

Sorption-Desorption Study of a Herbicide 2,4-Dichlorophenoxyacetic Acid on Acidic Tropical Soils

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ABSTRACT

The sorption and desorption of 2,4-dichlorophenoxyacetic acid (2,4-D) was evaluated on different soils with different range of organic matter content. The batch equilibrium technique under laboratory condition was used to determine the sorption/desorption behavior of 2,4-D in 4 different soil orders of Malaysia viz Histosols (peat), Inceptisols (Selangor and Bria) and Ultisols (Rengam and Serdang) and Oxisol (Munchong). Sorption data were fitted to the linear and Freundlich equations. The values of K_d and K_f ranged from 1.35 to 35.26 and 2.70 to 42.04, respectively. Highest sorption was observed in peat soil and the lowest was in Rengam soil. According to the sorption and desorption results, organic matter and clay seemed to be the most important factor influencing the sorption capacity of 2,4-D. Thus, the contributions of organic matter were evaluated by comparing changes in 2,4-D of sorption before and after organic matter removal. After organic matter was removed from the soils, the K_d values for sorption by Selangor and Munchong, which were calculated from linear and Freundlich equations, decreased by 26.7 % and 28.0 %, respectively. This revealed that soil organic matter greatly influenced the 2,4-D sorption. Based on their sorption capacity, the soils can be ranked in the following decreasing order: Peat > Selangor > Munchong > Bria > Serdang > Rengam Soil Series.

Keywords : 2,4-D, linear equation, Freundlich equation, organic matter, clay minerals

INTRODUCTION

In the recent years, 2,4-dichlorophenoxyacetic acid (2,4-D) (Fig. 1) has been widely used as post emergence herbicides to eradicate broadleaf weeds in oil palm plantation. This herbicide is also known by the trade name 2,4-D Amine, Barrage, Planatox and Weedone. Currently, the herbicides account for about 75.1 % of total agrochemicals used in Malaysia. The usage of 2,4-D in agricultural sector is expected to rise from 1.0 million litres in 1998 to 1.2 million litres in 2010 (Malaysian Agriculture Directory and Index 1999), with the expansion of oil palm hectareage. Extensive use of herbicides has become a concern since it is suspected to exhibit endocrine-disrupting activities (Rawling *et al.* 1998; Short

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and Colborn 1990) and has potentials to negatively impact soil and groundwater quality. However, little is known about their interaction with soil which might affect their environmental fates particularly in tropical acidic soils.

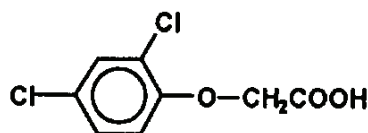


Fig. 1: Chemical structure of 2,4-D.

Sorption is one of the main processes influencing the mobility of herbicides in soil under laboratory (Socias-Viciano *et al.* 1999), as well as under field conditions (Laabs *et al.* 2000). Sorption tends to decrease the degradation rate of herbicides and their bioavailability (Yue *et al.* 2006). Herbicides behaviour in soils greatly depends on adsorption-desorption phenomena (Gao *et al.* 1998; de Jonge and de Jonge, 1999) which is important to predict their potential to leach into groundwater. Soil organic matter content appears to be a predominant factor influencing their retention (Stevenson 1972; Coquet 2002; Spark and Swift 2002) due to the porous nature and large surface of the humic substances. However, the association of 2,4-D with the mineral surfaces, pH, and clay content become significant in low organic matter content soil (Stevenson, 1976; Brownawell *et al.* 1990; Schwarzenbach *et al.* 1993; Baskaran *et al.* 1996 Celis *et al.* 1996, 1999). Interaction at the interface between organic and inorganic soil colloids via sorption may affect the movement of pesticides, resulting in contamination of groundwater.

Previous study on Malaysian soils was done by Cheah *et al.* (1997) on adsorption, mobility and degradation only on sandy loam and muck soils. Moreover, sorption of weak organic acids which can exist in molecular and anionic forms has been less studied (Dubus *et al.* 2001) particularly in variable charged soils. Desorption process is very important in order to know the release rate and their potential mobility in soil. Hence, the objective of this study was to determine the sorption and desorption of 2,4-D in Malaysian soils cultivated with oil palm. Data generated from this research is important to assess the providing information on behaviour of 2,4-dichlorophenoxyacetic acid which is vital to ensure the potential of groundwater contamination in Malaysian agroenvironment.

MATERIALS AND METHODS

Chemicals

Analytical standard of 2,4-D (purity 96%) was purchased from Riedel-de Haen (Germany). Physico-chemical properties of 2,4-D are shown in Table 1. Stock solution of 2,4-D was prepared by dissolving appropriate amount of 2,4-D in

methanol and kept refrigerated. Working solutions (0.25, 0.5, 1, 2.5, 5, 7.5, 10 and 15 mg L⁻¹) were prepared in 0.01 M CaCl₂ and 200 mg L⁻¹ HgCl₂ as background solution to prevent biological activity. Chemical structure of 2,4-D shown in Fig. 1.

Soils

Soil samples were collected from oil palm cultivated areas of West Malaysia. The soils used were from the Order Inceptisol, Ultisol, Oxisol and Histosol. The soil series were peat, Rengam, Serdang, Munchong, Selangor and Bria according to the USDA Soil Taxonomy. The peat, Rengam, Serdang, Munchong, Selangor and Bria soils were classified as Typic Haplohemists, Typic Kandiodult, Typic Paleodult, Haplic Hapludox, Typic Trophaquept, Fluvaquentic or Typic Endoaquepts, respectively (Paramanathan 2000). Surface (0-15 cm) and subsurface (15-30 cm) samples of each soil were air dried and passed through a 2 mm sieve prior to analysis. Selected physical and chemical properties of these soils are listed in Table 1. Soil pH was determined by a Beckman Digital pH meter at soil-water ratio of 1:2.5. Cation Exchange Capacity (CEC) was determined with 1 N ammonium acetate at pH 7 (Thomas 1982). Particle size distribution was determined by pipet method (Day 1965), clay minerals were determined using X-ray diffraction technique. Free Fe/Al oxides were extracted by dithionite-citrate-bicarbonate (Mehra and Jackson 1960) and measured by AAS. In order to evaluate contributions of soil organic matter to 2,4-D sorption, organic matter (OM)-removed soils were prepared by heating the soils at 750 °C for 2 h in a muffle furnace (Ying, 2005).

Table 1
Physico-chemical properties of 2,4-D.

Chemical	Molecular weight	pK _a	Solubility in water at 25 °C(mg L ⁻¹)
2,4-D	221.0	2.73	23,180 (pH 7)

(Source: Tomlin (2001))

Sorption-Desorption Experiment

Sorption of 2,4-D was conducted using a batch equilibrium method (OECD, 2000). The soil samples about 2 g were weighed into a centrifuge tubes. The solution containing either 0, 0.25, 0.50, 1.00, 2.50, 5.00, 7.50, 10.00 or 15.00 mg L⁻¹ 2,4-D in 20 ml of 0.01 M CaCl₂ and 200 mg L⁻¹ HgCl₂ as bioinhibitor were added. The concentrations were prepared in triplicates. The tubes were shaken for 24 h and centrifuged at 3,500 rpm for 15 min. The supernatant was decanted and determined for 2,4-D concentration at equilibrium. The amount of herbicide adsorbed (C_s) were calculated by taking the difference between the initial (C_i) and the equilibrium (C_e) 2,4-D solution concentrations. Sorption isotherms was

obtained by plotting the amount of chemical sorbed per unit weight of soil at equilibrium (C_s , mg kg⁻¹) versus chemical concentration at equilibrium (C_e , mg L⁻¹). Sorption isotherm data were fitted to both linear and Freundlich equation:

$$C_s = K_d C_e$$

and the Freundlich equation (log-transformed);

$$\log C_s = \log K_F + n \log C_e$$

where C_s is the amount of herbicides sorbed, C_e is the equilibrium concentration in solution and K_F is the Freundlich constant.

Desorption experiments were performed immediately after supernatant was decanted by adding 20 mL solution 0.01 M CaCl₂ and 200 mg L⁻¹ HgCl₂. The soils were resuspended by shaking the tubes for a further 24 h under the same experimental conditions as explained above.

HPLC Analysis of 2,4-D

The analysis of the 2,4-D was performed by HPLC (Waters) equipped with autosampler injector and Photodiode Array (PDA) detector. Determination was done with methanol and ammonium formate buffer at pH 4.5 (50:50) using isocratic mode, flow rate of 0.6 mL min⁻¹, the column was Zorbax 300 SB-C₁₈ (4.6 x 250 mm) and the injection volume used was 20 µL.

RESULTS AND DISCUSSION

Soil Physico-Chemical Properties

The physico-chemical properties of soils used are shown in Table 2. All the soils were acidic. In general, soil pH in the surface is higher than subsurface excluding Munchong soil. Peat soil had a greater CEC due to the higher organic matter content. Its CEC value was almost 2 times more than the mineral soils. In mineral soils, Munchong had a higher content of clay (56 %) than other soils. In general, the physico-chemical data observed were slightly lower than those reported by Paramanathan (2000).

Sorption-Desorption of 2,4-D

Table 2
Selected chemical properties of the soils.

Soils	Depth	Texture	% OC	% OM	pH (H ₂ O)	CEC (cmol ⁺ /kg)	% Clay	% silt	% sand
Peat	0-15	organic	17.55	30.54	4.37	20.99	-	-	-
Munchong	0-15	Clay	2.92	5.08	4.95	4.94	56.02	10.98	32.90
	15-30	Clay	1.93	3.36	5.03	4.56	57.66	9.11	33.14
Selangor	0-15	Clay	2.75	4.79	4.30	15.76	54.45	37.70	7.78
	15-30	silty clay	1.94	3.38	4.10	12.66	53.47	42.91	3.56
Rengam	0-15	Sandy clay	2.55	4.43	5.53	5.17	41.02	6.85	52.10
	15-30	Sandy clay	1.81	3.15	5.04	5.24	44.90	7.59	47.38
Serdang	0-15	sandy loam	1.70	2.96	5.04	9.32	22.02	5.73	72.25
	15-30	sandy clay loam	1.64	2.86	4.94	9.33	31.10	6.82	62.08
Briah	0-15	silty clay	1.67	2.91	4.59	11.80	48.53	41.97	9.42
	15-30	Clay	1.14	1.98	4.41	13.11	50.42	39.85	9.68

Soil Mineralogy

Clay mineral analysis showed that kaolinite, montmorillonite and vermiculite is the most abundant mineral in soil samples. The higher content of CEC in Selangor and Briah probably due to the higher content of 2:1 layer silicates, especially montmorillonite and vermiculite. Both of the soils were richer in free Fe/Al oxides as well as % silt, even the % clay is lower than Munchong soil (Table 3).

Table 3
Mineralogical content of the soils.

Soil	Depth	Al oxides	Fe oxides	Mineral					
				gibbsite	quartz	illite	kaolinite	montmorillonite	vermiculite
Munchong	0-15	7.32	20.80	+	++		++		
	15-30	10.88	23.03	+	+	+	+++		
Selangor	0-15	12.65	43.41		+	++	+++		+
	15-30	12.94	47.11		+	++	++	+++	+++
Rengam	0-15	7.69	14.36		+++		+++		
	15-30	10.38	27.31		+++		+++		
Serdang	0-15	7.76	10.43	+	+++		++		+
	15-30	7.99	10.06	+	+++		+++		+
Briah	0-15	20.70	79.79		+	+	++	++	++
	15-30	26.04	92.04		+	++	++	++	++

Note: +++ - abundant
++ - present
+ - traces

Sorption by Natural Soils

The sorption isotherms of 2,4-D on different soils are shown in Fig. 2. In general, both linear and Freundlich equations described the sorption data rather well as indicated by the R² values close to 1 for all sorption isotherms (Table 4). Sorption data for most of the soils fitted well with the Freundlich and values for K_d, K_F, n and R² for selected soils are given in Table 4. The values for K_F are larger than K_d values for all isotherms. Most of the soils are L types (n<1) curves (Giles *et al.*, 1960) indicates that sorption decreases with increasing concentrations of sorbates. The n value close to 1 is observed for sorption of non-ionic organic chemicals to soil with high organic matter content. However, some of the isotherms exhibit a C

types (e.g. Munchong with OM removed and Serdang with OM removed) curves, indicates that a constant partition between soil and solution over the concentration range used.

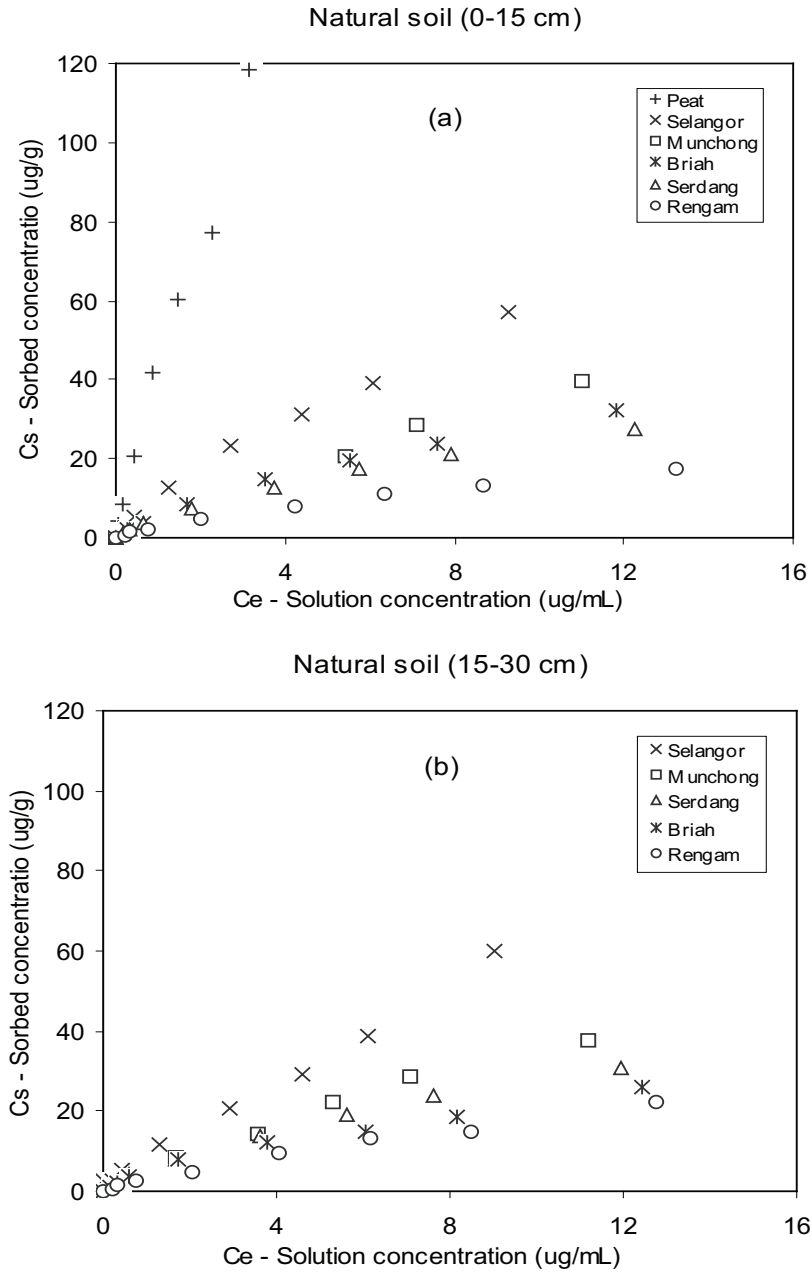


Fig. 2. Sorption isotherms of 2,4-D on selected natural soils at the surface (0-15 cm) and subsurface (15-30 cm).

Table 4
Linear coefficient (K_d), Freundlich coefficient (K_F), n and R values for sorption of 2,4-D by soils before and after removal of organic matter.

Soils	Depth	K_d (L kg ⁻¹)	R ²	K_F	R ²	n
Peat	0-15	36.26	0.99	42.04	0.98	0.95
Selangor	0-15	6.11	0.99	9.92	1.00	0.79
	15-30	6.30	1.00	9.55	1.00	0.79
Munchong	0-15	3.64	0.99	5.15	1.00	0.84
	15-30	3.52	0.98	5.00	1.00	0.86
Briah	0-15	2.77	0.97	5.35	1.00	0.75
	15-30	2.00	0.98	4.74	0.99	0.67
Serdang	0-15	2.29	0.97	4.69	1.00	0.73
	15-30	2.61	0.97	5.74	0.99	0.69
Rengam	0-15	1.35	0.97	2.70	0.99	0.75
	15-30	1.72	0.98	5.04	0.99	0.82
Selangor, OM-removed	0-15	4.48	0.86	12.14	0.95	0.61
	0-15	2.81	0.93	6.77	0.99	0.66
Munchong, OM-removed	15-30	2.58	0.71	0.71	0.46	1.30
	0-15	2.41	0.64	0.92	0.57	1.03
Briah, OM-removed	15-30	4.72	0.96	7.00	0.94	0.87
	0-15	4.56	0.96	7.00	0.94	0.87
Serdang, OM-removed	15-30	2.45	0.67	1.53	0.72	1.78
	0-15	2.20	0.63	1.34	0.53	0.73
Rengam, OM-removed	15-30	0.00	n/a	0.00	n/a	0.00
	0-15	0.00	n/a	0.00	n/a	0.00

As shown by K_F values, peat exhibited a higher sorption capacity, followed by Selangor, Munchong, Briah, Serdang and the lowest were observed in Rengam soil (Fig. 3). The differences in sorption capacities of the soils can be attributed to the differences in organic carbon content (Table 2). On the other hand, contribution of clay constituents to 2,4-D retention can be dominant in low-OM soils. This is shown in the present research where the Briah soils have the high retention of 2,4-D with low OM-content. For all soils, the K_F values in the surface had the highest sorption capacity excluding Serdang and Rengam, the highest K_F were observed for the subsurface soils. This could be related to high content of organic matter and its CEC in surface appeared to be larger than Serdang and Rengam even though the organic matter content in surface soils were also high. Furthermore, change of sorption with depth can be because of pH. The pH values for all soils were greater for the surface rather than subsurface soils. On the other hand, the evaluation of a single soil variable on sorption is always difficult due to the correlation among soil properties itself. Contribution of organic carbon to sorption of 2,4-D have been reported by other researchers (Reddy and Gambrell 1987; Kah and Brown 2007). Intra-particle diffusion into organic matter moieties could have been the mechanism for 2,4-D sorption by soils with high organic matter content (Aksu and Kabasakal 2004).

The K_F value for 2,4-D sorption in peat is 42.04 L kg⁻¹ (Table 2) and this value is much higher than the value of 6.95 L kg⁻¹, reported by Cheah *et al.*

(1997) for sorption of 2,4-D in muck soil taken from paddy field. Correlation study revealed high correlation between sorption and total carbon, CEC and % clay (Table 5). Although 2,4-D is a weak acid with a pKa around 2.64, results showed that sorption was the highest in peat and Selangor soil (a mineral soil with the highest organic matter content). It could be suggested that 2,4-D sorbs on organic matter and Fe oxide via electrostatic interaction between negatively charged carboxylic group and positively charged Fe oxide surface. At the high pH, there is little bonding of anionic molecules in soil as a result of the repulsion by negative charge of soil particles. The main sources of positively charged surfaces in soils are iron oxides, clay mineral and association with organic matter content.

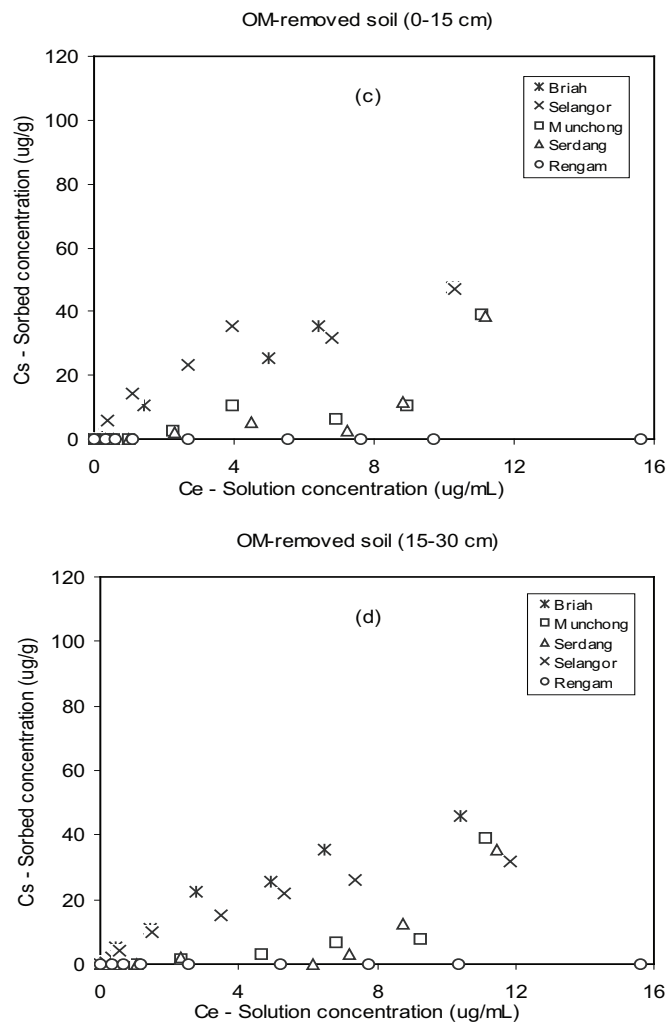


Fig. 3. Sorption isotherms of 2,4-D on selected soils with OM-removed at the surface (0-15 cm) and subsurface (15-30 cm).

Table 5
Simple correlation coefficients between K_d and K_F values and soil properties ($p=0.05$).

	Total C	pH	CEC	% Clay	% Silt	% Sand
K_d	0.983*	0.444	0.852	0.976*	0.968*	-0.978
K_F	0.971*	-0.476	0.883*	0.927	0.869	-0.891

Sorption-Desorption By Soils with OM-Removed

The sorption of 2,4-D by soils with OM removed are also well described by Freundlich equation (Fig. 2). The respective values for K_d , K_F , n and R^2 for soils with OM removed are shown in Table 4. The removal of organic matter from the soils greatly reduced 2,4-D sorption for Selangor and Munchong. Compared with their original soils, K_d value for soils with OM removed decreased by 26.7 % and 28.0 %, respectively (Table 4). For all soils with OM removed, the sorption capacities in surface were higher than subsurface. As shown in Table 4, the sorption capacity for soils with OM removed were higher in Selangor, followed by Briah, Serdang, Munchong and the lowest was observed in Rengam. However, in Briah soil there was 30% increase in sorption capacity after OM removal compared to the original soil. The high content of iron oxides which sorbed 2,4-D anions through electrostatic interactions with positive charges Fe oxides surface enhance sorption. This can be explained by the hypothesis of mineral blockage by organic matters which control the sorption process. Therefore, the contribution of mineral ionic sorption was expected after organic matter removal. This result suggested that soil organic matter provide about 25-30 % of sorption sites. The other 75-80 % of sorption took place on metal oxides and layer silicates fractions, indicating the significant contribution of soil mineral to sorption process. This findings support the study by Boivin *et al.* (2005), which stated that organic matter content appears to control 2,4-D sorption.

Desorption

Sorption and desorption patterns are shown in Fig. 4. The extent of pesticides release from the soils can also be seen. As a whole, the desorption were higher in Rengam, followed by Serdang, Briah, Munchong, Selangor and the lowest was observed in peat. This is related with the high organic matter content in soils which enhanced the sorption capacity. The desorption of pesticides from the soils were high, thus they will be easily leached or moved into groundwater system particularly in highly weathered soils. The desorption process occur due to the low bonding between 2,4-D molecules and active sites in soil resulting in high release rate.

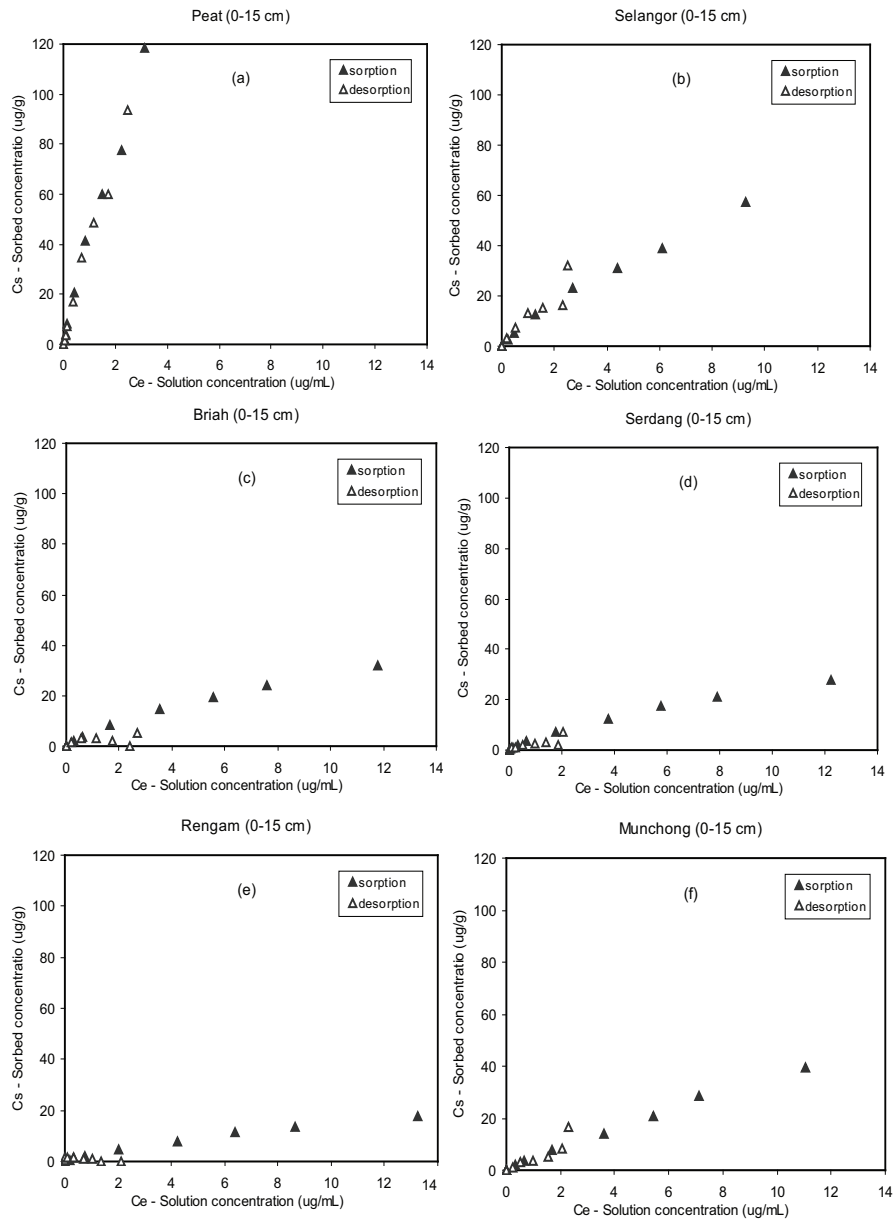


Fig. 4. Sorption-desorption isotherms of natural soils at the surface (0-15 cm).

CONCLUSIONS

As expected, sorption of 2,4-D was closely related with the organic matter content and clay. Even the small amounts of OM will contribute to the increase of 2,4-D sorption. Apart from that, CEC and clay also gave significant contribution towards sorption. It was noted that removal of organic matter from soils would unmasked the sorption sites on soil minerals that were originally covered by organic matter. Desorption was highest in soil with low organic matter resulting in the potential of groundwater contamination.

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