation for butachlor in soil was calculated by linear regression from the transformed first-order rate equation, $\ln C_i = \ln C_0 - K_t$, where C_i is the butachlor concentration as a function of time in days (t), C_0 is the highest butachlor concentration and K is the degradation rate constant. The time of dissipation of 50 % (DT_{50}) of the highest concentration was calculated from the equation $DT_{50} = 0.693/K$.

RESULTS AND DISCUSSION

Recovery study: Quantification of butachlor residue was accomplished by comparing the peak height response for samples with peak height of the standard. Residue values obtained for the unknown samples were not corrected for recovery. Approximate retention time of butachlor in GC-ECD was 3.32 ± 0.2 min. Limit of detection (LOD) and limit of quantification (LOQ) of the butachlor was found to be 0.001 and 0.005 μg/g. Average recovery of butachlor in straw, grain and soil was found to be 88, 91 and 97 %, respectively. Method linearity was evaluated by the calibration curve of standard solutions and spiked soil samples at different concentration of butachlor. In all cases, coefficient of determination (R_2) was higher than 0.985**.

Build up of butachlor residue in soil: An application of butachlor was done as pre emergence to control weeds for both the rice crops in a system and was applied at 750 g ai ha^{-1} continuously for 12 years under two sources of N. Residues were monitored in soil until harvest of rice after its last application. The concentration of butachlor decreased progressively irrespective of sources of N (Figs. 2 and 3) and seasons with the advancement of crop growth. On 1st day, initial concentration of mean butachlor residue detected was ranged from 0.294 to 0.599 and 0.205 to 0.600 μg/g of soil respectively during kharif and rabi across different sources of N and years of experiments. During all the years, initial residue was high in 100 % inorganic N source (0.304 to 0.599 and 0.226 to 0.600 μg/g respectively for kharif and rabi seasons) than the 75 % inorganic + 25 % organic N source (0.295 to 0.528 and 0.205 to 0.522 μg/g respectively for kharif and rabi seasons) treatment. This could be attributed to the sorption of applied butachlor to the sites of binding in soil due to the adding up of organic matter through green manure in later treatment. The influence of organic matter on increasing the sorption of chloracetaniline herbicide pretilachlor in rice soil has also been reported by Janaki & Chinnusamy [19] and Armanpour & Bing [20].

Dissipation of butachlor in soil: Dissipation kinetics of butachlor in rice soil under continuous use during kharif and rabi seasons as influenced by the sources of nitrogen are presented in Figs. 2 and 3 and Tables 2 and 3. Since the study was conducted from 2000 to 2012 years, the data were pooled based on the weather data into three groups *viz.*, 2000-04, 2005-08 and 2009-12 and accordingly the dissipation of butachlor was discussed here.

The butachlor dissipated linearly from soil and in an average 38.7 and 47.8 % dissipation was observed during kharif and rabi respectively, on 7th day after last application (Tables 2 and 3). On 15th day, more than 50 % was dissipated from the soil irrespective of seasons and N sources and 90 % of the initial concentration dissipated from the soil on 45th day. On day 60, the residue becomes below detectable level of 0.001 μg/g. The enhanced degradation of butachlor was observed during kharif under 75 % inorganic + 25 % organic N treatment but not the case with rabi season and might be the result of enhanced microbial degradation as influenced by the

TABLE-2
DISSIPATION OF BUTACHLOR IN SOIL UNDER RICE-RICE CROPPING SYSTEM AS INFLUENCED BY THE NITROGEN SOURCES DURING KHARIF SEASON

Days after herbicide application	100 % inorganic			75 % Inorganic + 25 % Organic		
	2001-04	2005-08	2009-12	2001-04	2005-08	2009-12
1	–	–	–	–	–	–
7	41.9	36.1	34.9	39.1	40.2	40.0
15	60.0	53.7	57.6	62.5	51.7	63.0
30	85.6	82.9	81.6	86.1	82.0	85.4
45	98.7	94.6	95.1	97.8	94.9	96.3

TABLE-3
DISSIPATION OF BUTACHLOR IN SOIL UNDER RICE-RICE CROPPING SYSTEM AS INFLUENCED BY THE NITROGEN SOURCES DURING RABI SEASON

Days after herbicide application	100 % Inorganic			75 % Inorganic + 25 % Organic		
	2001-04	2005-08	2009-12	2001-04	2005-08	2009-12
1	–	–	–	–	–	–
7	42.0	52.1	48.5	39.8	52.6	51.9
15	47.1	54.9	78.1	44.6	54.7	76.8
30	84.7	78.2	88.5	80.7	78.6	89.8
45	98.7	94.6	95.1	97.8	94.9	96.3

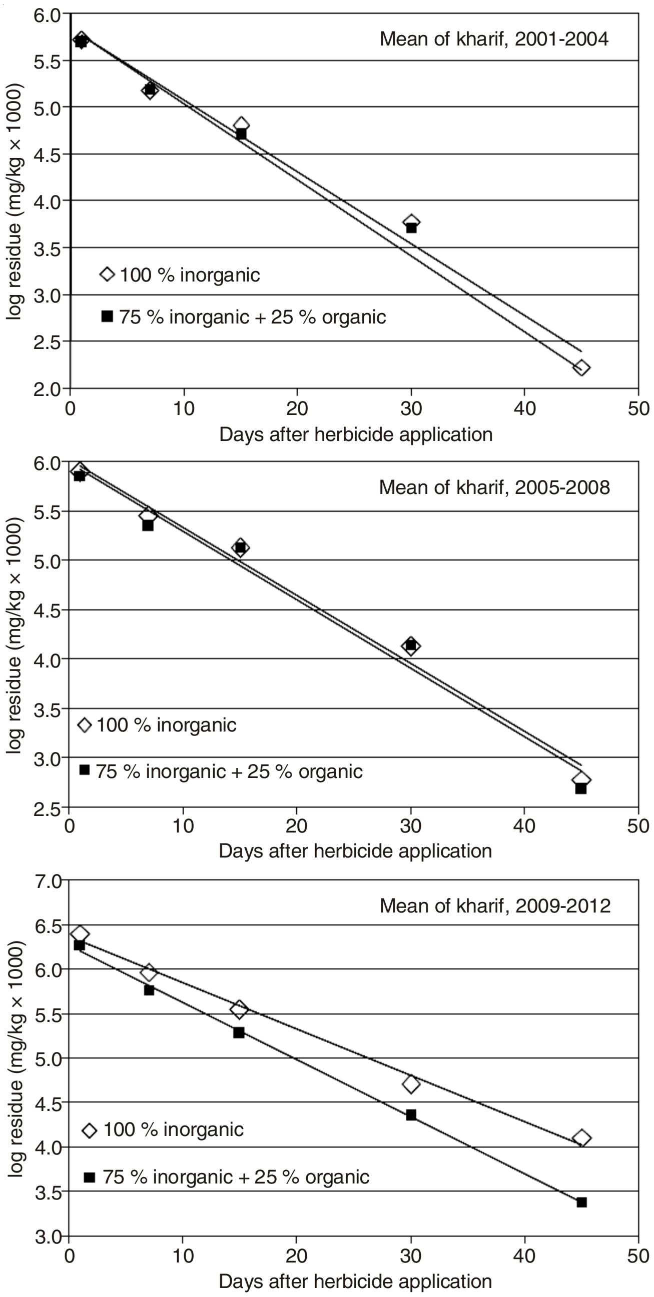


Fig 2. Dissipation kinetics of butachlor during kharif season in soil under continuous application as influenced by nitrogen sources

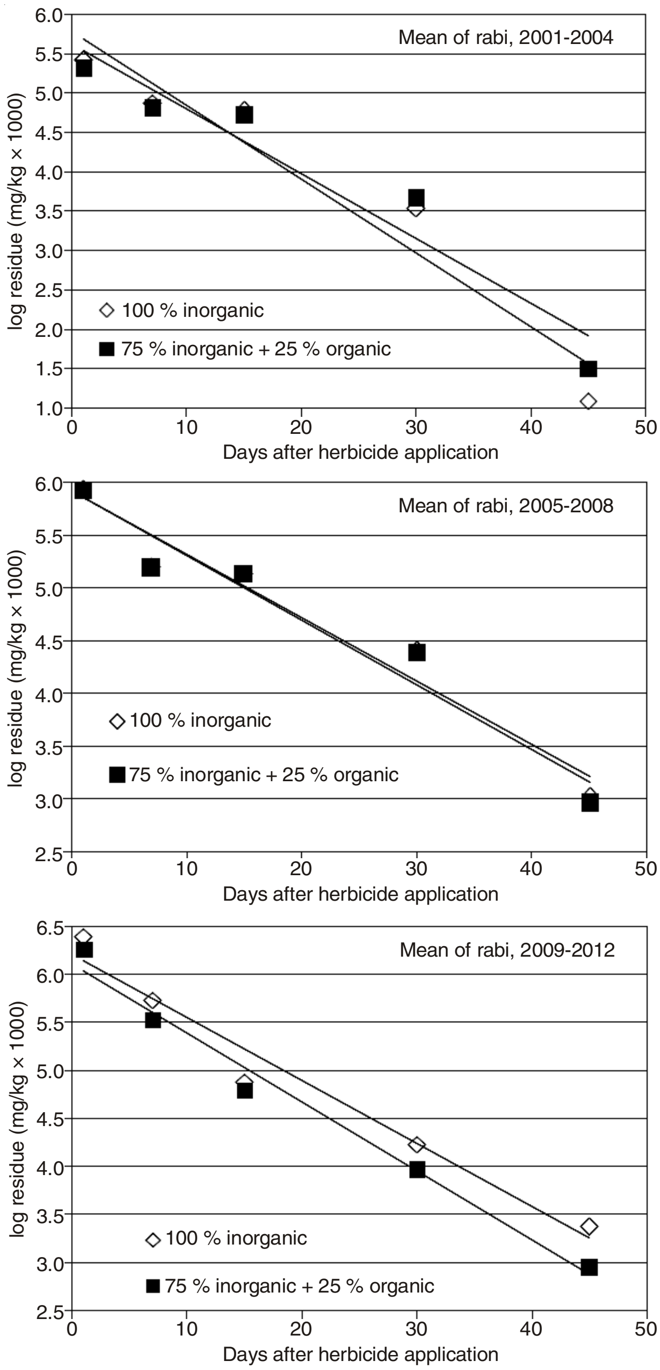


Fig 3. Dissipation kinetics of butachlor during rabi season in soil under continuous application as influenced by nitrogen sources

fresh organic matter addition during every kharif seasons [15]. Photo-decomposition could be the factor for the loss of butachlor residue from soil due to high temperatures prevailed in the initial periods of application during kharif seasons (Fig. 1) than rabi seasons. Chen [21] reported that the photo-decomposition at soil and water surfaces is also important, particularly when exposed to direct sunlight and found 95 % dissipation of butachlor after 24 h of exposure to light. In general the dissipation of butachlor was enhanced over the years irrespective of seasons (Tables 2 and 3) and N sources. When comparing the dissipation data of 2000-04 to 2005-08 and 2009-12, the dissipation of butachlor was enhanced and was more pronounced during rabi seasons than kharif seasons. It would be observed that the rate of dissipation on 15th day was increased from 2000-04 (45.8 %) to 2009-12 (77.4 %) during rabi seasons whereas in kharif seasons it was decreased from 2000-04 (61.3 %) to 2005-08 (52.7 %) and then increased during 2009-2012 (60.3 %). This could be attributed to the variation in climatic parameters (Fig. 1) especially the rainfall which was high during initial period (first month of cropping) of the butachlor application and also increased from 2000-12 with the mean annual rainfall of 362, 594 and 693 mm during 2000-04, 2005-08 and 2009-2012 respectively during rabi seasons as against of 120, 105 and 156 mm during 2000-04, 2005-08 and 2009-2012 respectively during kharif seasons. Higher initial rainfall during rabi might have diluted or washed the butachlor residue and enhanced the dissipation over a period of time. Decreasing and increasing trend of dissipation during kharif seasons also be attributed to the variations in rainfall received and the temperature conditions prevailed during the growing season (Fig. 1). Chen [21] also reported the influence of climatic conditions on butachlor degradation in rice soil. Janaki *et al*. [19] also reported the effect of climatic conditions on influencing the persistence of metamifop herbicide in rice soil.

The rate of disappearance of butachlor in soil from all the years followed first-order reaction kinetics under both the sources of N and seasons (Table-4) and found a good linearity between logarithmic concentration of butachlor residue and time (0.929 to 0.998**). Mean dissipation of butachlor during kharif was in accordance with the regression equation of y = 3.7508-0.0631x (r^2 = 0.999**) in 100 % inorganic source and y = 3.6998-0.0715x (r^2 = 0.991**) in 75 % inorganic + 25 % organic N source treatments. During rabi, it dissipates according to the kinetics of y = 5.9643-0.0719x (r^2 = 0.994**) in 100 % inorganic source and y = 5.9967-0.0692x (r^2 = 0.988**) in 75 % inorganic + 25 % organic N source. The degradation constant was high during rabi (0.052 to 0.076 $days^{-1}$) under 100 % inorganic N source than kharif (0.060 to 0.093 $days^{-1}$) and was similar in both the seasons under 75 % inorganic + 25 % organic N treatment (Table-4). Dharumarajan *et al.* [22] reported similar results of enhanced pretilachlor degradation in rice soil due to the application of green leaf manure.

The present research exposed that butachlor was comparatively more persistent in paddy soil during kharif than rabi season (Table-4). The mean DT_{50} values of butachlor ranged from 9.2-13.3 days and 7.5-11.6 days during kharif and rabi seasons respectively. The reason for variability was attributable to the changes in climatic conditions during crop growing period and Chen [21] also reported *in situ*, marked differences in half-life, from 0.8 to 5 days, due to the changes in climatic conditions like temperature and light intensity. However the higher half life in the present study could be attributed to the soil parameters and management conditions especially the clay content of the soil which enhanced the persistence by increasing the sorption of butachlor [23] and lowering the microbial degradation. Lower temperature at the later period of crop growth might have also enhanced its persistence in soil as reported by Kaur *et al*. [2]. Similar half life of 9 to 12 days for butachlor in rice soil under continuous application has been reported by Janaki *et al*. [9]. The half-lives of butachlor in the soils under different sources of N were calculated to be 9.1-13.3 days and 7.45-10.7 for kharif and rabi seasons respectively under 100 % inorganic N applied plots and in 8.6-10.8 days and 8.5-10.3 for kharif and rabi seasons respectively under 75 % inorganic N + 25 % organic N applied plots (Table-4). It was found that the application of 25 % green manure along with 75 % inorganic N, enhanced the rate of dissipation and has been confirmed by the decreased half life which could be the results of enhanced microbial activity and hastening of the redox potential drop by the decomposition of green manure in puddled rice soil. This was in line with the findings of Adhya *et al*. [24] and Chopra and Magu [25] for rice straw incorporation and Ferreria and Raghu [26] for green manure incorporation. The effect in the present study is more pronounced due to the low organic carbon status of the soil (0.53 %) as reported by the Castro and Yoshida [27]. When comparing the half lives of butachlor during different block of years, it was found that the half lives of butachlor was increased from 2000-04 to 2005-08 and then decreased to 2009-12 during rabi season. This could be the result of increased rainfall (Fig. 1) and high temperature prevailed during the initial period of butachlor application. Also the supply of 100 % N through inorganic source during every rabi season might have enhanced the dissipation and decreased the half life. However, during kharif season, the half life showed increasing trend from 2000-04 to 2009-12 (Table-4) irrespective of N sources. Since the rainfall was low and not much variation in temperature (Fig. 1) over the years during kharif, the rate of dissipation was slow down. This increased the half life through the build up of initial concentration of butachlor residues in soil due to its continuous and repeated application.

Harvest residue studies in rice grain and straw: Butachlor residues in rice grain and straw under both the N sources were below detectable level at harvest during all seasons which was well below maximum residue limit (MRL) set by FSSAI for butachlor (0.05 µg/g). Similar results were obtained by Sondhia [1,6] and Chinnusamy *et al.* [10] where butachlor residues were below MRL in rice at harvest.

Conclusion

Dissipation behaviour of butachlor in puddled transplanted rice soil followed first order reaction kinetics (R^2 greater than 0.93) irrespective of nitrogen sources and seasons. Continuous application of butachlor for 12 years showed build up in the initial concentration of its residue in soil for each season over years. The butachlor degradation was found to be highly manipulated by N supply partly through green manure, climatic conditions particularly rainfall, temperature and the soil

TABLE-4
REGRESSION EQUATION, CORRELATION COEFFICIENT, DEGRADATION CONSTANT AND HALF-LIFE OF BUTACHLOR IN SOIL UNDER RICE-RICE CROPPING SYSTEM AS INFLUENCED BY THE NITROGEN SOURCES DURING KHARIF AND RABI SEASONS

Sources of nitrogen	Kharif			Rabi		
	Regression equation	r^2	Half life (days)	Regression equation	r^2	Half life (days)
100 % inorganic nitrogen						
2001-2004	y = -0.076x + 5.837	0.984	9.12	y = -0.093x + 5.774	0.929	7.45
2005-2008	y = -0.069x + 6.023	0.986	10.04	y = -0.060x + 5.914	0.951	11.55
2009-2012	y = -0.052x + 6.360	0.992	13.33	y = -0.065x + 6.210	0.965	10.66
75 % Inorganic nitrogen + 25 % Organic nitrogen						
2001-2004	y = -0.081x + 5.845	0.981	8.56	y = -0.082x + 5.618	0.928	8.45
2005-2008	y = -0.072x + 5.985	0.976	9.17	y = -0.064x + 5.919	0.949	10.30
2009-2012	y = -0.064x + 6.266	0.998	10.83	y = -0.071x + 6.106	0.982	9.76

physico-chemical properties. In view of the fact that these factors differ considerably over years and seasons, the buildup of butachlor residue and its effective concentration in the soil will be varying significantly. Based on the findings, the butachlor at 0.75 kg/ha during both kharif and rabi seasons along with 25 % nitrogen through green manure for kharif season could be safely used as a pre-emergence herbicide in puddled transplanted rice in fine clay loam soils of the tropical regions. The residue in rice grain and straw at harvest was found below 0.001 μg/g established its safety to succeeding crop.

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