The Effect of Glufosinate Ammonium in Three Different Textured Soil Types under Malaysian Tropical Environment
(Kesan Glufosinat Ammonium pada Tiga Jenis Tanah Berbeza Tekstur dalam PersekitaranTropika Malaysia)

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ABSTRACT

Glufosinate ammonium is a broad spectrum, non-selective, contact and organophosphate herbicide which is commonly used in Malaysian oil palm plantations. Research area was one of the oil palm growing areas of Malaysia is located adjacent to the Tasik Chini, Pahang. Farmers use this herbicide to control several types of unwanted plants which could compete with the oil palm for nutrients. Rain water and the sprayed solution are easily adsorbed by soil particles. The glufosinate ammonium sorption was determined by the batch equilibrium technique. The collected soil samples (0-50 cm depth) divided into five groups at 10 cm depth intervals. Glufosinate ammonium adsorption coefficients were correlated with the soil pH, organic matter (OM), clay content, and cation exchange capacity (CEC). Series of glufosinate ammonium standard were as 0.01, 0.1, 0.25, 0.5, 1, 3, 5, and 10 µm/mL. The Linear and Freundlich equations were fitted for obtaining the adsorption and desorption isotherms. The result of the analyses showed that adsorption of glufosinate ammonium was correlated to the clay content. The clay fraction of the soil is the main absorbent of the glufosinate ammonium. Desorption from the soil was indicated by the high binding strength of the adsorbed glufosinate ammonium.

Keywords: Adsorption; desorption; glufosinate ammonium; linear equation; sorption isotherm

ABSTRAK

Glufosinat ammonium adalah herbisid yang berspektrum luas, tidak memilih, jenis sentuhan dan di dalam kumpulan organofosfat yang sering digunakan dalam ladang kelapa sawit di Malaysia. Tapak kajian adalah di kawasan penanaman kelapa sawit yang terletak bersebelahan dengan Tasik Chini, Pahang. Petani menggunakan herbisid ini untuk mengawal beberapa jenis rumpai yang mengganggu tanaman dan boleh bersaing dengan pokok kelapa sawit untuk mendapatkan nutrien. Air hujan dan larutan herbisid mudah terserap oleh zarah tanah. Penjerapan glufosinat ammonium ditentukan oleh teknik keseimbangan berkelompok. Sampel tanah yang dikumpulkan (kedalaman 0-50 cm) dibahagikan kepada lima kumpulan pada jarak 10 cm kedalaman. Koefisien penjerapan ammonium glufosinat dikaitkan dengan pH tanah, bahan organik (OM), kandungan tanah liat, dan kapasiti pertukaran kation (CEC). Siri piawai amonium glufosinat ialah 0.01, 0.1, 0.25, 0.5, 1, 3, 5 dan 10 µm/mL. Persamaan Linear dan Freundlich dipasang untuk mendapatkan isoterma penjerapan dan penyahjerapan. Hasil analisis menunjukkan bahawa penjerapan amonium glufosinat dikaitkan dengan peratusan kandungan tanah liat. Sebahagian tanah liat tanah adalah penjerap utama glufosinat ammonium. Penyahjerapan daripada tanah ditunjukkan oleh kekuatan ikatan penjerapan glufosinat ammonium yang tinggi.

Kata kunci: Glufosinat ammonium; isoterma penjerapan; penjerapan; persamaan linear; penyahjerapan

INTRODUCTION

Different types of unwanted plants like woody perennial herbs, broad-leaved weeds and grasses are the main problem in oil palm plantations. Malaysia is the second largest palm oil exporting country in the world. Glufosinate ammonium is the most commonly used herbicide in the plantation sector (especially oil palm) to eradicate weeds and unwanted plants. In the 1980s glufosinate was introduced to the Malaysian pesticide market (Halimah et al. 2016; Ismail et al. 2015c, 2013). Currently more than 60 countries of the world use glufosinate ammonium which is registered under the names: Finale®, Rely®, Challenge® and Basta®. In Malaysia glufosinate ammonium is known under the trade name Basta®.

The adsorption and desorption behaviour of the herbicide in soil can caused surface and ground water pollution. Many herbicides are strongly attached to soil particles because of their low solubility and high hydrophobicity (Ismail et al. 2015a; Tayeb et al. 2016; Zablotowicz et al. 2008). The adsorption/desorption or sorption of chemicals to the soil represents an important environmental process which affects the environmental fate of the chemicals. The migratory propensity of herbicides emphasizes the extent of herbicide sorption on environmental components such as soil, sediment, air and water. The leaching of herbicides through the soil column, photo-degradation, volatilization from soil and water as well as their concentration in natural water are essential

information required to estimate the sorption data. The transport pathway and bio availability of herbicides depend on their adsorption characteristics. A problem occurred when a research confirmed that the goosegrass biotype was the first case whereby the development of multiple resistance across the three non-selective herbicides, namely glyphosate, glufosinate and paraquat (Chuah et al. 2018). Nonetheless, this problem made the farmer use the same herbicides in higher concentration with the purpose of killing the weeds.

Herbicide adsorption in a tropical environment decreases its concentration in aqueous solutions. Laboratory determination of the adsorption and desorption nature of herbicides is necessary for estimation of the potential hazards they pose to the environment, especially to surface and groundwater resources (Nagatomi et al. 2013; Screpanti et al. 2005). Studies on adsorption and desorption of herbicides in Malaysian tropical agricultural soils are relatively new and limited (Chang & Liao 2002; Laitinen et al. 2008). Only a few studies on the persistence of glufosinate ammonium in Malaysian agricultural soils have been reported (Ismail et al. 2015b; Jariani et al. 2010). The present study aimed at reinforcing the content of existing literature and knowledge.

MATERIALS AND METHODS

CHEMICALS AND REAGENTS

The glufosinate ammonium standard was obtained from Dr. Ehrenstorfer (GmbH Germany). The basic properties and chemical structure of GLUF are shown in Table 1 and Figure 1, respectively.

HPLC grade acetone, acetonitrile and diethyl ether were purchased from Merck (Darmstadt, Germany). The analytical grade reagents such as potassium dihydrogenphosphate (KH_2PO_4), potassium hydroxide (KOH), disodium tetraborate decahydrate ($N_2B_4O_7.10H_2O$), 9-Fluorenylmethyl Chloroformate (FMOC-Cl) and hydrochloric acid (HCL, 37.9%) were purchased from sigma Aldrich. The standard stock solution of glufosinate ammonium (200 μg/mL) made up of ultra-pure water and

0.01M FMOC-Cl was prepared in acetone. Acetone, 0.125M borate buffer solution & 0.01M FMOC-Cl were used for the pre column derivatization.

COLLECTION OF SOIL SAMPLES

The collected soil samples represented the soil types of the Tasik Chini oil palm plantation. The soil samples were collected randomly from different locations at five soil depths of 10, 20, 30, 40, and 50 cm. Air dried soils were sieved by a 2 mm sieve. The Pipette Method (Day 1965) was used for soil texture determination. The soil textural class was determined using the Calgon dispersing agent and the USAD soil textural triangle. Soil CEC was determined by the leaching method (Method 9080) (EPA 1986). The Walkley and Black Method was used for soil organic carbon (OC) determination. Organic carbon of the soils was determined by the Walker-Black method (Allison 1965). The Blackman Digital pH meter was used to measure soil pH from the soil water suspensions (1:1v/v). The clay mineral content was analysed using the X-ray diffraction technique (XRD) (Figure 2).

The physico-chemical properties of collected soil samples were shown in Table 2. The clay loam soil texture contains high organic matter than other two soil texture. Clay soil contains highest CEC (14.79).

ADSORPTION AND DESORPTION EXPERIMENTS

The batch equilibrium technique was used for sorption isotherms as it permitted convenient evalution of the parameters that influenced the sorption process (OECD 2000). The analyses were completed using 7 different concentrations of the supplied glufosinate ammonium standard and they were done in triplicate. Sieved soil (2 gm) was transferred into 50 mL polypropylene tubes. Then, 20 mL solution containing 0.25, 0.5, 1, 3, 5, 7, and 10 μg/mL of glufosinate ammonium standard were added in the same tube. At room temperature, the tubes were equilibrated for 24 h. After equilibration, the tubes were centrifuged for 20 min at 3000 rpm. Equilibrium concentration of the supernatant was determined by HPLC-FLD. The adsorption isotherm was fitted to the Linear and Freundlich equations and expressed

TABLE 1. Features of glufosinate ammonium

Chemical structure	Molar mass	Water solubility	Dissociation constant (P^{Ka})
$C_5H_{15}N_2O_4P$	198.19	>500g/L at 20°C	9.15±0.07
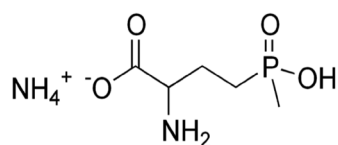
(Reference: http://www.chemicalbook.com/Chemical product Property CB2697882)

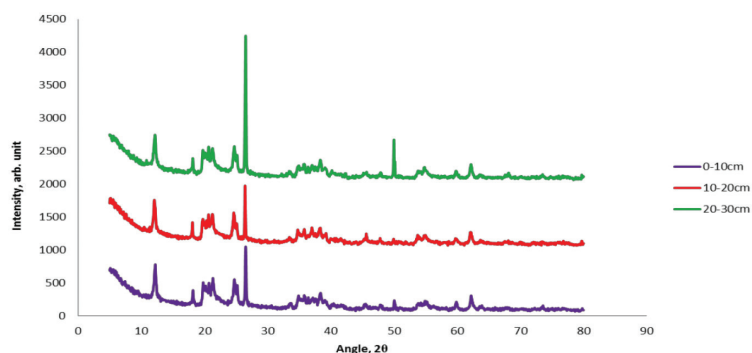FIGURE 1. Structural formula of glufosinate ammonium

FIGURE 2. Soil mineral content as determined XRD

TABLE 2. Average soil characteristics in the study area

Soil texture	pH	Organic matter (%)	Organic carbon (%)	Cation exchange capacity (cmol/kg)	Bulk density
Clay	4.20	3.53	2.41	14.79	1.08
Silty clay	4.24	3.58	2.55	9.54	1.13
Clay loam	4.33	4.67	3.38	5.67	1.20

as follows $Cs = \log Kf + {}^{1}/_{nf} \log Ce$. Adsorbed herbicide Cs, $\mu g/g$ and adsorption difference were $\mu g/L$ were calculated. After the adsorption experiment, desorption determination was done immediately.

DERIVATIZATION AND HPLC ANALYSIS

The clear supernatant solutions were subjected to pre column derivatization before the HPLC analysis was done. One (1) mL supernatant was derivatized by 0.125M borate buffer at 0.8 mL, acetone, 0.8 mL and 0.01M FMOC-Cl. The supernatant was swirled 30 seconds and left on the table for 30 min for chemical reactions to take place. The procedure was done in room temperature. After 30 min, the sample was washed with 1 mL Diethyl ether to remove the excess of FMOC-Cl and then placed on a plane surface until two layers formed (Ibanez et al. 2005; Sancho et al. 1994). After formation of the two layers, a 1 mL sample

was carefully collected from the bottom layer and filtered using a 0.25 μm syringe filter. The filtered sample was analysed using the HPLC - Fluorescence detector (FLD). (The FLD λ ex = 263 nm & λ em= 310 nm. The HPLC was Agilent 1220 infinity LC and the analytical column was EC 250/3 NUCLEODUR C_{18} Gravity, 5 μm with gradient system). The mobile phase used was Acetonitrile: 0.2% phosphoric acid solution (35:65). The mobile phase pH was maintained by using 2M KOH and 1M HCL. The derivatization process is shown in Figure 3.

RESULTS AND DISCUSSION

GLUFOSINATE AMMONIUM STANDARD CALIBRATION CURVE

Figure 4(a) shows that the linear regression (r2) obtained was 0.99. The correlation equation Y=239.44x+5.21

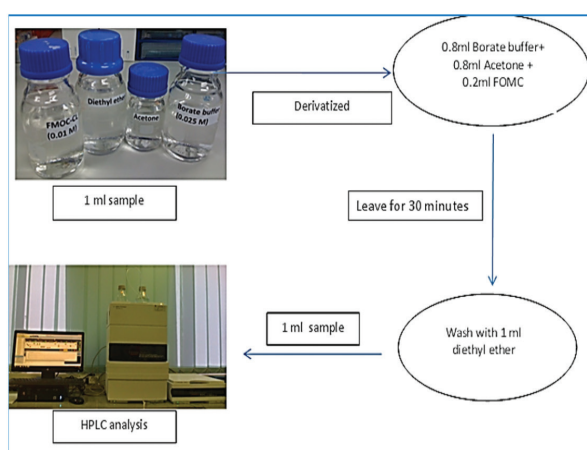FIGURE 3. Derivatization process of glufosinate ammonium

was prepared using the calibration data, where X and Y represented the concentration (μg mL^{-1}) and area of glufosinate ammonium. The retention time (5.855) of glufosinate ammonium standard is shown in Figure 4(b).

The chromatogram of the herbicide glufosinate ammonium was obtained during analysis of the calibration curve. For better recovery, the detector wavelengths were monitored as $\lambda_{em} / \lambda_{em}$ 260/310, 263/317, 265/317, 267/320 and 270/320. Based on the peak height and area it was observed that the maximum absorbance and best selectivity peak of glufosinate ammonium was at detector λ_{ex} = 263 nm / λ_{em} = 317 nm wavelength. The minimum detection limit was 0.001 μg mL^{-1}.

ADSORPTION EQUILIBRIUM TIME

With reference to clay particles, glufosinate ammonium was more strongly adsorbed to silty clay and clay loam particles as shown in Figure 5. The adsorption to silty clay and clay loam was 1.5 times higher than the adsorption to organic matter (Druart et al. 2011). The glufosinate ammonium adsorption to clay loam and silty clay particles became very slow and constant after it reached the equilibrium position among the soil particles and supernatant. After shaking for 3 h, the equilibrium position was achieved. At the initial stage herbicide adsorbed the outer surface of soil particle. Electrostatic binding played an important role in glufosinate ammonium sorption process (Ismail et al. 2015b). Internal surface diffusion and external surface depletion was the reason for decreased sorption at the later stage (Yun et al. 2014). The percentage glufosinate ammonium adsorbed on to the soil particles after 3 h was more than 90% for all the three types of soil tested. A similar result to the glufosinate ammonium adsorption patterns was found (Allen-King et al. 1995; Yun et al. 2014). The adsorptive equilibrium of glufosinate ammonium was attained within 75 to 100 min in the clay type soils. The result is supported by that of Accinelli et al. (2004). The earlier mentioned studies reported that glufosinate ammonium has similar equilibrium time in different soil types and it ranged from 45 to 180 min.

SOIL pH EFFECT ON ADSORPTION

Glufosinate ammonium adsorption on the previously mentioned soil types were affected by soil pH. Three pH numbers (2, 7 & 11) were investigated in this experiment. It was observed that the adsorption of glufosinate
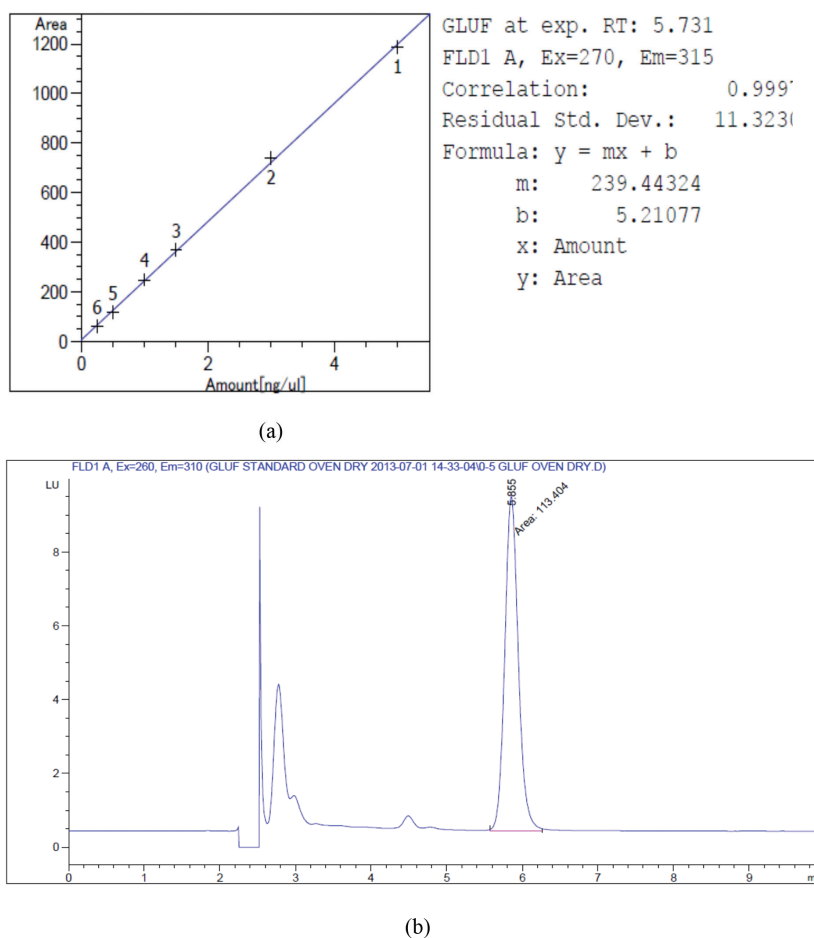

(a)

(b)

FIGURE 4. (a) Linear regression of glufosinate ammonium standard (HPLC-FLD) and b) Chromatogram of glufosinate ammonium standard (generated by HPLC-FLD) during analysis of the calibration curve at 0.25 μgmL^{-1} concentration
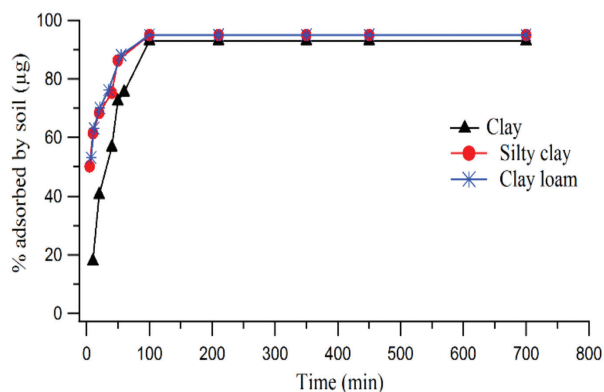
FIGURE 5. Adsorption equilibrium of glufosinate ammonium in clay, silty clay, clay loam soils of Tasik Chini

ammonium was highest at higher pH levels, for the three soil types, (the results are supported by Gerhartz & Markus 2010). At pH11, the adsorption percentages of glufosinate ammonium adsorbed on to the clay, silty clay and clay loam soils were 91.37, 92.4, and 92.9%, respectively, while, at pH 2, the adsorption percentages were not more than 84.12% for the three soil types. The statistical analysis, ANOVA (analysis of variance) showed a significant effect of pH ($P \leq 0.05$) on glufosinate ammonium adsorption on the three soil types. Among the three soil types glufosinate ammonium adsorption was slightly higher on the silty clay and clay loam soils compared to that on the clay textured soil.

Glufosinate ammonium adsorption increased with the increase of pH and the number of negative charges. The number of negative charges increased because of H^+ ion loss from the clay particle surface. The clay particle surface displayed negative charges (pH>2.0) on account of pH-dependent surface hydroxyl groups of variable charges. When the pH increased, the negative charges also increased, and this result is compatible with the findings of the study where adsorbed amounts were reported to be dependent on increase in pH (Corbett et al. 2004; Tayeb et al. 2015). These observations are in agreement with those of Tsuji et al. (1997), where it was reported that at high pH, glufosinate ammonium was strongly adsorbed to clay particles in the soil.

When soil pH decreases, the value of the adsorption coefficient K_d also decreases. This is compatible with studied soil pH have an effect on sorption. The distribution coefficient K_d, which is empirically derived, decreases when pH decreases. High K_d value indicated strong bonding of glufosinate ammonium with soil particle.

EFFECT OF TEMPERATURE ON ADSORPTION

Temperature has a significant effect on adsorption by soils through its effect on solubility, and this adsorption has been shown for parathion by Behrendt et al. (1990) as well as for atrazine (Dinehart et al. 2009; Ismail et al. 2015d). Druart et al. (2011) explored the dissimilarity in the adsorption mechanisms of the protonated and ionized forms of pentachlorophenol (PCP) by standardizing their adsorption coefficients at three different temperatures (4°C, 25°C and 55°C). In the case of the neutral form, a trend was observed whereby there was at decrease in K_d with increasing temperature (Kah & Brown 2006). Tseng et al. (2004) observed that the K_d value increased for chlorosulfuron when the temperature decreased from 30°C to 10°C. In the present study, clay, silty clay and clay loam soils sorption coefficient, K_{oc} and K_f decreased with rise in temperature from 30°C to 40°C, probably due to the effect of temperature on the weak binding between glufosinate ammonium and the soil particles (Table 3). A similar behavior was observed for glufosinate ammonium on the same soils. Temperature had an exiguous effect on adsorption. The total percentage glufosinate ammonium adsorbed on the three mentioned soil types was significant (P≤0.05) when temperature increased from 30°C to 40°C. These results are supported by those of the previous reports (Goodwin et al. 2003; Qian et al. 2011; You & Barker 2004, 2002).

Glufosinate ammonium was strongly adsorbed and inactivated by the soil clay particles. It averted the build-up of the active surface when it came into close contact with the soil particles. The glufosinate ammonium positive charge was strongly drawn to the negative charge of soil clay particles (Gallina & Stephenson 1992; Ismail et al. 2015e; Tebbe & Reber 1991).

TABLE 3. Sorption coefficient K_d, K_{oc} and K_f of three soil texture at 30, 35 and 40°C for glufosinate ammonium

Soil	Temperature (°C)	K_d	K_{oc}	K_f
Clay	30	1265.32	25.89	10.12
	35	1000.79	20.88	4.99
	40	439.21	9.08	1.23
Silty clay	30	892.31	51.12	16.77
	35	470.26	469.98	5.98
	40	320.05	319.54	2.33
Clay loam	30	893.45	51.60	16.89
	35	471.11	26.67	6.23
	40	320.61	17.57	2.45

EFFECT OF HERBICIDE CONCENTRATION ON ADSORPTION

Table 4 shows the glufosinate ammonium adsorption at different concentrations ranging from 0.10.5 ug g^{-1}in three different soil types. In clay 6.29, 13.25, 30.11, 53.45, 158.33 and 265.05 μgg^{-1} glufosinate ammonium were adsorbed; in silty clay soil 6.62, 13.43, 30.67, 53.72, 158.87 and 265.25 μgg^{-1} glufosinate ammonium were adsorbed. In clay loam 6.71, 13.97, 30.82, 53.59, 158.95, 265.25 μgg^{-1} glufosinate ammonium were adsorbed. The concentrations used were 0.1, 0.3, 0.5, 1, 3 and 5 μgmL^{-1}, respectively. The adsorption results for the above three types of soils showed no significant difference. The sequences of the studied soils were as follows: clay loam > silty clay > clay for the samples analyzed (in triplicate). The analysis of variance (ANOVA) showed that glufosinate ammonium adsorption was significantly affected ($P<0.05$) by the different concentrations in the three types of soil. These results were supported by those of Qian et al. (2011). Shin et al. (2011) used XRD crystallography for analyzing the clay fractions that contained adsorbed glufosinate ammonium.

The binding strength of glufosinate ammonium with soil particles is attributed to the soil adsorption coefficient K_d. Glufosinate ammonium adsorption to clay soil ($r^2 = 0.988$), silty clay ($r^2 = 0.895$) and clay loam ($r^2 = 0.893$), was obtained using the Freundlich adsorption isotherm as in Figure 6(a), 6(b) & 6(c). The K_f & K_d values of the three types of soil were as follows: 382.87, 881.42 Lkg^{-1}; 366.10; 911.05 Lkg^{-1}, and 359.98, 924.10 Lkg^{-1}; the 1/n values were 1.32, 1.41 and 1.48 (Table 5). The 1/n values for the three

TABLE 4. Glufosinate ammonium adsorption % in three different textured soils (n=5)

Soil type	Initial amount (μgmL^{-1})	Amount absorbed (μgg^{-1})
Clay	0.1	6.29(±0.05)
	0.3	13.25(±0.21)
	0.5	30.11(±0.02)
	1	53.45(±0.01)
	3	158.33(±0.05)
	5	265.05(±0.03)
Silty clay	0.1	6.62(±0.01)
	0.3	13.43(±0.04)
	0.5	30.67(±0.05)
	1	53.72(±0.03)
	3	158.87(±0.02)
	5	265.25(±0.01)
Clay loam	0.1	6.71(±0.05)
	0.3	13.97(±0.03)
	0.5	30.82(±0.01)
	1	53.59(±0.01)
	3	158.95(±0.04)
	5	265.25(±0.03)

types of soil were quite similar, indicating that increasing the solution concentration resulted in increased adsorption of the compound. The results showed high adsorption of the glufosinate ammonium onto the three soil types (Yun et al. 2014). Glufosinate ammonium adsorbs easily to
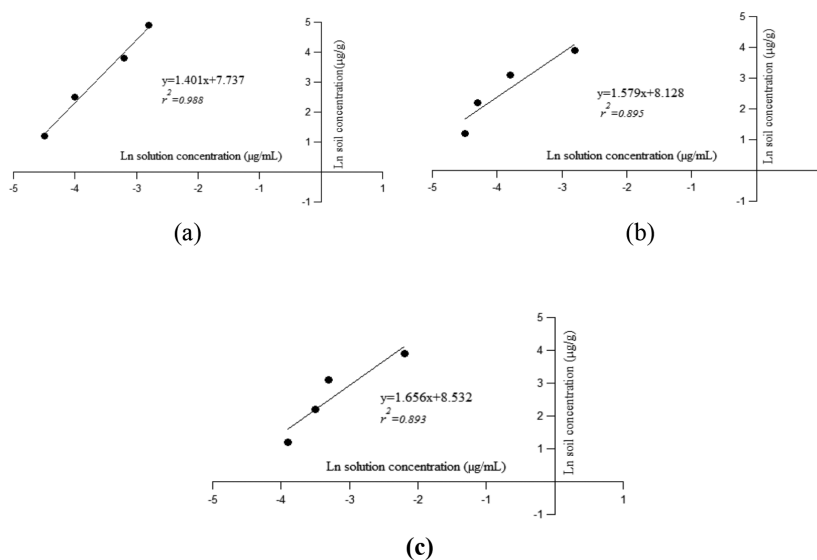

FIGURE 6. Glufosinate ammonium adsorption in (a) clay soil, (b) silty clay soil and (c) clay loam soil

TABLE 5. Percentage OM and K_d, K_f, 1/n values for the adsorption of glufosinate ammonium in soils

Soil Type	%OM	r^2	K_f	1/n	K_d
Clay	2.13	0.988	382.87	1.3	881.42
Silty clay	4.05	0.895	366.10	1.4	911.05
Clay loam	4.22	0.893	359.98	1.4	924.10

the negatively charged soil particles. The mechanism of glufosinate ammonium adsorption is charge transfer ionic bonds (Druart et al. 2011). The soil sorption coefficient K_d value was correlated with soil properties such as soil organic carbon (K_{oc}) (Weber et al. 2004). The (K_{oc}) value is not useful, when the ionic herbicide glufosinate ammonium binds with soil particles by electrostatic mechanisms involving soil mineral and organic matter (OM).

CONCLUSION

The herbicide adsorption process binds glufosinate ammonium with clay particles in a manner similar to that of iron fillings getting attached to a magnet. Experimental studies were carried out to investigate the soil pH, temperature and glufosinate ammonium concentration of its sorption behaviour on three selected soil texture. The adsorption behavior of glufosinate ammonium under the Malaysian tropical environment showed that it could be modeled well by the Freundlich isotherm. Glufosinate ammonium adsorption was found to be affected by temperature, and adsorption capacity significantly increased as the temperature was reduced. However, results demonstrated that the total amount of glufosinate ammonium adsorption onto the three mentioned soils was not significant ($p \leq 0.05$). Glufosinate ammonium remained stable in the soil samples up to 2 months when stored at -20°C. However, due to the high adsorption and low desorption tendency, the potential environmental risk connected with glufosinate ammonium movement from the soil column to ground water would be minimal.

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