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Research Article

ADSORPTION, DESORPTION AND MOVEMENT OF ENDOSULFAN IN AGRICULTURAL SOIL

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ABSTRACT

The effect of soil properties like adsorption, desorption, movement and distribution of endosulfan (1, 2, 3, 4, 7, 7-hexachlorobicyclo (2, 2, 1)-2-heptene-5, 6-bisoxymethlene sulfite) in two tropical soils, sandy loam and sandy clay soil from Mysore, Karnataka state were studied respectively. The results indicated that soils with different physico-chemical properties have different effects on the adsorption, movement and distribution of pesticide, especially at higher concentration levels. Two composite soil samples were collected that having diversified physico-chemical properties were analysed. Adsorption coefficient values (K_{ads}) as described by Freundlich's model ranged from 0.04 to 0.31. Higher adsorption coefficient was measured in Halekesare soils as compared to Hinkal soils. Desorption as attributed to hysteresis was also measured. Desorption coefficient values (K_{des}) were higher than adsorption (K_{ads}) in each study. Movement of endosulfan was restricted to subsurface soil upto 0-15 cm depth only in both the soil types. However, vertical movement of endosulfan was faster in sandy loam soil than sandy clay soil. Results indicated that applied endosulfan retained in the upper layers of soil would be persistent or slow to degrade in the soils studied.

Keywords: *Absorption, Desorption, Endosulfan, Movement, Soil*

INTRODUCTION

Agriculture is the main occupation in India. Most synthetic pesticides applied to field agro ecosystems are xenobiotic substance (Munneckae, 1982). Persistent Organic Pollutants (POPs) are a set of chemicals that are toxic; persist in the environment for long periods of time, and biomagnify as they move up through the food chain. POPs have been linked to adverse effects on human health and animals, such as cancer, damage to the nervous, reproductive disorders, and disruption of the immune system. Because they circulate globally via the atmosphere, oceans and other pathways. POPs r leased in one part of the world can travel to regions far from their source of origin (Sandra *et al*. 2006). Organochlorines (OCs) represent an important group of POPs which have caused worldwide concern as toxic environmental contaminants (Law *et al*. 2003, Covacia *et al*., 2005) and (Wurl and Obbard, 2005). The fate of pesticides in soils is a great environmental concern due to their toxicity, persistence and bioaccumulation. The retention of pesticides in the environment involves complex mechanisms that are influenced by many processes, including volatilization, leaching, adsorption, as well as chemical and biological degradation. Pesticide characteristics (water solubility, tendency to adsorb to the soil and pesticide persistence) and soil characteristics (clay and organic matter) are important in determining the fate of the chemicals in environment (Anonymous, 2009; Pignatello, 1989; Becker *et al.*, 2011) and having most important processes affecting the mobility, persistence, bioactivity, toxicity and efficacy of pesticides in the soil environment. Retention is influenced by pesticide adsorption by soil components. Pesticides which were used to control insect pests and weeds will ultimately reach soil and thus contaminate food and environmental system (Deo *et al*, 1994, Matsumura, 1982). Endosulfan, a chlorinated organochlorine insecticide is used to control various pests in fruits, vegetables and other agricultural commodities. Most of the Cyclodiene insecticides have been banned because of their persistence, bioaccumulatability and broad-spectrum activity in both target pest and non-target organisms (Katpal *et al*, 1997). The use of this chemical is banned in India and as a result of earlier application, the fate of endosulfan in the environment and its effects on the organisms in tropical ecosystem is of great concern. Properties of chemicals, soil characteristics and environmental conditions will have influence on the fate of chemicals in the environment (Hsu and Bartha, 1979). Environmental conditions such as temperature and moisture also play roles. A number of soil properties can influence the retention and mobility of insecticide in a soil profile (Munnecke *et al.*, 1982). They include organic matter content, soil texture, soil acidity, iron (Fe) and manganese (Mn) oxide and clay content. Hysteresis effects are often observed in pesticide adsorption-desorption studies (Koskinen and Harpper, 1990). There are several causes for hysteresis effects. Among these are, artifacts created by the methodology; failure to establish complete equilibrium during adsorption; non equilibrium caused by the exposure of new sorption sites resulting from the abrasion of soil and slow diffusion to sites within the soil organic matter; changes over time in binding mechanisms of the pesticide to the soil; affecting exchange reaction, and formation of precipitates. Centrifugation suspension step in batch-type adsorption-desorption method could also be responsible. Losses of pesticide by volatilization, chemical and/or microbial transformation of the pesticide during the experiment can also cause hysteresis (Gan *et al.*, 1996; Hamaker and Thomson, 1972; Koskinen *et al*., 1979; Locke, 1992*)*. The significant amount of organic matter in examined soils and the chemical characteristics of the pesticides are largely responsible for their adsorption and consequent movement and distribution through soil.

In this study, we focused on studying movement, adsorption and desorption characteristics of endosulfan on two types of soils though they are of fundamental importance to quantify the transport of pesticides and the selection of proper remediation technique. The importance of organic matter, particle size, as well as pH of the soil for sorption has been emphasized by many workers (Huang and Mckercher, 1984, Barriuso *et al*., 1992; Gao *et al*., 1998). These factors however, have not been studied in details for endosulfan, which was used widely in agriculture in India. Therefore, investigation of these processes will provide a better understanding of its sorption and transport in soil environments. With the above context, the aimed at to this study the effect of these soil properties on endosulfan in organic rich soils of Mysore area.

MATERIALS AND METHODS

Soil Samples

Two representations of soils sandy loam and sandy clay soil from Mysore, India were selected for use in this study. Soil samples used in this study were collected from 0-10 cm and 10-20 cm depth from paddy fields around Hinkal and Halekesare area of Mysore district. The samples were air dried and sieved through 2 mm and stored in cold room until use. Samples were characterized using standard methods for pH, partical size, organic matter, nitrogen and phosphorous content.

Chemicals

Analytical and technical grades of endosulfan were procured from Excel Industries (India). Endosulfan formulation (35 % EC) was procured from Cheminova India Pvt Ltd. All other solvents used were of analytical grade.

Materials for ELISA

Bovine Serum Albumin, Horse Radish Peroxidase, Tween20, analytical standards of endosulfan was obtained from M/S Sigma Chemicals, St Louis. Polystyrene 96-microwell plates were purchased from Nunc, Roskilde, Denmark. Solvents used were of analytical grade and purchased locally.

Leaching and mobility studies in columns

Leaching and mobility studies of the technical endosulfan in Hinkal and Halekesare soil of paddy field were carried out using soil columns in the laboratory. Air-dried soil samples in triplicates were filled in PVC column (2.5mm thickness, 15 mm diameter, 50 cm length). The soil columns were mounted vertically on iron stands. The inner wall of the columns was lined using aluminum foil to avoid absorption of pesticide by column. Prior to applying the endosulfan, the soil columns were saturated with distilled water. Stock solutions of endosulfan 5 ppm and 10 ppm concentrations were applied on top of the column. The column was periodically washed with distilled water. Leachate samples were collected periodically in glass tubes and stored in cold room until analysis.

In order to study the downward movement of endosulfan in soil columns, residues of the chemical was determined from 0-10, 10-20, 20-30, and 30-40 cm soil depth after 30 days of the experiment.

Adsorption and desorption studies

Adsorption-Desorption of the surface soil samples were conducted using batch equilibrium technique. Duplicates of each 10 g soil sample containing 10 mL of 0.01N $cacl_2$ and solution of endosulfan with a concentration of 3, 5, 7 and 10 ppm were separately centrifuged and shaken at room temperature for 24h. Sampling was done at different time intervals ranging from 10 min to 24 h. then centrifuged at 5000 rpm for 10 min in refrigerated centrifuge. The superannuated layer was analysed for the presence of endosulfan by Enzyme Linked Immuno Sorbent Assay (ELISA) method. The amount of endosulfan sorbed by soils was calculated by subtracting initial concentration from amount left in aqueous solution following equilibration. The adsorption isotherm as described by Freundlich equation was employed in this study, in which, (at equilibrium) endosulfan sorbed by soil and left in soil solution is as follows: where; x/m is amount sorbed by soils, mg/g Ce is equilibrating concentration in aqueous solution, ng/mL *Kads* is an adsorption constant (Freundlich adsorption coefficient) 1/n is constant (slope) providing an estimate of the intensity of adsorption, when the 1/n value approaching to 1 implies that adsorption curve is close to linear (Chiou *et al.*, 1983; Hamaker and Thomson, 1972; Pignatello and Huang, 1991).

Desorptions were conducted by measuring successive dissolution of the sorbed material from soil using deionized distilled water. Following equilibration and centrifugation, 5 mL of aqueous solution was drawn from a soil solution and replaced with the same amount of water. The soil solution was resuspended in 0.01N $cacl_2$ with a vortex mixer and shaken again to establish a new equilibration. One mL of aqueous solution was withdrawn and analyzed after extraction with suitable solvent using ELISA method. Desorption isotherms were constructed using the Freundlich equation.

Preparation of Standard Stock Solution for Endosulfan

Authentic stock solution was prepared by dissolving analytical grade endosulfan to get a concentration of 1mg/ml in methanol (1000 ppm). From 1000 ppm stock, 100 µl of pesticide was made up to 5 ml to get 20 ppm. The 20 ppm solution was serially diluted in borosilicate tubes using 0.1% Fish skin gelatin phosphate buffer to get endosulfan in the range of 0.1, 0.4, 1.2, 3.7, 11.1, 33.3, 100 ppb.

ELISA Procedure

The antibodies and pesticides conjugates used in this assay have been described earlier (Karanth *et al.* 1999). The antibodies were diluted in 50 mM carbonate buffer (pH 9.6) and were coated to get 1µg/well and kept overnight at room temperature ($22^{0\ \pm}\ 4^{0}$C). The microwell plates were washed twice with PBS (50 millimolar (mM) sodium phosphate buffer, pH 7.2) containing 0.05% (v/v) tween20 (PBS/T) and then blocked with 150 µl BSA (1%bovine serum albumin) in PBS. 100 µl of pesticide standard containing 0.1% Fish skin gelatin and 100 µl of Horse Radish Peroxidase were loaded and incubated for 1hr. There after the contents were removed. Plates were washed twice with PBS/T. 150 µl hydrogen peroxide substrate / chromogen were added and incubated for 30 min at room temperature. Finally, colour development was stopped by adding 50 µl of 1.25N sulfuric acid and the color intensity was read at 450 nm using ELISA automatic reader Model Elx 800 Bio-Tek instrument

RESULTS AND DISCUSSION

Physico-Chemical Properties of Soils

The physico-chemical properties of soils such as particle size and texture classification of soils are shown in Table 1, 2 and 3. The data indicated that Halekesare soil was sandy loam in texture with a clay content of 17.6% in surface and 16.0% in subsurface soil. Hinkal soil was sandy clay loam in texture with clay content of 27.6% in surface soil and 19.45 in subsurface soil respectively. The field moisture content was lowest (7.7% in Hinkal surface soil as compared to Halekesare surface soil (12%). The bulk density of both the soil types was 2.6g/cm. Organic matter content was highest in Hinkal (22.6mg/g) compared to Halekesare (18.21mg/g). However, the variations in soil pH at both the sites were found to be marginal.

Table 1: Particle size distribution, texture classification and endosulfan detection of soils

Parameters	**Hinkal**		**Halekesare**	
Partical size distribution	**Surface**	**Subsurface**	**Surface**	**Subsurface**
Sand (%)	60	62.0	62.4	70.4
Silt (%)	12.4	18.6	16.0	13.6
Clay (%)	27.6	19.4	17.6	16.0
Texture class	Sandy clay loam	Sandy clay loam	Sandy loam	Sandy loam
Endosulfan residue	Nil	Nil	Nil	Nil

Table 2: Physico-chemical properties of Hinkal soil at various depths

Depth (cm)	**Bulk density (g/cm^3)**	**Water content (%)**	**Dry density (g/cm^3)**	**Conductivity (mhos.cm)**	**pH**	**Nitrate (mg/g)**	**Phosphate (mg/g)**	**Organic matter (mg/g)**
0-13	2.68	7.7	2.44	0.2	8.67	0.00	6.88	22.63
13-26	2.45	6.2	2.30	0.2	7.36	0.02	1.22	11.32
26-39	2.38	7.8	2.20	0.2	7.28	0.02	1.40	12.82
39-52	2.38	5.4	2.25	0.2	7.27	0.02	2.76	2.26
52-65	2.35	5.8	2.22	0.2	7.24	0.02	0.56	1.50
65-78	2.44	7.8	2.26	0.2	7.23	0.02	0.90	2.26
78-91	2.46	8.3	2.27	0.2	7.50	0.00	0.24	0.00
91-103	2.51	6.7	2.35	0.2	7.51	0.00	2.52	6.00

Table 3: Physico-chemical properties of Halekesare soil at various depths

Depth (cm)	**Bulk density (g/cm^3)**	**Water content (%)**	**Dry density (g/cm^3)**	**Conductivity (mhos.cm)**	**pH**	**Nitrate (mg/g)**	**Phosphate (mg/g)**	**Organic matter (mg/g)**
0-13	2.63	12	2.39	0.72	7.91	2.02	0.32	18.20
13-26	2.54	8.7	2.33	0.71	7.68	6.40	0.34	9.20
26-39	2.44	7.8	2.26	0.54	7.67	5.00	0.35	0.00
39-52	2.40	5.6	2.27	0.34	7.62	3.00	1.80	0.00
52-65	2.45	5.9	2.31	0.34	7.62	3.90	0.34	0.00
65-78	2.43	6.2	2.27	0.38	7.68	4.20	0.47	0.00
78-91	2.41	6.0	2.27	0.38	7.68	3.80	1.90	0.00
91-103	2.43	6.0	2.29	0.39	7.66	2.00	1.80	0.00

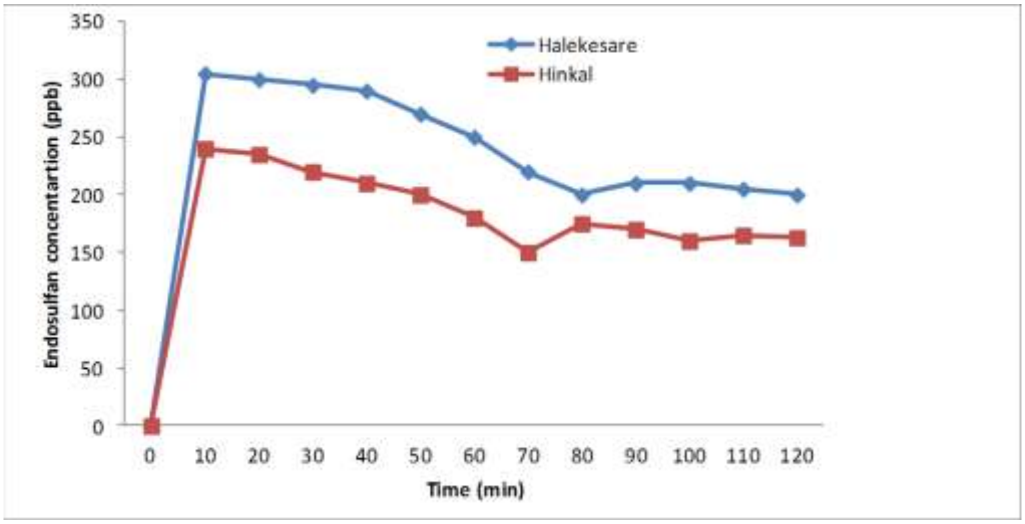

Figure 1: Leaching loss of Endosulfan in Hinkal and Halekesare soil (5 ppm)

Movement, Leaching and distribution of Endosulfan in soil

Data on movement and leaching of endosulfan is shown in Figure 1. When the soil columns were drenched with distilled water, greater movement of endosulfan was observed in Halekesare soil than Hinkal soil. The percentage of endosulfan in lechate effluents of Hinkal and Halekesare are 42% and 52% respectively when 5ppm of initial endosulfan was applied and 49% and 54.5% was observed when 10ppm of endosulfan was applied. The leaching loss of endosulfan was more in Halekesare soil than the Hinkal soil, which could be due to less adsorption of endosulfan to Halekesare soil. The leachate loss was less in Hinkal soil due to more clay content and organic content in the soil, than the Halekesare soil.

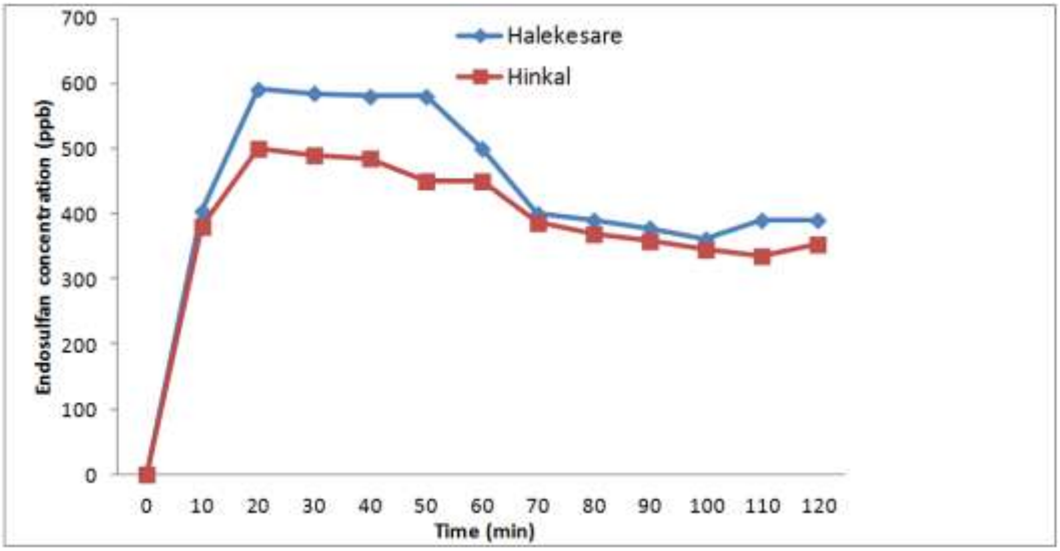

Figure 2: Leaching loss of Endosulfan in Hinkal and Halekesare soil (10 ppm)

The quantity of endosulfan retained in soil decreased with depth of the soil as shown in Figure 2. Endosulfan content is more in upper layer (0-15cm) than the lower layers (15-40cm) of both the sites. This is due to more organic and clay content increased adsorption.

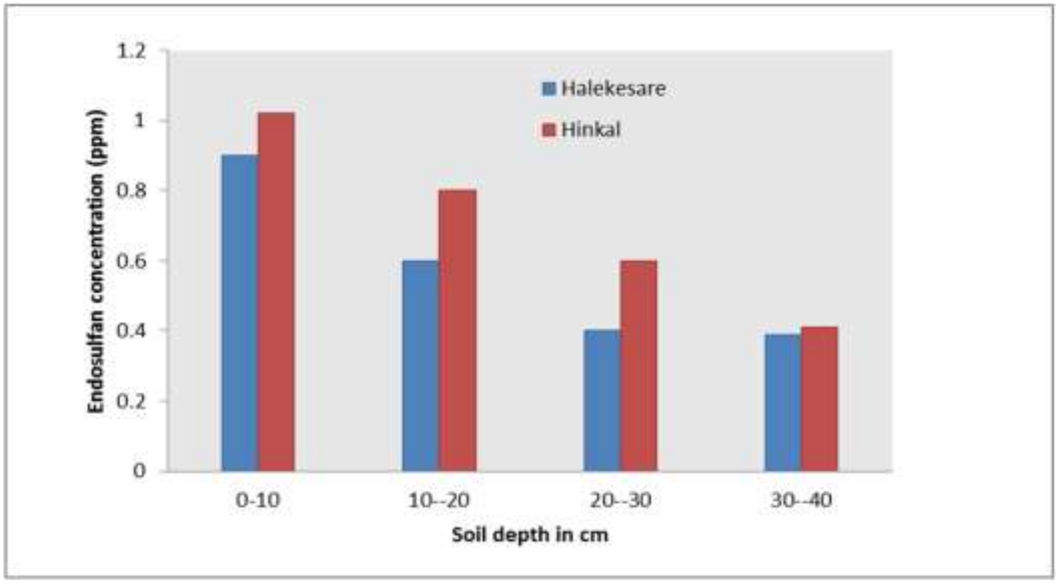

Figure 3: Variation of the endosulfan at different depths (5 ppm)

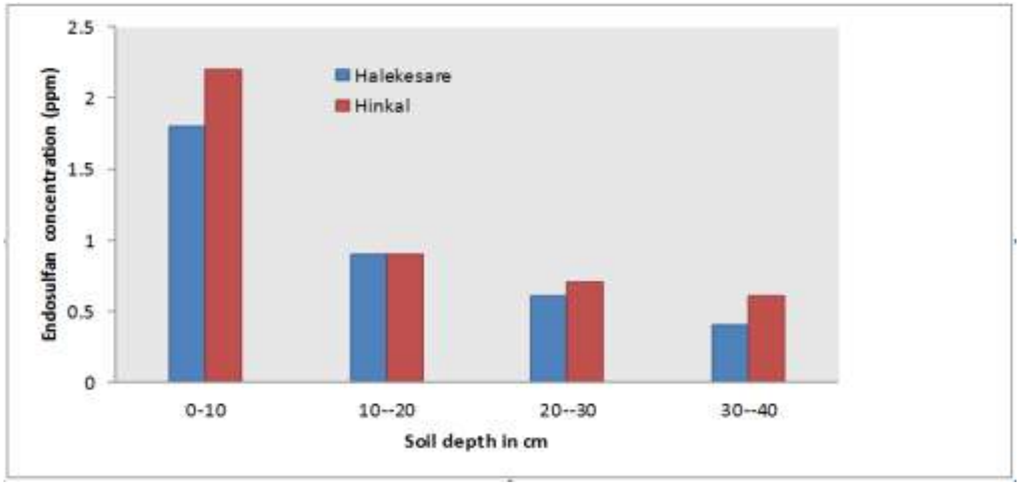

Figure 4: Variation of the endosulfan at different depths (10 ppm

Absorption and desorption studies of endosulfan in soil

The results of adsorption and desorption studies of endosulfan in two different soils are shown in Table 4-6 shows the plot of sorption of endosulfan with time for Hinkal and Halekesarae soil, respectively. The equilibration time required for Hinkal soil to achieve maximum adsorption was 210 min and for Halekesare soil was 330 min. Sorption equilibrium of Endosulfan in each soil showed fast initial reaction followed by steady state a phenomenon often described as bicontinum (Brusseau *et al*., 1991; Locke., 1992). During the first stage of sorption, it is hypothesized that diffuses into the soil matrix and is quickly sorbed at exposed sites. As the remaining exposed sites are filled up, sorption becomes slower. It is also clear from the studies that Hinkal soil with higher clay content and higher organic matter attained equilibrium faster because of active sorptive sites on clay and humic acid of the organic matter.

Adsorption of Endosulfan on to surface soil

The results of adsorption rate of endosulfan on Hinkal and Halekesare soils are presented in Table 4 and 5. The adsorption curves indicated (Figure, 5) that the sorption in Halekesare soil was more than the Hinkal soil. It is amazing to note that factors other than organic matter, exchangable ions and surfactants influence the sorption pesticides to soil (Singh 1997).

Further, measured sorption by Hinkal and Halekesare soils fit well with Freundlich sorption isotherms as shown in figure 3 and 4. This implies that energy of adsorption decreases logarithmically as the fraction of the covered surface increases which is due to surface heterogeneity (Chiou and Porter, 1983; Roy and Krapac, 1994). Blank samples i.e., without any soil, indicated that losses of Endosulfan retained on the centrifuge tubes were found to be insignificant when compared to the amount sorbed by soil.

Table 4: Equilibrium sorption time for endosulfan in Hinkal and Halekesare Soils

Sl. No.	**Time (min)**	***Hinkal Soil***		***Halekesare Soil***	
		$C_e\ (\frac{\mu g}{ml})$	$\frac{X}{m}\ (\frac{\mu g}{g})$	$C_e\ (\frac{\mu g}{ml})$	$\frac{X}{m}\ (\frac{\mu g}{g})$
1	30	7.01	4.99	6.7	5.3
2	60	6.68	5.32	5.8	6.2
3	90	6.33	5.67	5.5	6.5
4	210	5.68	6.32	4.9	7.1
5	330	5.68	6.32	4.8	7.2
6	450	5.68	6.32	4.8	7.2
7	590	5.68	6.32	4.8	7.2
8	690	5.68	6.32	4.8	7.2
9	1000	5.68	6.32	4.8	7.2

Table 5: Adsorption characteristics of endosulfan to Hinkal and Halekesare surface soil

Sl. No.		***Hinkal Soil***		***Halekesare Soil***	
	$C_0\ (\frac{\mu g}{ml})$	**$C_e(\frac{\mu g}{ml})$**	**$\frac{X}{m}\ (\frac{\mu g}{g})$**	**$C_e(\frac{\mu g}{ml}\}$**	**$\frac{X}{m}\ (\frac{\mu g}{g})$**
1	3	1.99	1.01	1.54	1.46
2	5	3.02	1.98	2.67	2.33
3	7	4.12	2.88	3.74	3.26
4	10	5.88	4.12	5.76	4.24

Table 6: Freundlich desorption parameters

Soil samples	**K_{des}**			**$1/n_{des}$**			**Parameters**			
	3 ppm	**5 ppm**	**K_{ada}**	**$1/n_{ada}$**	**%OC**	**K_{oc}**	**10 ppm**	**3 ppm**	**5 ppm**	**10 ppm**
Hinkal	0.93	1.49	**0.31**	**1.31**	**1.28**	**24.22**	2.48	0.08	0.18	0.19
Halekesare	1.35	1.78	**0.04**	**0.78**	**1.05**	**3.80**	2.13	0.14	0.22	0.36

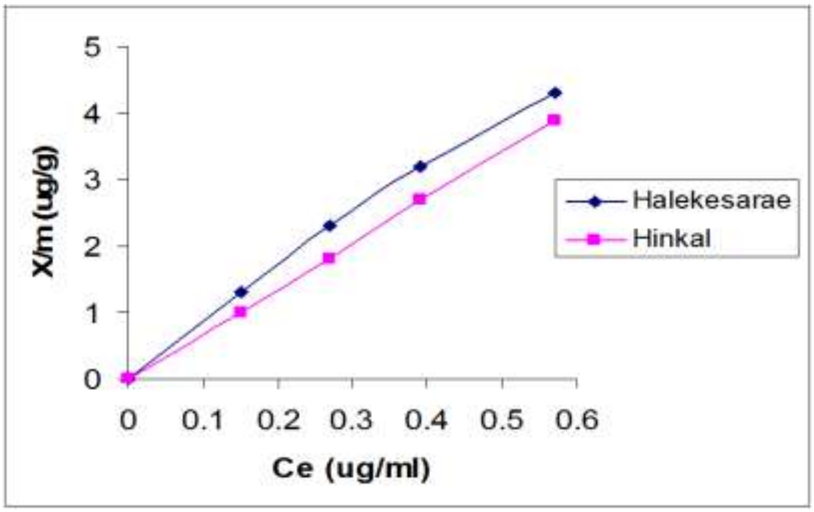

Figure 5: Adsorption curves of Endosulfan of Hinkal and Halekesara soils

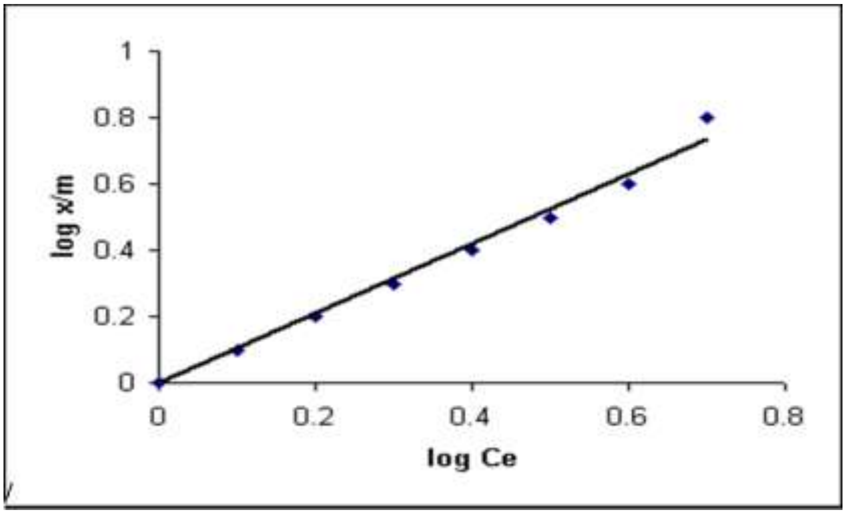

Figure 6: Freundlich adsorption of endosul fan in Hinkal soil

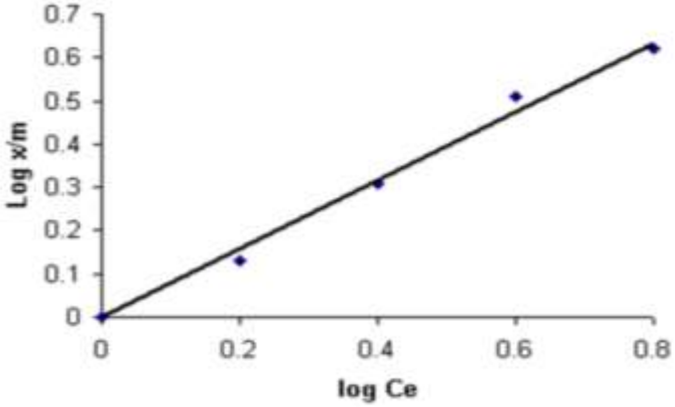

Figure 7: Freundlich adsorption of endosulfan in Halekesare soil

Freundlich adsorption coefficients (K_{ads}) and slope 1/n of each soil are shown in Table 8. Although isotherms were non-linear as indicated by 1/n with value not equal to 1 but ranges from 0.78 of Halekesare soil to 1.31 of Hinkal soil. Similar results have been previously reported for non-ionic compounds as in the range of 0.7 to 0.3 (Hamaker and Thomson, 1972; Pignatello and Huang, 1991). The low concentrations of endosulfan used in this adsorption study might have contributed to linearity observed in the experiments. K_{oc} expresses the concentration of the adsorbed endosulfan per unit weight of organic carbon. The K_{oc} values varied considerably depending on the type of soils. In the present study K_{oc} values for Hinkal and Halekesarae soil are presented in Table 8. The K values in the present study ranged from 24 to 3.80. The higher K_{oc} value of Henkel soil indicates a much greater capacity to adsorb Endosulfan than the Halekesare soil.

Desorption of Endosulfan by Hinkal and Halekesare surface soils

Desorption studies were performed after the adsorption studies for samples treated with 3, 5 and 10 ppm of initial endosulfan concentrations using surface soils. Adsorption (K_{ads}) is based on the experimental data that provides information on the degree of affinity between the pesticide and soils. However, data does not properly predict desorption from soil (Koskinen *et al.*, 1979). Desorption which is the reversibility to the sorption process plays a significant role in determining chemical mobility of pesticide in soil (Seybold and Mersie, 1996).

Freundlich desorption parameters of endosulfan of Hinkal and Halekesare soils are shown in Table 6. Desorption of endosulfan of both soils indicated a significant hysteresis existed at all concentrations studied. Hysteresis indicated as desorption isotherms did not coincide with adsorption isotherm, or k_{ads} did not equal to k_{des}. Hysteresis has often been observed in pesticide sorption studies (Calvet, 1989; Koskinen and Horper, 1990). A positive hysteresis effect was observed in Hinkal and Halekesare soil i.e., in the present study $1/n_{des} < 1/n_{ads}$ .This is true for non-ionic organic compounds such as insecticide Endosulfan (Barriuso *et al.*, 1994). When the pesticide concentration in solution is diluted with each successive desorption step, movement of weakly sorbed pesticide shifts in favour of desorption until desorption solution equilibrium is re-established. The quantity of weekly sorbed pesticide is greatest in first dilution and decline with each subsequent dilution (Reddy *et al.*, 1995; Locke, 1993). The desorption amount of endosulfan to Hinkal and Halekesare soil decreased in the latter steps of successive desorption as shown in Table 5.

Vertical movement of endosulfan in soil

The data on residues of endosulfan in soil at different depths drawn at the time of harvest (45 days after the insecticidal treatment) are presented in Table 7. The analysis of soil samples from different depths after insecticidal treatment showed the presence of endosulfan residues in surface (0-15 cm) as well as sub-soil samples. In lower layer there is little or no microbial activity, leading to accumulation of leaching residues. This soil predominantly contained silt or sand with very little organic matter (< 0.3%) or clay to withhold the pesticide molecules. As a result, the pesticide was carried to the lower layers of soil without much hindrance. Table 7 shows that the vertical movement and accumulation of endosulfan in Hinkal and Halekesare soils. The vertical movement of endosulfan occurred due to insolubility in water gets tanslocated to lower depths under the influence of downward movement of irrigation water.

Table 7: Vertical movement of Endosulfan (concentration in ppm

Dept(cm)	Hinkal	Halekesare
0-15	0.099	0.097
15-30	0.067	0.050
30-45	0.027	0.032
45-60	0.061	0.074
60-75	0.070	0.054
75-80	0.021	0.051
80-95	0.035	0.050
95-110	0.027	0.012

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