

Original Research

Adsorption of Herbicide Diuron in Pineapple-Growing Soils of Eastern Thailand

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Abstract

Four pineapple-growing soils (topsoil) were collected from Chonburi and Chachoengsao provinces in Thailand to analyze the physiochemical properties, chemical composition of organic carbon and diuron adsorption isotherm (batch experiment). Results revealed that diuron adsorption rate of soils was low (ranging between 1.81 and 27.63%) and adsorption data fit well with Freundlich equation ($R^2 = 0.83-0.97$), in which the adsorption coefficient (K_f) is between 0.57 and 4.87 L kg⁻¹. Soil organic matter and clay content were responsible for the adsorption of diuron in these soils ($r = 0.95^{**}$ and 0.78^{**} , respectively). In addition, total nitrogen, silt content and cation exchange capacity also had a positive correlation with diuron adsorption in the soils. Organic carbon partitioning coefficient (K_{oc}) of soils ranged from 125.84 to 480.36 L kg⁻¹ and showed a positive correlation with the functional group of organic carbon, including aromatic carbon and carbonyl ($r = 0.80^{**}$ and 0.67^* , respectively). This low rate of diuron adsorption in pineapple-growing soils posed a risk in diuron contamination in the environment. The increase of organic matter in these soils, particularly those coarse to medium-textured soils, through proper soil management would increase the herbicide adsorption ability of these soils and should subsequently reduce the contamination markedly.

Keywords: diuron, adsorption, pineapple growing soils, soil organic matter

Introduction

Nowadays, social direction focuses on natural and organic farming, which bans or restricts the use of pesticides. However, chemicals such as herbicides and insecticides are still importantly essential in crop and livestock productions – particularly in the tropics, where severe outbreaks of weeds and pests are common and threaten the loss of such productions. Diuron (*N*-(3,4-

dichlorophenyl)-*N,N*-dimethylurea) is the main systemic phenyl urea herbicide which inhibits the photosynthesis at PS II of plants [1-2]. Diuron formulation is usually in wettable powder (WP) and suspension concentration (SC). It is a broad-spectrum residual herbicide registered for pre-emergence and post-emergence to control of both broadleaf and annual grassy weed [3]. The application of diuron has been directed moderately persistently in agricultural soil and water, air, organisms and other environments [4-7]. It could result in soil and water pollution and other ecological problems. For its toxicity, diuron is slightly toxic to mammals and birds, moderately toxic to aquatic invertebrates and exerts

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harmful effects on fetal development and damages human health [8-9].

Thailand is an agricultural-based country. Pineapple is one of main economic crops of the country. The average production of pineapple in Thailand is reportedly more than 1.7 million tons per year, which makes it one of the top five largest pineapple producers in the world [10]. In an attempt to reduce the incidence of pests, a large number of pesticides are usually applied for pineapple production. Diuron is one of the main herbicides, having been used by farmers in pineapple growing areas to control a wide range of weed such as Chick weed (*Ageratum conyzoides*) and Crowfoot grass (*Dactyloctenium aegyptium*) for more than 10 years [11]. However, the application is often inappropriate due to incorrect application techniques (e.g. over-dosed rate as recommended and improperly mixed with other pesticides), poorly maintained or totally inappropriate spraying equipment, etc., which results in residue in plants, water sources and especially soil. Moreover, pineapples commonly requires well-drained soil that rapid leaching or vertical movement of water through the soil leads to increasing the risk of pesticide contamination in the environment.

The study on adsorption in soils is useful for understanding key processes that affect transportation, degradation and bioavailability of pesticides. Soil organic matter plays the main role in adsorption of hydrophobic organic compound of soils [12-13], and diuron molecules also have similar hydrophobicity characteristic [14]. Many studies have reported about diuron adsorption on many cultivated soils [3, 13, 15], but there is a lack of information for pineapple growing soils which have a high risk of diuron environmental contamination. Thus, this study was carried out to investigate the adsorption behavior of diuron in pineapple-growing soils and the relationship between diuron adsorption and soil properties, especially soil organic matter. The finding will provide proper soil management guidance for lessening the risk of diuron contamination from pineapple growing areas to the environment.

Material and Methods

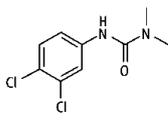
Chemicals

Technical-grade diuron (99% purity) was purchased from Sigma-Aldrich in Germany. Physical and chemical properties of the diuron used are shown in Table 1. All other chemicals were analytical grade.

Soil Sample Collection and Preparation

Topsoil (Ap horizon; 0-20 cm) with three replicates of four pineapple growing soils were collected from farmers' pineapple growing areas in Chonburi and Chachoengsao provinces situated in eastern Thailand, where pineapple is grown extensively. Coordinates, site

Table 1. Physical and chemical properties of diuron [1-2].

Properties	Diuron
Structure	
CAS numbers	330-54-1
Molecular weight	233.11
pK _a	Neutral
Solubility (mg kg ⁻¹)	42
Octanol/water partition coefficient (K _{ow})	648-747

description, soil parent material and soil classification were recorded as shown in Table 2. Soil samples were air-dried and sieved through 2.0 and 0.5 mm for the analysis of physicochemical properties, soil organic carbon composition and diuron adsorption.

Physicochemical Analysis of Soils

The particle size distribution was determined using the pipette method [17]. Mineral composition of whole soils and clay fractions were determined by X-ray diffraction method (Cu K α radiation at 35 kV) [18]. Chemical properties of soils were analyzed as described by [19] as follows: soil pH was measured in water using 1:1 (soil:water); soil organic matter was estimated by the determination of soil organic carbon (OC) (dichromate oxidation procedure) and then multiplied by 1.724; cation exchange capacity (CDC) was extracted with a 1M NH₄OAc solution at pH 7.0; total nitrogen was determined by Kjeldahl method; available phosphorus was determined by Bray II method; available potassium was extracted by 1M NH₄OAc (pH 7.0) and analyzed using an atomic absorption spectrophotometer (AAS); electrical conductivity (ECe) was determined in a saturated paste extract.

Chemical Composition of Soil Organic Carbon

Soil samples were HF-treated basing on a procedure described by [20]. Briefly, the HF treatment consisted of nine successive treatments with 2% hydrofluoric acid solution. Each treatment involved shaking 3 g portions of soil and being shaken with 50 mL of 2% hydrofluoric acid solution 1 h (5 times), 16 hrs (3 times) and 64 hrs (once), respectively. Between treatments, the sample was centrifuged at 2,000 rpm for 20 min. After the final extraction, the soil residue was washed three times with distilled water, freeze-dried and then analyzed the functional group of organic carbon by CP-MAS ¹³C nuclear magnetic resonance spectroscopy (NMR).

Table 2. Location, province, district, parent material and classification of pineapple-growing soils.

Soil sample	Location		District, Province	Parent material ^a	Soil classification ^a
	X	Y			
CL	763277	1493924	Plaeng Yao, Chachoengsao	Shale and basalt	Ultisols
SL	767170	1448386	Nong Yai, Chonburi	Shale, siltstone and sandstone	Ultisols
SCL	769190	1444879	Nong Yai, Chonburi	Shale	Ultisols
LS	745063	1446691	Nong Yai, Chonburi	Granite	Ultisols

^a Source: [16]

Adsorption Experiment

Adsorption experiment was carried out using a batch equilibrium method. Diuron solution was prepared by dissolving weighed diuron in methanol and subsequently diluted it to different concentrations in DI water containing 0.01 M CaCl₂ and 0.02% NaN₃. The triplicate samples of 2.5 g soil (<2 mm size) were placed in a 50 mL polypropylene centrifuge tube and mixed with 25 mL of diuron at different concentrations of 0.5, 1.0, 2.0, 4.0, 8.0, 16 and 32 mg L⁻¹, respectively. The samples were shaken for 18 hrs at 210 rpm, then centrifuged at 10,000 rpm for 10 min. The supernatant was decanted and filtered using 0.45 µm syringe filters. Diuron equilibrium concentrations (C_e) were determined by ultra-high performance liquid chromatography (UHPLC) Series 1290 (Agilent, U.S.A.) equipped with a diode-array detector at 250 nm. The column was Merck C18 (4.6 mm × 250 mm × 5 µm). The flow phase was acetonitrile: distilled water (60:40, v/v). The injection volume was 20 µL. The total run time was 10 min. The retention time of diuron was 4.61 min.

Adsorption data were fitted to the Freundlich model (Eq. (1)) in log format, log K_F and 1/n are the adsorption parameters calculated from the linear regression (Eq. (2))

$$C_s = K_F C_e^{1/n} \quad (1)$$

$$\log C_s = \log K_F + 1/n \log C_e \quad (2)$$

...where the Freundlich adsorption isotherm represents the relationship between the corresponding adsorption capacity C_s (mg kg⁻¹) and the concentration of the diuron in solution at equilibrium C_e (mg L⁻¹). The K_F is the adsorption coefficient, and value n represents the energy distribution of adsorption sites.

The organic carbon partitioning coefficient (K_{OC}) was also calculated as a function of K_F and organic carbon content in soils as the following Equation (3).

$$K_{OC} = (K_F / \%OC) \times 100 \quad (3)$$

Statistical Analysis

Statistical evaluation was performed using Pearson correlation test (two-tailed) to study the relationship between the adsorption data and soil properties.

Results and Discussion

Physicochemical Property of Four Pineapple-Growing Soils

The physicochemical property of four soils collected from pineapple growing is summarized in Table 3. There were sandy loam (SL) and loamy sand (LS) soils represented by coarse-textured soil, a sandy clay loam (SCL) soil represented by medium-textured soil, and a clay loam (CL) soil representing fine-textured soil. Mineralogical property of whole soils showed that quartz was a dominant mineral, whereas kaolinite was a major clay mineral in clay fraction of all soils, which was common in highly weathered soil, especially in Ultisols [21]. Moreover, whole soils and clay fraction in this study also contained trace amounts of montmorillonite, illite, anatase, muscovite, zircon, microcline and dolomite.

The pH of SL, SCL and LS soils were extremely acid, but the CL soil was moderately acid. Soil organic matter and total nitrogen of all soils were low; however, the CL soil had a higher value than did the other soils. Cation exchange capacity of these soils was very low (1.11-1.96 cmol_c kg⁻¹), except for CL soil that had moderately low value (6.28 cmol_c kg⁻¹), which was consistent with organic matter content and mineralogical properties of soils. Available phosphorus was medium to very high (13.97-112.41 mg kg⁻¹) and available potassium was very low in SL, SCL and LS soils (12.42-24.76 mg kg⁻¹), but the CL soil had a very high value (544.72 mg kg⁻¹). Electrical conductivity was in the range of 0.21-2.08 dS m⁻¹, therefore they were non-saline soils. Most studied soils had a low soil pH, organic matter, cation exchange capacity and total nitrogen because they are highly weathered soils and basic cations were leached. However, the increase of available phosphorus and

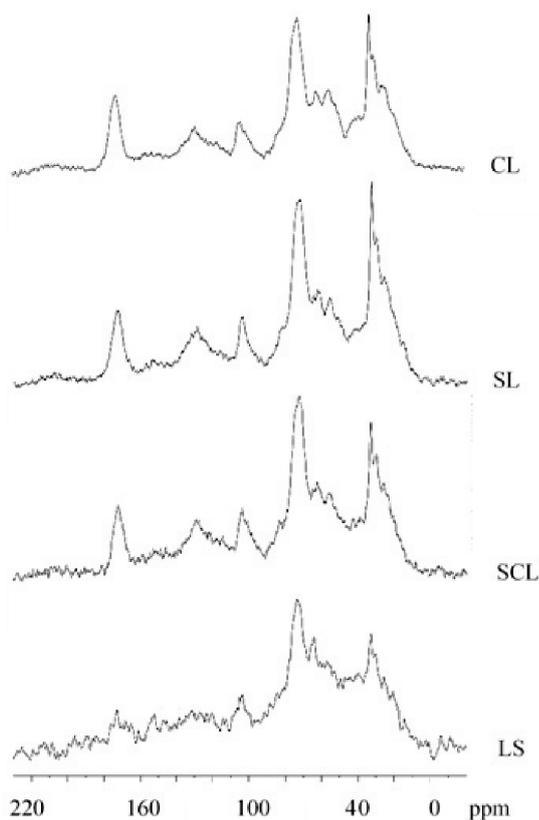


Fig. 1. Solid-state ^{13}C NMR spectra of pineapple-growing soils.

available potassium may be influenced by fertilization and intensive agricultural practices.

Chemical Composition of Organic Carbon

Results of ^{13}C NMR spectra and relative proportions of organic carbon are shown in Fig. 1 and Table 4, respectively. All soil samples had the highest O-alkyl carbon content (at 45-110 ppm) in the range of 47.71-58.81%, which was similar to that of forest and paddy soils [22-23]. The dominant signal of this region was found at 72 ppm and 104 ppm, which was assigned

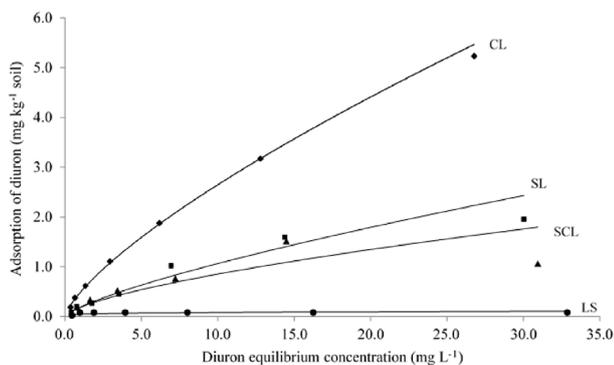


Fig. 2. Adsorption curve of diuron in four pineapple-growing soils.

as polysaccharide [24]. The signal at 104, which was between 95 ppm and 112 ppm, was also the signal region of sugar (alkyl O-C-O) [25]. Moreover, at signal 56 ppm, it represented the carbon from methoxyl group derived from lignin and α -protein [26].

Alkyl carbon (at 0-45 ppm) was the second-highest organic carbon content in the studied soils with the amounts ranging between 24.07 and 31.59%. It was carbon that derived from long chain of lipid and peptide [27]. In addition, at signal 33 ppm, carbon from crystalline and amorphous polymethylene ($-\text{CH}_2-$) was presented [28]. Aromatic carbon (at 110-160 ppm) was found in the range of 4.16-14.37%, which was the third highest amount of organic carbon in these soils, with the exception of that in LS soil. Carbon in this signal region was derived from lignin (at 129 ppm), which was found near 125 ppm signal (Exomethylenes, $-\text{C}=\text{CH}_2$) [29]. Due to no burning of crop residues in pineapple cultivation, these pineapple-growing soils had a smaller signal of aromatic carbon than did banana plantation and forest soils (19-35%) [22]. This was because high content of aromatic carbon in soils arose from fire or burning of plant biomass, which accelerated oxidation processes [22, 30-31]. For the carbonyl carbon (at 160-190 ppm), it was very low in all soil samples (7.22-9.26%) and was found in carboxylic acid ($-\text{COOH}$) dominant peak at 173 ppm [25].

Diuron Adsorption

Diuron adsorption curve and data of four pineapple-growing soils are presented in Fig. 2 and Table 5, respectively. Results showed that 27.63% of diuron was adsorbed in CL soil, of which this soil had the highest organic matter content, while respective amounts of 12.77%, 12.00% and 1.81% were adsorbed in SL, SCL and LS soils. The adsorption data fitted well with the Freundlich model. The CL, SL and SCL soils showed an L-type isotherm ($n > 1$), indicating that diuron could also adsorb onto the clay fraction apart from organic matter, whereas the LS soil showed a C-type isotherm ($n < 1$), demonstrating that the adsorption of diuron in this soil was mainly a partition between soil organic matter and soil solution [3]. The adsorption coefficient (K_F) ranged from 0.57 to 4.87 L kg^{-1} with high regression coefficients ($R^2 = 0.83-0.97$). The adsorption rate and the K_F indicated the adsorption of diuron in soils being in the sequence of $\text{CL} > \text{SL} > \text{SCL} > \text{LS}$.

Relationship between Diuron Adsorption and Soil Properties

The K_F of four pineapple-growing soils in this study statistically had a significant positive correlation with contents of organic matter, total nitrogen, silt content, cation exchange capacity and clay content ($r = 0.95^{**}$, 0.93^{**} , 0.90^{**} , 0.80^{**} and 0.78^{**} , respectively), but it negatively correlated with sand content ($r = -0.90^{**}$), as illustrated in Table 6. The organic matter played

Table 3. Average values of physicochemical properties of pineapple-growing soils.

Properties	Soil sample			
	CL	SL	SCL	LS
Soil texture	Clay loam	Sandy loam	Sandy clay loam	Loamy sand
Sand (g kg ⁻¹)	252	738	697	800
Silt (g kg ⁻¹)	437	149	101	106
Clay (g kg ⁻¹)	311	113	202	94
Mineralogy of whole soil	Quartz ^a Kaolinite ^c Anatase ^c Muscovite ^c	Quartz ^a Zircon ^c	Quartz ^a Kaolinite ^c Dolomite ^c	Quartz ^a Microcline ^c
Mineralogy of clay fraction	Kaolinite ^c Montmorillonite ^d Quartz ^d	Kaolinite ^b Quartz ^d Montmorillonite ^c Illite ^c	Kaolinite ^a Quartz ^c	Kaolinite ^b Quartz ^d Illite ^c
Soil pH 1:1 (soil:H ₂ O)	5.79	3.92	3.74	3.83
Soil organic matter (g kg ⁻¹)	2.25	0.69	0.97	0.79
CEC (cmol _c kg ⁻¹)	6.28	1.96	1.81	1.11
Total N (g kg ⁻¹)	1.54	0.37	0.28	0.21
Avail. P (mg kg ⁻¹)	87.95	13.97	17.70	112.41
Avail. K (mg kg ⁻¹)	544.72	19.67	12.42	24.76
E _{Ce} (dS m ⁻¹)	1.69	0.59	0.21	2.08

^a dominant; ^b large; ^c moderate; ^d small; ^e trace

a major role in affecting the adsorption of diuron in these soils because non-polar and hydrophobicity ($K_{ow} = 700$) of diuron made the organic matter adsorb diuron by partition mechanism [3]. This finding was in agreement with [3], who reported that soil organic matter content plays an important role in the adsorption and desorption of diuron. The SL and SCL soils had similar diuron adsorption rates and also organic matter content. For LS soil, it showed the lowest diuron adsorption rate, although it had a similar organic matter content with SL and SCL soils. This was due to the LS soil having low clay content as this soil particle, having a small particle size and high specific surface area, also partly affected the adsorption rate. Furthermore, clay fraction adsorbed diuron more than bulk soils, sand and silt fractions due to their higher soil organic carbon content [32]. In contrast, the SL soil had less clay content than did the SCL soil, but the former had a slightly higher diuron

adsorption rate than did the latter. In this case, dolomite, which the SCL soil contained small amount of dolomite, should play a part in the adsorption [33]. In addition, cation exchange capacity also had a positive effect on diuron adsorption because this soil property was closely related to soil organic matter and clay content. This was consistent with the studies of [3], who found that the adsorption of diuron had a relationship with cation exchange capacity.

Soil pH has no significant effect on the adsorption of diuron because diuron is a non-ionic pesticide [13]; nevertheless, it affects variable surface charges of organic matter and clay mineral in soil [34]. In addition, it has been reported that potassium chloride can increase the adsorption of diuron in smectite due to the increase of available potassium-added ionic strength to cause a reduction of water available, effecting the attraction of diuron by solid phase of soils [35]. However, the diuron

Table 4. Proportions of each carbon region in NMR spectra (%).

Soil sam- ple	Alkyl (0-45 ppm)	O-alkyl (45-110 ppm)	Aromatic carbon (110-160 ppm)	Carbonyl (160-190 ppm)
CL	31.37	47.71	11.81	9.11
SL	26.12	50.25	14.37	9.26
SCL	24.07	58.81	9.90	7.22
LS	31.59	56.25	4.16	8.01

Table 5. Adsorption parameters of diuron in pineapple-growing soils.

Soil sample	Adsorption rate (%)	Freundlich model			K_{OC} (L kg ⁻¹)
		K_F (L kg ⁻¹)	n	R^2	
CL	27.63	4.87	1.37	0.97	377.51
SL	12.77	1.86	1.35	0.95	480.36
SCL	12.00	1.82	1.59	0.83	324.95
LS	1.81	0.57	0.85	0.89	125.84

adsorption in four pineapple-growing soils in this present study was not affected by available potassium, available phosphorus and ECe.

Correlation between Organic Carbon Partitioning Coefficient (K_{OC}) and Functional Group of Organic Carbon

Organic carbon partitioning coefficient (K_{OC}) ranged from 125.84 to 480.36 L kg⁻¹ (Table 5) and had

a significantly positive correlation with a functional group of organic carbon, including aromatic carbon and carbonyl ($r = 0.80^{**}$ and 0.67^* , respectively) (Fig. 3). However, it negatively correlated with O-alkyl and alkyl carbon ($r = -0.65^*$ and -0.21^* , respectively) (Fig. 3). Although aromatic carbon content in soil organic carbon structure was very low, it had the most effect on the K_{OC} of diuron because the aromatic carbon is non-polar carbon and diuron is non-ionic pesticide. This was consistent with [30], who found that the aromatic carbon had a positive correlation with the K_{OC} of five pesticides (Imidacloprid, Clothianidin, Thiacloprid, Methidathion and Chlorpneb). Likewise, [15] showed a positive correlation between the aromatic carbon and the K_{OC} of diuron and a negative correlation between O-alkyl carbon and Alkyl carbon.

Conclusions

Diuron adsorption in four pineapple-growing soils was in the range 1.81-27.63%, and the adsorption data fit well with the Freundlich model. Soil organic matter

Table 6. The correlation coefficients (r) between the adsorption coefficient (K_F) and soil properties

Parameter	Sand	Silt	Clay	pH	OM	CEC	Total. N	Avail. P	Avail. K	ECe
K_F	-0.90**	0.90**	0.78**	0.53	0.95**	0.80**	0.93**	0.18	-0.53	0.22

**Correlation is significant at the 0.01 level (2-tailed).

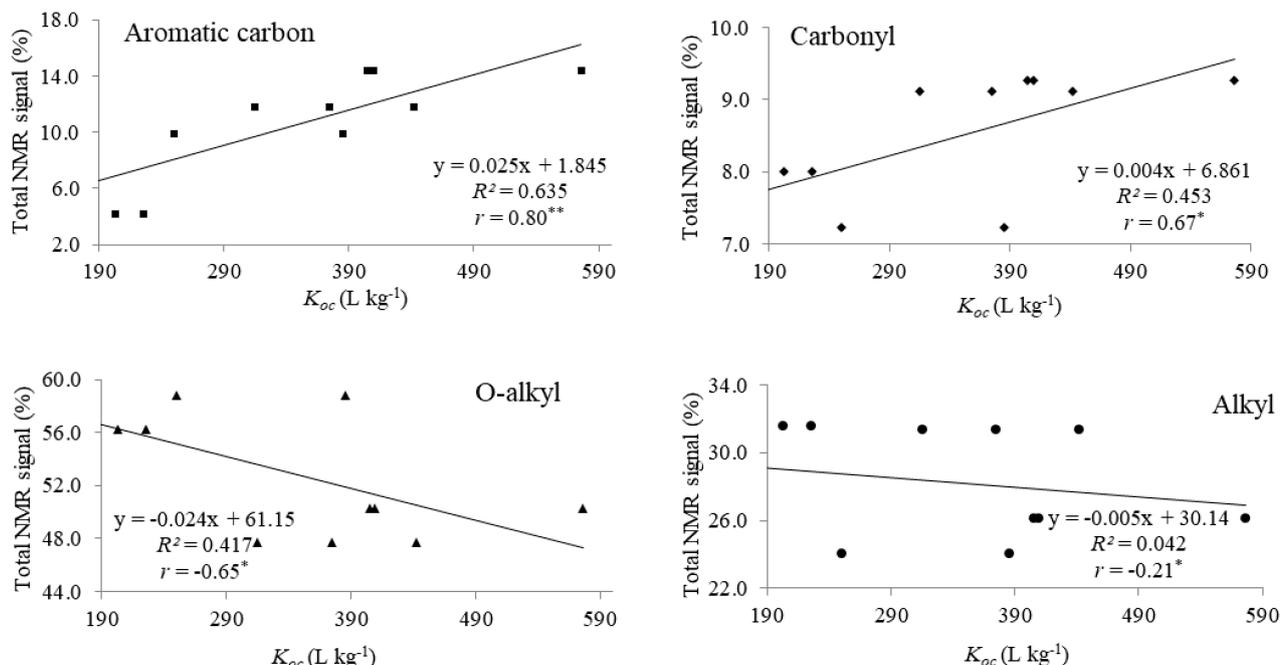


Fig. 3. Correlation coefficients (r) for organic carbon partitioning coefficient (K_{OC}) of pineapple-growing soils and percentage of total NMR sign.

* Correlation is significant at the 0.05 level (2-tailed).

** Correlation is significant at the 0.01 level (2-tailed).

and clay content were the main factor that influenced the adsorption of diuron in these soils, although the soils had low soil organic matter and clay contents. Total nitrogen, silt content and cation exchange capacity had a positive correlation with diuron adsorption in these soils, but sand content showed otherwise. Organic carbon partitioning coefficient of the soils had a significantly positive correlation with aromatic carbon and carbonyl in soil organic matter; however, it negatively correlated with O-alkyl carbon and alkyl carbon. Low diuron adsorption in these pineapple-growing soils posed a risk of diuron and perhaps other pesticides used in the cultivation contamination in the environment. A proper practice on increasing organic matter content in these soils would be ideal in terms of decreasing this risk.

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Conflict of Interest

The authors declare no conflict of interest.

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