Bull. Environ. Contam. Toxicol. (2006) 76:930–934

DOI: 10.1007/s00128-006-1007-x

Environmental Contamination and Toxicology

Persistence and Movement of Paclobutrazol Residues in a Mango Orchard Soil

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Received: 30 September 2005/Accepted: 20 April 2006

Mango, one of the most important fruit crops of India, suffers from alternate bearing habit which reduces its productivity. Paclobutrazol (β - [(4-chlorophenyl) methyl] - α - (1-1-dimethylethyl)-1H-1,2,4-triazole-1-ethanol] is a plant growth regulator, that has been primarily used in India to counter this alternate bearing habit of mango , especially the distinctly alternate bearing 'Alphonso' mango (Rao and Srihari, 1998; Hoda et. al., 2001). It may be applied to mango either as a foliar spray or as a soil drench in the root zone or collar region of mango tree but studies have found that the soil drench application is most effective (Pujari et. al.,1999). Soil persistence of paclobutrazol is known to be high, however, the vertical mobility of paclobutrazol residues in soil has not been studied earlier either in laboratory conditions or in the field. The present study was therefore carried out to determine the persistence and mobility of paclobutrazol residues in soil following its soil application in mango orchards from the point of environmental significance of such residues.

MATERIALS AND METHODS

Paclobutrazol, analytical standard was obtained from M/s. Riedel-de-Hain, Germany. The formulation used viz. Cultar 23 SC was procured from Bangalore market. Organic solvents were obtained from M/s. Qualigens Fine Chemicals, India. Florisil was obtained from M/s.Sigma Chemical Co., U.S.A.

A field experiment was conducted at a mango orchard (50m x 50m) consisting of fifty 5 year old mango trees (cv. Totapuri) located at IIHR farm, Hessaraghatta, Bangalore. Paclobutrazol was applied as Cultar-23 SC, to the tree basin soil (under the canopy) by making a 5cm deep and 15 cm wide furrow in the soil at a radial distance of 60 cm from the tree trunk. Trees in triplicate were maintained for each of the treatments, viz. 5g and 10g a.i. per tree. The required quantity of Cultar 23SC was mixed with 5 litres of water and then applied at the above furrow in the soil by line pour method for uniform application. The trees were irrigated every day for 15 days after application. The physico-chemical properties of the soil is listed in Table1. Soil samples were drawn in triplicate from different soil depths viz. 0-15 cm, 15 – 30 cm, 30-45 cm and 45-60 cm at 1 hour after application (0 day) and thereafter at 5, 10, 15, 30, 60, 90, 120, 150, 180 and 210 days using a mechanical soil auger.

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Table 1. Physico-chemical properties of soil.

S.No.	Parameters	
1	Texture –Clay %	23.50
	- Silt %	27.40
	- Sand %	49.10
2	Textural Class	Sandy loam
	Taxonomic classification	Kandic haplustalfs
3	Organic carbon (%)	0.5
4	pH (1 : 2.5)	7.09
5	Electrical Conductivity dSm^{-1}	0.19
6	Cation exchange capacity (cmol (p^+) kg^{-1})	13.88

The residue analytical method followed was modified from that of Stahly and Buchanan, 1986. 50 g soil sample was taken in an Erlenmayer flask and extracted with 70 mL chilled methanol using a rotary mechanical shaker for 1 hour at 200 rpm. The extract was filtered using a Buchner Funnel. The soil was re-extracted along with the filter paper with 2 x 50 mL chilled methanol. The extracts were filtered and all the filtrates combined. The combined filtrate was evaporated to aqueous phase in a rotary evaporator at 35°C. The pH of the extract was adjusted to 11 with 1N sodium hydroxide solution. The aqueous extract thus obtained was partitioned against 3 x 50 mL of dichloromethane in a 500 mL separating funnel. The lower dichloromethane layers were collected each time, combined and passed through anhydrous sodium sulphate. This combined extract was evaporated under vacuum to 5 mL for further clean-up step.

A glass column (50 cm x 1.3 cm) was packed with 5 g activated florisil in diethyl ether. The above extract in dichloromethane was added slowly to the column. The column was eluted with 10 mL of diethyl ether + methanol (97+3 v/v), the eluate collected and evaporated to dryness in a rotary vacuum evaporator. The residues were immediately re-dissolved in 5mL of double-distilled AR grade acetone and determined by a Varian Model 3800 Gas Liquid Chromatograph (GLC) equipped with a thermionic sensitive detector (TSD). The column used was Varian CPSil 8CB (30 m x 0.53 mm, df = 0.25μ) and the column oven, injector oven and detector temperatures were 210°C, 250°C and 300°C respectively. The gas flow rates were 7.6 mL/min (nitrogen), 4 mL/min (hydrogen) and 175 mL/min (zero air). The TSD bead current was set at 3.0 amp. The retention time of paclobutrazol at the above GLC parameters was 13.2 min. The limit of detection of paclobutrazol at signal to noise ratio of 5:1 and the limit of quantification (LOQ) of the residues in soil was 0.01 μg/g Recovery study of paclobutrazol was carried out in soil by adding 0.5 or 1 mL of 5 μg g^{-1} paclobutrazol standard solution (fortification level = 0.01 to 0.1μg g^{-1}), to 50 g soil.

RESULTS AND DISCUSSION

The per cent mean recovery of paclobutrazol residues from soil at fortification levels of 0.01 and 0.1 μg g^{-1} was 85.4 to 89.2 per cent. The data on residues of paclobutrazol found at different soil depths at various sampling intervals from 0 to 210 days is given in Tables 2 and 3. The residues of paclobutrazol in surface soil

Table 2. Paclobutrazol residues at different soil depths following its treatment at 5g a.i./tree.

DAT	D1	D2	D3	D4
0(1hr)	1.77 (±0.13)	0.89 (±0.07)	1.99 (±0.06)	0.65 (±0.09)
5	1.01 (±0.01)	1.05 (±0.04)	0.45 (±0.04)	1.27 (±0.08)
10	0.86 (±0.10)	0.51 (±0.02)	0.43 (±0.03)	1.29 (±0.04)
15	0.72 (±0.07)	1.15 (±0.10)	0.26 (±0.01)	0.07 (±0.01)
30	0.52 (±0.00)	1.05 (±0.02)	0.10 (±0.00)	0.07 (±0.01)
60	0.37 (±0.01)	0.38 (±0.01)	0.72 (±0.00)	0.08 (±0.01)
90	0.13 (±0.13)	0.24 (±0.02)	0.39 (±0.02)	0.36 (±0.04)
120	0.08 (±0.01)	0.03 (±0.00)	0.18 (±0.01)	0.06 (±0.01)
150	0.05 (±0.00)	0.11 (±0.00)	0.09 (±0.01)	0.04 (±0.00)
180	0.02 (±0.00)	0.02 (±0.00)	0.06 (±0.01)	BDL (±0.00)
210	0.01 (±0.00)	BDL (±0.00)	BDL (±0.00)	BDL (±0.00)

Table 3. Paclobutrazol residues at different soil depths following its treatment at 10g a.i./tree.

DAT	D1	D2	D3	D4
0(1hr)	4.87 (±0.07)*	4.33 (±0.02)	1.82 (±0.03)	0.38 (±0.02)
5	3.03 (±0.02)	3.99 (±0.02)	1.22 (±0.02)	0.47 (±0.07)
10	2.99 (±0.04)	2.96 (±0.04)	0.95 (±0.05)	0.12 (±0.02)
15	2.60 (±0.13)	2.48 (±0.02)	0.37 (±0.02)	0.05 (±0.00)
30	2.28 (±0.02)	0.27 (±0.02)	0.45 (±0.03)	0.21 (±0.02)
60	2.19 (±0.04)	0.14 (±0.02)	0.39 (±0.04)	0.47 (±0.06)
90	1.84 (±0.02)	0.30 (±0.08)	0.14 (±0.02)	0.07 (±0.01)
120	0.93 (±0.06)	0.11 (±0.01)	0.52 (±0.02)	0.04 (±0.00)
150	0.34 (±0.02)	0.53 (±0.03)	0.60 (±0.03)	0.03 (±0.00)
180	0.07 (±0.00)	0.04 (±0.00)	0.02 (±0.00)	0.02 (±0.00)
210	0.01 (±0.00)	0.01 (±0.00)	0.01 (±0.00)	BDL (±0.00)

D1(Depth 1)= 0-15cm, D2(Depth 2) =15-30cm, D3(Depth 3) = 30-45cm, D4(Depth 4)= 45 -60cm.
DAT = Days after treatment. 0(1hr) = one hour after paclobutrazol application.
*The figures in parenthesis represent the SD values

or soil depth 1 (D_1, 0-15 cm), decreased gradually with time from 1.77 and 4.87 μg g^{-1} at 0 day after treatment to 0.37 and 2.19 μg g^{-1} at 60 days and just above the detectable level (0.01 μg g^{-1}) at 210 days at both the treatment concentrations. Almost 50% of the residues degraded within 10 and 30 days from the lower and higher treatments respectively but the remaining 50% dissipated very slowly over a period of next 180 – 210 days. Dissipation kinetics of paclobutrazol residues in surface soil is presented in Table 4. The data on persistence of paclobutrazol residues in D_1 and the regression coefficients (-0.95 and – 0.90) indicated that the degradation of paclobutrazol residues with time followed first order negative exponential equation $y = a.e^{-bx}$ where x (days after application) and y (residues in ppm) are the independent and dependant variables, a and b are constants. The half life of paclobutrazol residues in soil at D_1 varied from 30.7 to 29.7 days from the lower and higher treatment concentrations respectively. Although the half life of residues following either treatment concentration were similar, the time required

for residues to reach below detectable limit (T_{BDL}) was 216.7 and 275.1 days from the two treatments respectively.

The coefficients of regression (r) values (– 0.95 and – 0.90) indicated high negative correlation of the data to the first order equation. The initial residues of paclobutrazol at soil depth 2 (D_2, 15-30 cm), were 0.89 and 4.33 ug g^{-1} from the lower and higher treatment concentrations respectively (Tables 2 and 3). The residues from treatment 1(lower concentration) increased up to 15 days to 1.15 ug g^{-1} with an interim decrease to 0.51 µg g^{-1} at 10 days. Thereafter, the residue levels reduced gradually from 30th day onwards till it was below detectable level at 210 days. The residues from treatment 2 (higher concentration) decreased from 4.33 ug g^{-1} at 0 day to 0.4 µg g^{-1} at 60 days but thereafter increased at 90 days after treatment. It was clear that the leaching of residues from upper soil layer and degradation of the residues were taking place simultaneously in this layer due to which the residues did not always reduce with time. At soil depth 3 (D_3, 30– 45 cm.), the initial residues were higher than those at depth 2 (1.99 and 1.82 ug g^{-1} from the lower and higher treatment concentrations respectively) which gradually reduced to levels below or just above detectable levels at 210 days. There were inter-mittant increases at 60 days from the lower concentration and at 30, 120 and 150 days from the higher treatment concentration respectively. At soil depth 4 (D_4, 45– 60 cm.) the residues at all the sampling intervals were comparatively lower than at other depths (Tables 2 and 3) with the exception of residues at 5 and 10 days after treatment from the lower treatment concentration which were 1.27 and 1.29 ug g^{-1} respectively. The paclobutrazol residues from both the treatments (5 and 10 g a.i. per tree) persisted at all soil depths for at least 150 days. The residues were therefore highly persistent which dissipated with half lives of 29-31 days in the surface soil (0-15 cm depth). Paclobutrazol is known to be stable to degradation in the pH range of 4 to 9 (Roberts, 1996). The experimental results from the present study are in agreement with this. The pH of the soil in this study was 7.09.

Table 4. Regression equation and half-life for first order dissipation of paclobutrazol in surface soil.

S. No.	Treatment	Regression Equation	Half-life ($t_{½}$) days	T_{BDL} days	Regression Coefficient (r)
1	T_1 (5 g a.i /tree)	Y= 0.08-0.01X	30.70	216.78	-0.95
2	T_2 (10 g a.i /tree)	Y= 0.71-0.01X	29.67	275.14	-0.90

Persistence of paclobutrazol in soil has been earlier estimated to be high by indicator plant (*Vicia faba*) bioassay. Paclobutrazol residues have been thus found in soils of sweet cherry (Jacyna and Dodds, 1999) and apricot (Jacyna and Dodds, 1995) orchards for 3 years and 20 months respectively. However since the residues were not estimated analytically in the above studies they could not be quantified. The residues of paclobutrazol have also been reported to remain for upto 11 months (Subhadrabandhu et. al., 1999) at physiologically significant levels following its soil application in mango orchards. The results from the present study established that paclobutrazol residues quickly leached vertically down the soil. The residues persisted at all depths upto 60 cm. for at least 150 days at levels equal

to or above 0.01 ug g^{-1}. Thus the residues were highly persistent as well as mobile. The mobility of paclobutrazol to lower soil depths probably does not interfere with its uptake by mango tree as it has a very deep root system. However, the high mobility of paclobutrazol residues down the soil may result in contamination of ground water sources especially in areas where the water table is high. Since the surface (0-15 cm.) soil residues of paclobutrazol also persist for a long time, these residues may contaminate nearby water bodies through surface run off.

Acknowledgments. We thank the Director, Indian Institute of Horticultural Research, Bangalore, for providing facilities for this study.

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