

## Effect of pH on the Persistence Behavior of the Insecticide Buprofezin in Water Under Laboratory Conditions

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Buprofezin (2-tert-butylimino-3-isopropyl-5-phenyl-perhydro-1,3,5-thiadiazin-4-one) developed by Nihon Nohyaku in 1981, is an insect growth regulator that acts on the insects by inhibiting chitin biosynthesis and integumentary cuticle deposition (Izawa *et al.* 1985; Uchida *et al.* 1985). This novel chitin synthesis inhibitor was recently introduced in the Indian subcontinent by Aventis Crop Science (I) Ltd.. Buprofezin is primarily active against homopterous insects, but its larvicidal activity extends to some coleoptera and acarina. It is effective against brown plant hopper in rice, whitefly in citrus, cotton and vegetables, scale insects and mealybugs in citrus (Tomlin, 1994). It is considered as a potential component in integrated pest management (IPM) programme due to its high degree of selectivity, low mammalian toxicity and safety to predators, parasites and other beneficial organisms. Although some information is available on the fate of buprofezin in soil (Funayama *et al.* 1986; Uchida *et al.* 1982) and also on the residue and persistence in crops (De Cock *et al.* 1990; Shuzhao *et al.* 1994), but little is known about its persistence behaviour in water. The present investigation has therefore, been intended to evaluate the effect of pH on the persistence behaviour of buprofezin in water under laboratory condition.

### MATERIALS AND METHODS

Analytical grade buprofezin (purity 99%) was obtained from Aventis Crop Science (I) Ltd.. Solvents such as dichloromethane, hexane, acetone were distilled in glass apparatus. Other chemicals used were of commercially available reagent grade.

The buffer solutions (Titrisol of pH-4.0, 7.0 and 9.0, E. Merck India Ltd.) were added @ 50 mL/500 mL of sterilized distilled water. Buffered water (200 mL) was taken in 250 mL wide mouthed flask in replication ( $R_1$ ,  $R_2$  and  $R_3$ ) and treatment wise ( $T_1$  and  $T_2$ ) to facilitate the sampling in required time. Buprofezin standard solution (in acetone) was added to the flasks to give a final concentration of 0.25  $\mu\text{g/mL}$  ( $T_1$ ) and 0.50  $\mu\text{g/mL}$  ( $T_2$ ). The flasks were topped with non adsorbent cotton plug and were incubated at 25°C in the dark. The untreated control experiment was also carried out simultaneously. Water samples were collected at various intervals (0, 3, 7, 15, 30, 45, 60, 90 and 120 d after

application of the pesticide) for analysis of buprofezin residues.

Water samples were extracted with dichloromethane (100 mL + 50 mL + 50 mL) using a separatory funnel after adding 10% sodium chloride solution (10 mL). The organic layers were combined, dried over anhydrous sodium sulphate and evaporated completely by rotary vacuum evaporator. The remaining residual portion was dissolved in distilled hexane. Distilled hexane fraction was concentrated to 5 mL volume for final analysis. GLC (Hewlett Packard Model 5890) equipped with an ECD ( $^{63}\text{Ni}$ ) and coupled with a HP-3392 A integrator was used for estimation of buprofezin residue. The capillary column (DB-1701) of 30 m length, 0.53 mm id and 1.5  $\mu\text{m}$  film thickness was used with  $\text{N}_2$  as the carrier (52 mL/min) gas. The oven, injector and detector temperatures were 230 $^\circ$ , 250 $^\circ$  and 275 $^\circ\text{C}$  respectively. Under these operating conditions of the GLC, the retention time of buprofezin was found to be at 2.64 min.

In order to evaluate the efficiency and reliability of the analytical method adopted, recovery study was carried out by fortifying water with analytical grade buprofezin at different levels (0.5  $\mu\text{g/mL}$ , 1  $\mu\text{g/mL}$  and 2  $\mu\text{g/mL}$ ). The average recovery was found to be in the range of 86.00 – 87.06%. The limit of detection for buprofezin in water was 0.01  $\mu\text{g/mL}$ .

## RESULTS AND DISCUSSION

The residues of buprofezin occurring in water (maintained at different pH) at different time intervals are presented in Table 1. The initial concentrations of buprofezin residue were found to vary from 0.212-0.217  $\mu\text{g/mL}$  for  $T_1$  and 0.435 – 0.438  $\mu\text{g/mL}$  for  $T_2$  in water of different pH. After 120 d of application, the residues remaining in water were found in the range of 0.084 – 0.101  $\mu\text{g/mL}$  for  $T_1$  and 0.182 – 0.206  $\mu\text{g/mL}$  for  $T_2$ .

The initial concentration of buprofezin dissipated about 9.43-16.58% within 15 d, which is increased gradually to 31.60-38.70% within 60 d and 52.64-61.29% within 120 d (Table 2). Comparatively higher dissipation was observed in water at pH 4.0 than that exhibited in water at pH 7.0 and 9.0 especially during the later stage of the experiment (from 30 d onwards).

The dissipation of buprofezin followed the first order reaction kinetics irrespective of pH condition of water and application dose (Figure 1).

The regression equations obtained from the residues of buprofezin in water under different pH conditions are presented in Table 3 along with the calculated half life values. The half life values were found to vary from 91.2 – 100.3 d for pH 4.0, 115.8 – 120.4 d for pH 7.0 and 111.5 – 115.8 d for pH 9.0. Therefore, the rate of dissipation of buprofezin was found to be maximum in water of pH 4.0 followed by pH 9.0 and pH 7.0.

Factorial analysis with interaction was followed to see the effect of two factors namely, pH and dose on the half-life values of buprofezin in water. The results are presented in Table 4. The mean half-life values were found to vary significantly at 5% level of significance due to pH. The mean half-life value for pH 4.0 (95.37 d) was significantly lower than those obtained for pH 7.0 and pH 9.0 while the mean half-life values due to pH 7.0 (116.67d) and pH 9.0 (112.37 d) appeared to be homogeneous. Moreover, the mean effect of application dose of buprofezin on its dissipation in water was also observed to differ significantly. The mean half-life value exhibited by T<sub>1</sub> (104.99 d) was significantly lower in comparison to that exhibited by T<sub>2</sub> (111.28 d).

**Table 1.** Persistence of buprofezin in water of different pH.

Dose	pH	Concentration* remaining in water (µg/mL) at different d								
		0	3	7	15	30	45	60	90	120
0.25 µg/mL (T <sub>1</sub> )	4.0	0.217	0.204	0.198	0.181	0.171	0.149	0.133	0.105	0.084
	7.0	0.216	0.207	0.194	0.187	0.178	0.158	0.145	0.121	0.101
	9.0	0.212	0.209	0.204	0.192	0.176	0.159	0.145	0.120	0.100
0.50 µg/mL (T <sub>2</sub> )	4.0	0.438	0.414	0.384	0.366	0.348	0.306	0.276	0.224	0.182
	7.0	0.437	0.397	0.384	0.369	0.353	0.317	0.291	0.244	0.206
	9.0	0.435	0.422	0.396	0.377	0.361	0.322	0.295	0.246	0.206

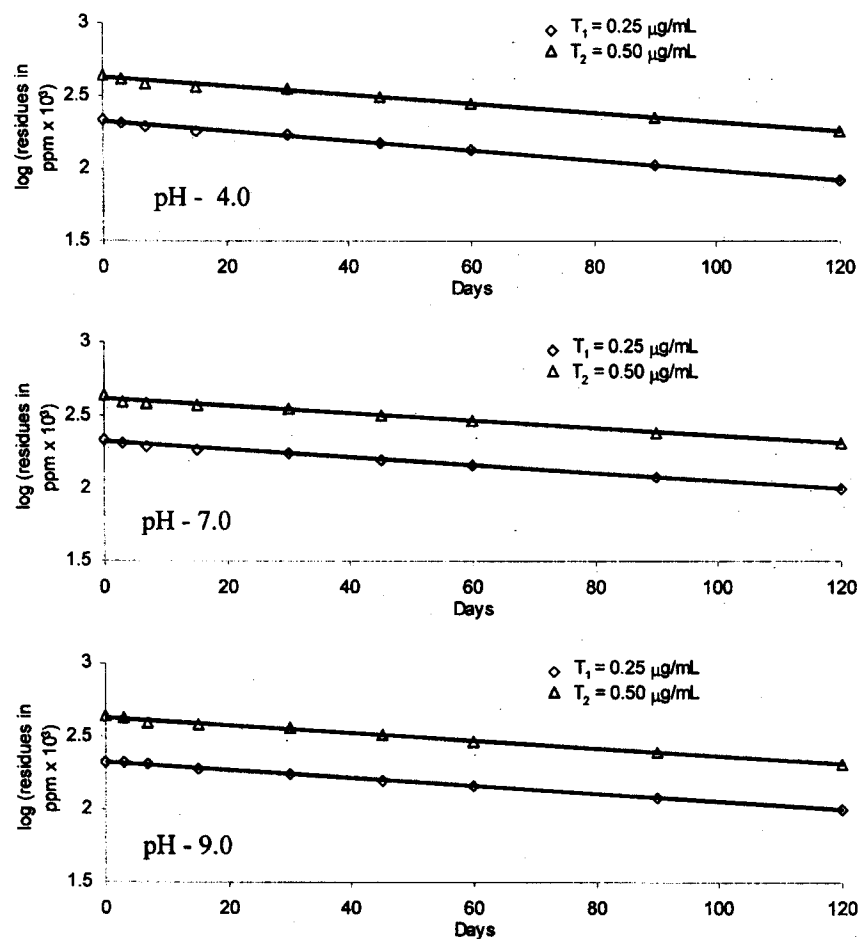
\* values are average of three replications

**Table 2.** Percentage dissipation of buprofezin in water of different pH.

Dose	pH	Percentage dissipation								
		0	3	7	15	30	45	60	90	120
0.25 µg/mL (T <sub>1</sub> )	4.0	-	5.99	8.75	16.58	21.19	31.33	38.70	51.61	61.29
	7.0	-	4.16	10.18	13.42	17.59	26.85	32.87	43.98	53.24
	9.0	-	1.42	3.77	9.43	16.98	25.00	31.60	43.39	52.83
0.50 µg/mL (T <sub>2</sub> )	4.0	-	5.47	12.32	16.43	20.54	30.13	36.98	48.85	58.44
	7.0	-	9.15	12.12	15.56	19.22	27.45	33.40	44.16	52.86
	9.0	-	2.98	8.96	13.33	17.01	25.97	32.18	43.44	52.64

**Table 3.** Statistical interpretation of the residual data.

Dose	pH	Residual half life (RL <sub>50</sub> or t <sub>1/2</sub> ) [d]	Regression equation
T <sub>1</sub> (0.25 µg/mL)	4.0	91.22	Y = 2.323 - 0.0033x
	7.0	115.78	Y = 2.321 - 0.0026x
	9.0	111.49	Y = 2.327 - 0.0027x
T <sub>2</sub> (0.50 µg/mL)	4.0	100.34	Y = 2.620 - 0.0031x
	7.0	120.41	Y = 2.615 - 0.0025x
	9.0	115.78	Y = 2.628 - 0.0026x



**Figure 1.** Linear plot for first order dissipation reaction of buprofezin in water at different pH.

**Table 4.** Effect of pH and dose on the half life value of buprofezin.

pH	Half life value (d) of buprofezin for different doses		Mean
	T <sub>1</sub> (0.25 µg/mL)	T <sub>2</sub> (0.50 µg/mL)	
4.0	90.33	100.42	95.37
7.0	114.47	118.87	116.67
9.0	110.17	114.57	112.37
Mean	104.99	111.28	108.14
SEm (±) CD (0.05)	Dose	pH	Interaction
	1.328	1.627	2.30
	4.091	5.013	Not significant

Therefore, the persistence of buprofezin was found to be affected by both dose and pH condition of water. The order of dissipation obtained as  $T_1 > T_2$  and  $\text{pH } 4.0 > \text{pH } 7.0 \cong \text{pH } 9.0$ .

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