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Mobility risk and desorption irreversibility of glyphosate and aminomethylphosphonic acid in highly weathered tropical soils of Sri Lanka

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Abstract

Glyphosate (GLY) and its primary metabolite aminomrthylphosphonic acid(AMPA) are among the most used and reported residues in the vicinity of application, yet the environmental dissipation behaviours in mineral-rich weathered tropical soils, particularly the mobility risk, are insufficiently studied. Sorption- desorption hysteresis of GLY and AMPA was characterised across 15 wet-zone tea estate soils in Sri Lanka using single-point Freundlich isotherms, with the objective of evaluating the site-specific mobility risk. Sorption experiments were conducted based on the batch equilibrium method, and residual glyphosate was analysed by LC-MS/MS. Adsorption was confirmed to follow pseudo-second-order kinetics and the Freundlich isotherm in all soils, consistent with a hybrid chemisorption process on heterogeneous surfaces and metal oxides-dominated surfaces. In contrast, desorption was slow and partial, exhibiting a significant hysteresis attributed to inner-sphere complexation at Fe and Al surfaces. A negative hysteresis with values greater than unity in 73% (GLY) and 27% (AMPA), but a crossover hysteresis pattern in log curves- in which the desorption isotherm exceeded the adsorption isotherm at low concentrations but converged at higher loadings- was observed for the same, indicating concentration -dependent irreversibility. Therefore, two novel indices- the Hysteresis Control (HCI) and the normalised Mobility Risk Index (MRIn,100) were introduced to integrate the hysteresis behaviour, desorption potential, and mineralogical buffering capacity into a unified risk framework. Alkaline, sandy-textured soils with low Fe and Al oxide content demonstrated very high risk (MRIn,100 = 100), while acidic, mineral-rich, high-clay tropical soils with less desorption effectively immobilise both compounds (MRI_{n100} <0.02) with less bioavailability.

Keywords: Hysteresis, Desorption, Glyphosate, Mobility risk, AMPA, Freundlich

Introduction

Glyphosate (GLY) based formulations, chemically known as N-(phosphanomethyl)glycine, are among the most extensively utilised as broad-spectrum weedicides globally, due to cost-effectiveness, which is attributed to their non-selective and post-emergent properties(Meftaul et al., 2021). GLY inhibits 5-enolpyruvylshikimate-3-phosphate synthase (EPSPS) enzyme in the shikimate pathway, resulting in preventing the synthesis of aromatic amino acids and effectively controlling annual and perennial weeds. Consequently, found in widespread applications in agriculture, horticulture, silviculture, and urban weed management. In addition, the global usage of glyphosate has increased significantly following the introduction of glyphosate-resistant crops. Aminomethylphosphonic acid (AMPA), the primary metabolite of GLY degradation, co-occurs with the parent compound in glyphosate-applied soils. Yet, the environmental fate is far less studied than the parent compound. Therefore, a simultaneous assessment of GLY and AMPA requires evaluating their fate in agriculturally important soils.

The widespread use of glyphosate has increased the concern of unintentional contamination in the environmental compartments in the vicinity, and causes a noticeable threat to water resources and aquatic ecosystems(Lacroix & Kurrasch, 2023; LanctÔt et al., 2014; Meftaul et al., 2021). The concern of contamination is also associated with the surfactants used in commercial glyphosate formulations to increase the solubility and permeability of glyphosate in plant tissues. The surfactant, polyethoxylated tallow amine (POEA), which is frequently found in commercial formulations, is reported as more toxic than glyphosate itself (Meftaul et al., 2021; Tush & Meyer, 2016).

Among primary environmental dissipation pathways of glyphosate, such as adsorption, biodegradation, photodegradation, leaching, and transport, adsorption is considered to be the most critical, as it governs the bioavailability and persistence in soil(Napoli et al., 2016). The adsorption and desorption of glyphosate have been studied extensively in temperate soils through batch experiments based on the Organization for Economic Co-operation and Development (OECD) 106 guideline, and fewer studies were found with field lysimeter experiments (Bergström et al., 2011; Borggaard & Gimsing, 2008; Gimsing et al., 2004; Kanissery et al., 2019; Nomura & Hilton, 1977; Pereira et al., 2019; Sidoli et al., 2016; Vereecken, 2005). The Freundlich and Langmuir models have been used to evaluate the isotherms, and the reaction kinetics were often studied by using pseudo-second order kinetic models. Previous findings reported a positive correlation of K_{ads} with Iron and Aluminium oxide content, moisture, clay content, and temperature, while a negative correlation was observed with soil pH(Cáceres-Jensen et al., 2009; Hiera Da Cruz et al., 2007; Remlinger et al., 2023). In addition, the phosphate in the soil has been reported to compete with glyphosate for binding sites, resulting in a negative correlation (Divisekara et al., 2018; Munira et al., 2018). Interestingly, the values reported in previous studies for K_{ads} and the Freundlich constants are changing considerably with the soil type. However, the majority of the reported literature originates from temperate soils of Europe and North America, where illite, smectite, and vermiculite mineralogy dominate in soils that are fundamentally different from kaolinite, gibbsite, and Fe/Al oxide-rich soils found in weathered tropical soils.

Attention to the sorption-desorption hysteresis, which contributes to the persistence and leaching of GLY and AMPA in soils, is inadequate even in temperate soils. Hysteresis is the non-coincidence of adsorption and desorption isotherms due to different rates, paths, and/or mechanisms associated with both processes (Graziano et al., 2023; Mamy & Barriuso, 2005). When the desorption process diverges from adsorption, the reversibility is affected and

influences the pesticide persistence, bioavailability, and eventually the risk of groundwater and environmental contamination. Therefore, conventional pesticide mobility indices such as the organic-carbon normalized distribution (KOC), Groundwater Ubiquity Score (GUS), and Dissociation constant (K_d) may not be suitable for the risk evaluation. (Vereecken, 2005). The formation of inner sphere complexation with Fe/Al oxides in soil clay, metastable sorbed states with soil organic matter, and concentration-dependent heterogeneous binding sites have been reported as contributing factors to GLY hysteresis (Graziano et al., 2023; Mamy & Barriuso, 2005; Padilla & Selim, 2020). A positive hysteresis is observed when the adsorption exceeds the desorption, resulting in HI values less than unity, indicating a strong irreversibility. However, when the desorption exceeds the adsorption, a negative hysteresis is observed, characterised by a HI greater than unity, which is advantageous for the desorption. The HI closer to unity represented a reversible process, where the rates of adsorption and desorption are more or less similar(Barriuso et al., 1994; Wu et al., 2024).

Additionally, the tropical weathered soils consist of unique physicochemical characteristics such as variable mineralogy, acidity, organic carbon content, and metal oxide content, which may alter the glyphosate adsorption and desorption process differently than in temperate soils. The elevated temperature and frequent rain have introduced pH-dependent variable-charge soil surfaces and subsequently influenced the adsorption of zwitterionic GLY. Notwithstanding the rapid increase in the use of GLY in South and Southeast Asian tropical agricultural systems, published data on GLY and AMPA adsorption-desorption remain limited. In Sri Lanka, GLY is extensively used in Tea and rubber plantations, and due to the systemic nature of GLY, residues of glyphosate can be transported to commercial products. Given the significant role of the tea industry in the Sri Lankan economy and the economic threat posed by glyphosate residues, which are used for weed control at low labour costs, this study seeks to examine the adsorption, desorption, and hysteresis of Glyphosate in wet zone tea estate soils. Additionally, it aims to assess the risk of mobility and environmental contamination.

Materials and Methods

Materials

The reference standards of Glyphosate and AMPA were purchased from LGC Standards, and commercial formulations were generously provided by the Registrar of Pesticides, Sri Lanka. LCMS grade Methanol was ordered from VWR- UK, and $CaCl_2$ was taken from Sigma-Aldrich, USA.

Soil Sampling

Soil samples were collected from tea estates having records of the use of glyphosate and from uncontaminated soils within the same area having the same soil texture. A composite sample was collected by collecting samples using a transect sampling approach since tea fields are arranged on a slope, and one slope is maintained as a separate field. Therefore, sampling points were selected from the top to the bottom of the field along a transect line. GPS coordinates of the top of the hill were recorded(*Guidelines on Soil and Vegetation Sampling for Radiological Monitoring | IAEA*, n.d.; *JOINT MEETING OF THE CHEMICALS COMMITTEE AND THE WORKING PARTY ON CHEMICALS,*

PESTICIDES AND BIOTECHNOLOGY Guidance Document for Conducting Pesticide Terrestrial Field Dissipation Studies Series on Testing & Assessment No. 232 Series on Pesticides No. 82 JT03391244, 2016).

The top litter layer was removed, and approximately 500 g of soil sample was taken from the A horizon (10 cm depth) using a spade. Then all samples in a single field were collected into a single container and sent to the lab. Upon receipt at the lab within 24 hr, soil moisture, soil pH, and clay content were measured after homogenising. One-half of the soil was air-dried over three days, large particles removed, and sieved through a 2 mm sieve before being preserved under refrigerated conditions until the pH measurement and adsorption study. The other half was kept under frozen conditions for long-term storage.

Soil Characterization

Samples were homogenised, and 10.0000 ± 0.0005 g was accurately measured and oven-dried at 102 ± 2 °C until a constant mass was obtained(*Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass*, 2019). The organic carbon content was calculated for 2.000 ± 0.001 g accurately measured soil by oven drying at 103 ± 2 °C for 4 hr followed by heating at 450 ± 25 ℃ for 6 h. The oven-dried weight and final weight were used to calculate the organic matter and converted to organic carbon content by dividing by 1.724(*Organic and Organo-Mineral Fertilisers. Determination of the Total Organic Carbon (TOC) Content by Dry Combustion*, 2022).

The pH of the soil samples was measured in ASTM type I water and 0.01 mol L^{-1} $CaCl_2$ solution. Approximately 10 g of air-dried soil sieved through a 2 mm sieve was measured into a glass beaker, and approximately 10 mL of ASTM type I water or 0.01 mol L^{-1} $CaCl_2$ solution was added. The slurry was mixed thoroughly and allowed to settle for 1 h. The pH was measured in the settled samples with a calibrated pH meter(*Test Methods for PH of Soils*, 2024).

The iron, aluminium, calcium, and magnesium contents of the sample were determined using an in-house-validated method involving microwave digestion followed by ICP-MS. The clay content of the samples was determined using ASTM D 7928 by using a 152H soil hydrometer(Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis, 2021). Soil samples were sieved through an ASTM No 10 sieve to determine the soil texture, and soil collected in the pan was taken for the hydrometer analysis.

Analysis of Glyphosate and AMPA

The analytical methods employed were adapted from our previous research using a Sciex QTRAP 4500 LC-MS/MS. Glyphosate-spiked $CaCl_2$ solutions were analysed following a method adopted by Dissanayake et al (2022), which has a Limit of Quantification (LOQ) of 0.1 µg L^{-1}. Additionally, the method described in the Proceedings of the 4th Biennial Research Symposium of ITI, Sri Lanka (2019) by Dissanayake et al. concerning the direct determination of GLY and AMPA in soil by Liquid Chromatography Tandem Mass Spectrometry- Single Laboratory method validation, was used for soil samples achieving an LOQ of 0.025 mg kg^{-1}. (Dissanayake et al., 2022).

Adsorption/ Desorption kinetics and isotherm Studies

Adsorption/desorption and isotherm studies were conducted according to the OECD 106 guidelines at 27±2 ℃ room temperature(Meftaul et al., 2021; *Test No. 106: Adsorption -- Desorption Using a Batch Equilibrium Method*, 2000). The soil solution ratio was selected as 1:5, as this ratio provided measurable residues at the end of the equilibrium and reached more than 90% adsorption.

A precisely measured 2.00 ± 0.05 g (n=3) of soil was placed into a 50 mL Falcon tube, followed by the addition of 10.0 ± 0.1 mL of 0.01 mol L^{-1} $CaCl_2$. The pre-equilibrated samples were spiked on the following day, and shaking continued for another 24 hours. Two samples were randomly selected and removed at predefined intervals.

Adsorption kinetics

Samples were removed at 2, 4, 6, 8, and 24 h intervals, assuming equilibrium was reached after 24 hours. Immediately after collecting samples at various time points, they were subjected to centrifugation at 8000 rpm (r 4 cm, 10,000g) for 10 minutes at room temperature using a fixed-angle rotor. Subsequently, 100.0 ± 0.1 µL of the supernatant was mixed with 9.9 ± 0.1 mL of 1% acetic acid prepared in ASTM type I water and stored in a refrigerator until the study concluded. In the final step, all diluted samples were passed through 0.22 µm syringe filters and analysed isochronously using LC-MS/MS. The residual concentration of glyphosate in the solution over time was calculated, and the adsorption percentage (A%), distribution coefficient (Kd), and adsorption kinetics and adsorption isotherms were evaluated as per OECD 106.

$$A\% = \left(\frac{m_{soil}^{ads}}{m_0}\right) * 100\ (\%) \quad \text{(E1)}$$

Where A% is the adsorption percentage at the time point ti (%), m_{soil}^{ads} is the mass of glyphosate adsorbed on the soil at the time ti (µg) m_0 *is* mass of glyphosate in the falcon tube at the beginning of the test (µg)

$$Kd = \frac{A_{eq}}{(100-A_{eq})} \times \frac{V_0}{m_{soil}}\ (cm^3\ g^{-1}) \quad \text{(E2)}$$

Where Aeq is the percentage of adsorption at adsorption equilibrium, V_0 is the initial volume, and m_{soil} is the mass of soil used for the experiment.

Desorption kinetics

Following the 24-hour adsorption test, the $CaCl_2$ solution in the Falcon tube was removed and filled with fresh unspiked solution, and the test was continued according to the OECD 106, and the samples were treated as explained above and analyzed using LC-MS/MS.

The desorption is defined as the percentage of the test substance that is desorbed, related to the quantity of substance previously adsorbed, under the test conditions. The percentage desorption was calculated according to E3.

$$D\% = \frac{m_{aqs}^{des}\ (t_1)}{m_s^{ads} \times (eq)} \times 100\ (\%) \quad \text{(E3)}$$

where: D% is the desorption percentage at a time point ti (%), m_{aqs}^{des} is the mass of the test substance desorbed from soil at a time point ti, (µg), m_s^{ads} is the mass of the test substance adsorbed on soil at adsorption equilibrium (µg).

Kinetics studies assessed how quickly glyphosate reaches equilibrium. The adsorption and desorption kinetics of both glyphosate and AMPA were evaluated by linearised pseudo-first-order (PFO), linearised and non-linear pseudo-second-order (PSO) models. Model performances were evaluated by the coefficient of determination (R^2) and the sum of squared errors (SSE).

The linearised PFO equation can be given as follows,

$$\log(q_e - q_t) = \log(q_e) - k_1 t \,. \quad \text{(E4)}$$

The linearised PSO model is represented as follows,

$$\frac{t}{q_t} = \frac{1}{k_{2n} q_e^2} + \frac{1}{q_e} \quad \text{(E5)}$$

Where q_e is the amount of glyphosate adsorbed or desorbed at equilibrium (mg kg^{-1}), q_t is the amount of glyphosate/AMPA adsorbed or desorbed at time t (mg kg^{-1}), k_1 is the pseudo-first-order rate constant, k_2 is the pseudo-second-order rate constant, and t is the time in contact with the test solution in minutes.

However, in desorption, both PFO and PSO linear kinetics parameters resulted in unrealistic negative values; thus, non-linear PSO kinetics models were run to calculate the kinetic constants. The non-linear model of PSO is given in E6.

$$q_t = \frac{k_{2n} q_e^2}{1 + k_{2n} q_e t} \quad \text{(E6)}$$

Where, q_e is the amount of glyphosate adsorbed or desorbed at equilibrium (mg kg^{-1}), q_t is the amount of glyphosate/AMPA desorbed at time t (mg kg^{-1}), k_2n is the pseudo -second-order nonlinear rate constant (min^{-1}), and t is the time in contact with the test solution in minutes.

Adsorption and Desorption Isotherms

$CaCl_2$ solution was spiked at 5, 10, 25, 50, 75, 100, and 150 mg $L^{-1,}$ and all samples were shaken at 75 rpm for 24 h. After 24 h, following sample preparation, the kinetics study was explained until the LC-MS/MS isochronous analysis. The residual concentration of glyphosate and AMPA was used to evaluate the applicability of the Freundlich and Langmuir Isotherm models. The Freundlich adsorption/desorption equation can be given as,

$$C_s^{ads/des}\,(\text{eq}) = K_F^{ads/des} \times (C_{aq}^{ads/des}\,(eq))^{1/n}\ (\mu g\ g^{-1}) \quad \text{(E7)}$$

Linear form can be written as,

$$log\ C_s^{ads/des}\ (\text{eq}) = log\ K_F^{ads/des} + \frac{1}{n} log C_{aq}^{ads/des}\ (eq)\cdot \quad \text{(E8)}$$

Where, $K_F^{ads/des}$ is Freundlich adsorption/desorption coefficient (mg $^{1-1/n}$ $L^{1/n}$ g^{-1}), n is regression constant and $C_{aq}^{ads/des}$ is mass concentration of the substance in the aqueous phase at adsorption/desorption equilibrium (µg cm-3).

The hysteresis index was calculated by taking the ratio of (1/n des) / (1/n ads).

Mobility Risk Assessment

A mobility risk assessment was conducted as per the conventional Gustafson groundwater ubiquity score based on Koc and two new experimental and mechanistic indices introduced based on pedological effects and hysteresis.

Gustafson groundwater ubiquity score (GUS) = log_{10} (DT50) × $[4 - log_{10}K_{OC}]$ (E10)

DT0 – time taken to reduce to half the amount of the pesticide from the initial concentration

Organic Carbon Water partition coefficient, K_{OC} = K_d/ OC (E11)

Hysteresis Control Index (HCI) = $(Fe + Al) \times Clay / p^H$ (E12)

Mobility Risk Index (MRI) = $(D\% / HI \times HCI)$ (E12)

Statistical Evaluation

The results were statistically evaluated by Minitab, IBM SPSS, and Datatab statistical software packages. Minitab was used to conduct Tukey's Post Hoc Analysis to identify significant differences between glyphosate RS and commercial formulations. A cluster analysis was conducted on pedological parameters, and a dendrogram was generated to identify similar soil types. The correlation of pedological parameters and the adsorption-desorption coefficient was evaluated by conducting Pearson's Correlation, and heatmaps were generated by the Datatab software. Finally, Partial Least Squares Regression (PLSR) analysis was conducted for adsorption and desorption data to identify the influencing pedological factors.

Results and Discussion

The pedological properties of soil samples were measured as soil samples received at the lab, and the average values are given in the supplementary material, as Table 1 (Physicochemical properties of soil samples) as ESM_1.

A hierarchical Cluster analysis was performed with pedological parameters, and it classified soil samples into three main clusters. Cluster 1 consists of soils containing very high soil minerals, high moisture content, and high clay values (S3A, S3B, S3C, and S1B). The second cluster consists of alkaline soils containing high pH, low Fe and Al content, low organic carbon content, and low clay content (S4B and S4C). The third cluster displayed mixed and

moderate physicochemical properties and was further subdivided into three subgroups. (S1A, S1C, S2A, S2B, S2C, S4A, S5A, S5B, S5C). The dendrogram is given in Fig. 1.

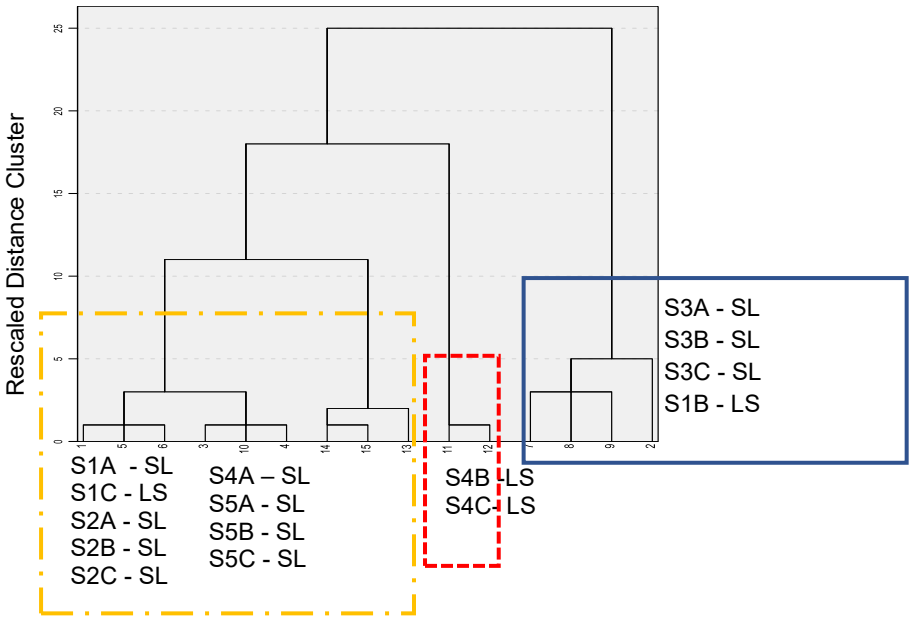


Fig 1 Dendrogram on Soil Classification on pedological parameters
USDA Classification SL- Sandy Loam soil, LS – Loamy Sand soil

Overall, despite the limited variability among soil types for both compounds, a consistently strong adsorption was observed. The percentage adsorption of glyphosate and AMPA in 12 of the 15 soil samples exceeded 98.4% and 96.5%, respectively, whereas the remaining three soil samples displayed noticeably lower adsorption, 75.6% and 82.3%, respectively. Fig. 2 summarises the adsorption and desorption behaviour of glyphosate and AMPA.

Even though the adsorption process is fast and attained within 24 h, the desorption process was slow and low in most of the soils for both glyphosate and AMPA, and has not reached equilibrium within 24 h.

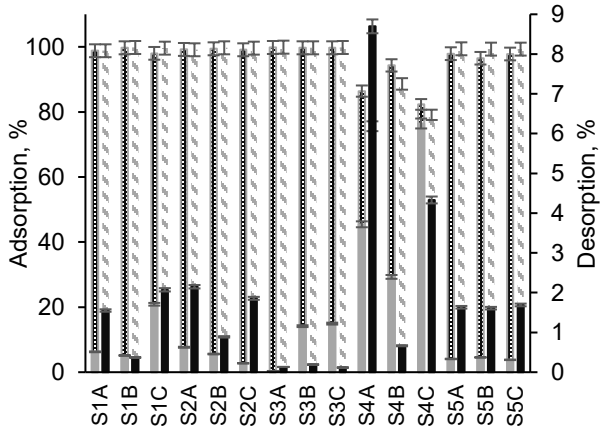


Fig 2. Adsorption and Desorption percentages of GLY and AMPA

(A% - Percentage adsorption-Black dots – AMPA, Grey lines- GLY, D% - percentage desorption - Black – AMPA, Grey- GLY)

A reciprocal relationship of adsorption and desorption was observed, as soils that demonstrated a strong adsorption also demonstrated the lowest desorption, and soils with lower adsorption evinced comparatively higher desorption.

The effect of surfactants in the glyphosate formulation on Adsorption

A one-way ANOVA analysis was carried out to check the significant difference among the three spiking solutions, and Tukey's HSD and Dunnett's comparisons with the control glyphosate reference standard were applied. The Dunnett's and Tukey's grouping information for the three glyphosate sources spiked in SC3 and SC4 soils at 50 mg kg-1 is given in Table 2. A significant difference was not observed between the reference standard and formulations; thus, the average of all 9 replicates was considered in the adsorption studies. Further, this observation emphasises that the surfactant molecules in the formulations do not influence the unique binding sites involved in the adsorption of glyphosate.

Table 2 The comparison of Tukey's and Dunnett's grouping data of soil spiked with three glyphosate sources

Soil	Dunnett's †			Tukeys'††		
	Level	Mean n=3	Grouping	Level	Mean n=3	Grouping
S3A	Kd RS (Control)	3774	A	Kd RS	3774	A
	Kd F1	3779	A	Kd F1	3779	A
	Kd F2	3755	A	Kd F2	3755	A
S4A	Kd RS (Control)	19.7	B	Kd RS	19.7	B
	Kd F1	19.0	B	Kd F1	19.0	B
	Kd F2	18.8	B	Kd F2	18.8	B

†Means not labeled with letter A are significantly different from control level mean.

††Means that do not share a letter are significantly different.

RS- Reference standard, F – commercial formulation

The correlation of pedological parameters on the adsorption of glyphosate and AMPA

Pearson's correlation analysis was performed to identify any correlation of the adsorption of glyphosate/ AMPA with soil physicochemical parameters after standardising the values. The correlation heatmaps of adsorption of glyphosate (a) and AMPA (b) are given in Fig. 3, and desorption heatmaps are given in Fig. 4. A strong positive correlation was observed between Kd and Mg content, organic carbon (OC), Al content, Clay content, and Fe content in the soil. This correlation highlights the collective influence of mineralogical and chemical soil properties on glyphosate/AMPA adsorption.

The positive association of Kd with Fe and Al suggests the predominance of metal oxide/metal hydroxide surface complexation, as reported in many previous studies (Borggaard & Gimsing, 2008; Padilla & Selim, 2020; Sidoli et al., 2016).

These observations are aligned with chemisorption predominance and are further evident by the positive correlation with clay content. The sharp negative correlation between soil pH and Kd emphasises the pH dependence of glyphosate/AMPA adsorption. Alkaline conditions promote deprotonation of soil surfaces and glyphosate, increasing the competition for metal oxide binding sites. Consequently, adsorption decreases with increasing pH.

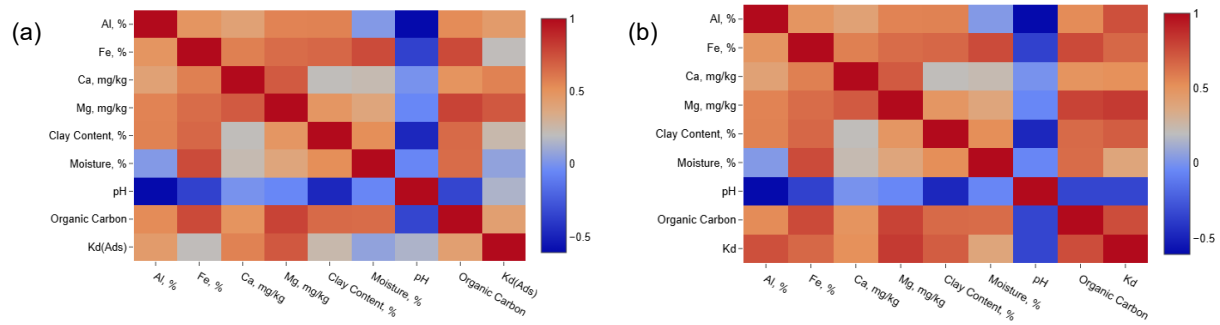


Fig 3 Correlation heatmap on soil physicochemical parameters and adsorption distribution coefficients of GLY (a) and AMPA (b) (Al%- percentage of total aluminium, Fe%- percentage of total Iron, Ca - Calcium, Mg- magnesium, K_d - Distribution coefficient)

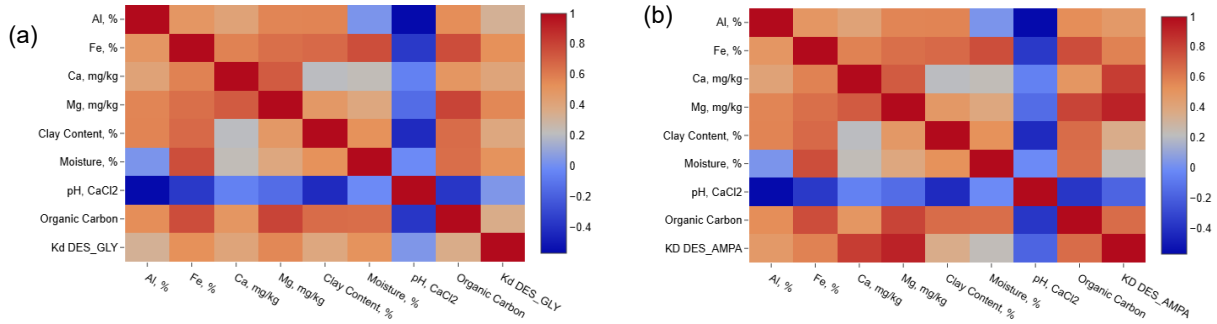


Fig 4 Correlation heatmap on soil physicochemical parameters and desorption distribution coefficients of (a) Glyphosate (b) AMPA

Al%- Percentage of total aluminium, Fe%- Percentage of total Iron, Ca -Total Calcium, Mg- Total magnesium, K_d - Distribution coefficient in the soil

A partial least squares regression analysis was conducted to identify the most influential factors on adsorption and desorption. The VIP plot for both glyphosate and AMPA is given in Table 3. The latent factors greater than one indicate a highly important variable for the adsorption and desorption of glyphosate and AMPA. Thus, Moisture%, Clay content %, Organic carbon%, Fe%, Al%, and Calcium contents have the strongest influence on adsorption and desorption of glyphosate and AMPA.

To further understand the underlying mechanisms of adsorption-desorption of glyphosate and AMPA with soil, identify the controlling processes and governing factors, the experimental data were subsequently analysed with kinetic and isotherm models.

Table 3 Variable Importance in the Projection calculated for pedological parameters and Adsorption and Desorption Parameters of Glyphosate and AMPA

	Latent Factors	
Variables	Glyphosate	AMPA
Al %	1.025	1.219
Fe %	1.002	0.952
Ca, mg kg^{-1}	1.118	1.270
Mg, mg kg^{-1}	0.923	1.156
Clay, %	1.194	1.275
Moisture, %	1.207	0.736
pH_Water	0.650	0.612
pH_CAL	0.707	0.719
Organic Carbon, %	1.075	0.967

Kinetic Modelling of Adsorption and Desorption

The speed of the adsorption of glyphosate and AMPA into the soil matrix is explained by kinetics. A near-unity coefficient of determination and Qe values aligned with the experimental values suggested a satisfactory goodness-of-fit of the adsorption data of glyphosate and AMPA with the pseudo-second order (PSO) kinetic model. While the desorption of both glyphosate and AMPA was aligned only with the non-linear pseudo-second order in both the coefficient of determination and the realistic degradation constant values. This observation further strengthens the results of previous studies on the applicability of the PSO kinetic model for adsorption and desorption through chemisorption (Besghaier et al., 2021; Dissanayake Herath et al., 2019; Qiu et al., 2024; Sethi et al., 2024).

A comparison of Kd, adsorption percentages, and PSO adsorption kinetics of glyphosate and AMPA is given in Table 4 (A summary of the results obtained from the adsorption and desorption kinetics of glyphosate and AMPA) in the supplementary material (ESM_2). Three different patterns can be identified in kinetic rates and Kd values, and the speed and strength of the adsorption were evaluated, respectively. Soils with low clay and mineral contents exhibited slow, weak adsorption, whereas high clay, organic carbon, and mineral-rich soils showed fast, strong adsorption. Furthermore, moderately fast adsorption is observed in some soil samples with modest clay and mineral content. The results are well aligned with the soil groups given in the dendrogram.

Freundlich Isotherm

The experimental values fit the Freundlich isotherm model with a goodness-of-fit greater than 0.95, indicating a heterogeneous surface with non-uniform binding energies in glyphosate and AMPA adsorption. The K_F of samples having higher organic carbon content, higher mineral amounts, and higher moisture percentage is comparatively higher. In addition, the Freundlich intensity parameter (1/n), across all soils, is less than one, implying a favourable adsorption governed by heterogeneous binding sites and multilayer adsorption (Dissanayake Herath et al., 2019; Meftaul et al., 2021; Padilla & Selim, 2019). The heterogeneity of adsorption in the Freundlich model evidences the availability of multiple sorption domains, including mineral surfaces, oxide coatings, and organic matter (Borggaard & Gimsing, 2008; Gimsing et al., 2004; Ololade et al., 2014; Padilla & Selim, 2020). A summary and comparison of

the Freundlich parameters of glyphosate and AMPA are given in Table 5 (A summary of the Freundlich Isotherm parameters obtained from the adsorption and desorption of glyphosate and AMPA) is in the supplementary material (ESM_3).

The desorption processes of both glyphosate and AMPA behaved in a different manner, as the lowest $K_{F\ DES}$ soil (S4C and S4A, respectively) contained the lowest amounts of Al, Fe, clay, and organic carbon. However, the highest $K_{F\ DES}$ soil (S3A and S2C) is not the highest of all the above properties, and reported a combination of moderate to high values. This is clear evidence for the heterogeneous nature of glyphosate desorption.

Hysteresis and Mobility Risk Assessment

Subsequently, a considerable asymmetry is evident when comparing the Freundlich parameters between adsorption and desorption, indicating the presence of hysteresis. The Freundlich exponent/intensity factor 1/n reflects the surface heterogeneity and adsorption intensity, and the HI is calculated by taking the ratio of the Freundlich intensity of desorption and adsorption.

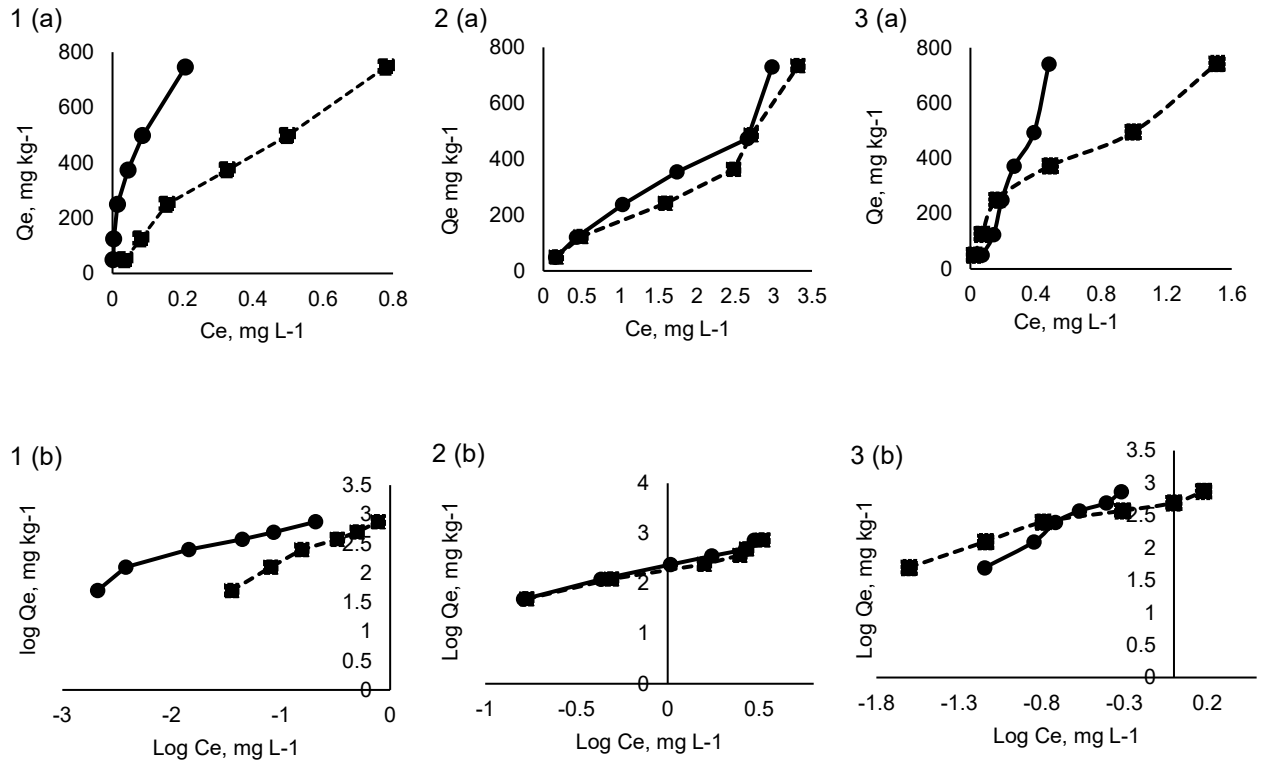


Fig 5 Hysteresis 1 (a) Positive Curves (Glyphosate in S3A), 1 (b) Positive log curve (Glyphosate in S3A), 2 (a) Reversible Curves (AMPA in S1B), 2 (b Reversible log curve (AMPA in S1B), and 3 (a) Negative Crossover Curves (Glyphosate in S2B), 3 (b) Negative Crossover log curves (Glyphosate in S2B). Adsorption- Black dashed-line, square points, Desorption - Black, round points

When the HI is unity or closer to one, it indicates reversible adsorption. When HI > 1, it indicates negative hysteresis, and more compounds are desorbed than predicted from adsorption. While HI<1 demonstrates a strong irreversible condition. (Besghaier et al., 2021; Cáceres-Jensen et al., 2009; Graziano et al., 2023). This study observed three main patterns for both compounds, and the hysteresis curves and the respective log curves selected from soil samples are given in Fig. 5.

Four soils for glyphosate and seven soils for AMPA demonstrated positive hysteresis out of 15 soils, signifying strong irreversibility. Hysteresis arises due to the fractional desorption or selective release of weakly bound glyphosate, leaving a strongly bound fraction remaining on the soil(Barriuso et al., 1994; Raman et al., 1988; Rojas et al., 2022). The incomplete desorption may result from the inner-sphere complexation of glyphosate with iron and aluminium oxides. Interestingly, ten soils for glyphosate and four soils for AMPA exhibited a dual hysteresis with a crossover.

The HI of all these soils was greater than one, but the desorption curve lies above the adsorption curve at low concentrations, and the position of the desorption curve switched, and it went to the top at high concentrations. This kind of observation has been limitedly reported, and a similar phenomenon was reported for sorption hysteresis of Atrazine in sandy loam soils. The authors proposed the initial negative hysteresis due to the desorption of compounds from the interlayer or intraquasi-crystal water layer. Therefore, a steep low-concentration desorption could be generated, but a higher number of compounds is sequestered in the inner metal oxide layers. (Anagu et al., 2011; Barriuso et al., 1994). Finally, the cumulative effect of all these processes explicates the slow and incomplete desorption (Besghaier et al., 2021; La Cecilia & Maggi, 2018).

Generally, pesticide risk assessment is conducted by using GUS, which has a combined effect of both DT_{50} and K_{oc}. However, the calculated Koc values for glyphosate and AMPA are far above 4000, and mobility is classified as essentially immobile, and GUS becomes negative.

Therefore, in this study, we introduced new indices to evaluate the mobility and risk of contamination by glyphosate and AMPA. Since the glyphosate retention was influenced by metal oxides, clay content, and soil pH, the following hysteresis control index (HCI) was developed. HCI interprets how feasible the hysteresis is.

The mobility risk index was introduced to evaluate the combined effect of desorption potential, adsorption hysteresis, and mineralogical control on glyphosate retention.

The MRI was normalised, scaling it to a 0 to 100 range. The proposed risk indices and normalised MRI are given in Table 6. The mobility risk index score can be categorised as <1 as very low, 1 to 10 as low, 10 – 50 as moderate, 50–80 as high, and >80 as very high.

A noteworthy observation arises in soil S4B, where, despite exhibiting a positive HI, the $MRI_{n,100,}$ ranked as the second highest among all soils. This apparent condition is solved by the disproportionately high desorption percentage and the lowest HCI in the data set, which, combined, override the moderating influence of positive hysteresis in the MRI formulation.

Table 6 Mobility Risk assessment of glyphosate and AMPA

Soil			GLY					AMPA				
Code	Texture	HCI	D%	HI	Graph	$MRI_{n\ 100}$		D%	HI	Graph	$MRI_{n\ 100}$	
S1A	SL	315	0.51	0.9	POS	0.6	VL	1.6	1.1	R	4.0	L
S1B	LS	1472	0.42	0.8	POS	0.2	VL	0.4	1.0	R	0.2	VL
S1C	LS	524	1.71	1.4	CRS	1.3	L	2.1	1.0	R	3.3	L
S2A	SL	581	0.63	1.9	CRS	0.7	VL	2.1	1.0	R	3.1	L
S2B	SL	507	0.46	1.9	CRS	0.7	VL	0.9	1.3	CRS	1.2	L
S2C	SL	573	0.22	1.8	CRS	0.2	VL	1.9	1.6	CRS	1.8	L
S3A	**SL**	**3812**	**0.03**	**0.7**	**POS**	**0.0**	**VL**	**0.1**	**1.1**	**CRS**	**0.000**	**VL**
S3B	SL	5199	1.16	1.0	CRS	0.2	VL	0.2	1.1	CRS	0.003	VL
S3C	SL	2283	1.22	1.1	CRS	0.4	VL	0.1	0.9	POS	0.019	VL
S4A	LS	573	3.72	1.5	CRS	5.0	L	8.7	0.9	POS	14.0	M
S4B	LS	172	2.40	0.7	POS	17.0	M	0.7	0.6	POS	5.5	L
S4C	**LS**	**41**	**6.25**	**1.3**	**CRS**	**100.0**	**VH**	**4.3**	**0.9**	**POS**	**100.0**	**VH**
S5A	SL	443	0.33	1.3	CRS	0.5	VL	1.6	0.9	POS	3.6	L
S5B	SL	803	0.37	1.5	CRS	0.3	VL	1.6	0.9	POS	2.0	L
S5C	SL	696	0.31	1.4	CRS	0.3	VL	1.7	0.9	POS	2.4	L

D% - Desorption percentage, HI – Hysteresis Index, HCI – Hysteresis Controlling Index, Norm MRI_{100}- Normalised Mobility Risk Index score, SL- Sandy Loam, LS – Loamy Sand

This is a good example that hysteresis alone is insufficient to characterise mobility risk, and the magnitude of desorption and the mineralogical buffering capacity of the soil are equally decisive.

Among soils exhibiting crossover hysteresis (CRS), 72% of soils were ranked as VL risk for GLY mobility, reflecting the dominant role of HCI values in surpassing the net desorption despite complex hysteresis behaviour. Nevertheless, AMPA mobility in the same soils exhibited comparatively elevated risk: Only 26% and 60% of all soils were identified as VL and L risk soils, respectively, confirming that AMPA mobility cannot be inferred from GLY pattern. Interestingly, soil S4C, which has a crossover HI for GLY and a positive HI for AMPA, returned the highest $MRI_{n,100}$ score for both compounds, driven by the lowest HCI and the highest desorption in the dataset. The combined effect of alkaline pH, minimal mineral content, and coarse loamy sand texture is reflected in the lowest HCI; conversely, the SA3 soil recorded the lowest MRIn, 100 with an exceptionally high HCI, low desorption percentage, and positive hysteresis.

The proposed MRI, therefore, integrates desorption potential, hysteresis behaviour, and soil mineralogical control into a single, interpretable risk score that outperforms any individual parameter in discriminating high-risk from low-risk soils across contrasting soils.

Conclusion

In this study, sorption-desorption hysteresis of 15 Sri Lankan tropical soils was studied and revealed a predominantly concentration-dependent Crossover hysteresis pattern for GLY(11/15 soils), fitting with the Freundlich Isotherm and second-order kinetics models. AMPA, in contrast, demonstrated a predominant positive hysteresis (8/1 soils) fitting with the same isotherm and kinetics models. The compound-level divergence in the hysteresis within the same soil demonstrated that AMPA mobility cannot be inferred through GLY sorption behaviour, and simultaneous characterisation is essential.

The novel hysteresis control index (HCI) and mobility risk index (MRI) across all soils demonstrated that the conventional adsorption-based indices, such as GUS, classify GLY as essentially immobilised due to the extremely high K_{OC} values, substantially misinterpret the mobility of GLY in alkaline sandy soils with low mineral content. HCI encapsulates the combined influence of mineral content, clay content, and soil pH on desorption reversibility, and soils with higher HCI values consistently demonstrated the lower mobility risk for both compounds. The $MRI_{n,100}$ index integrates the experimental desorption percentage, with mechanistic pedological parameters, and redefines the effect on hysteresis into a single risk score.

MRI identified SC4, which represents the lowest clay, Fe, and Al content, alkaline sandy loam soil, as the highest risk soil reaching the normalised ceiling of 100 for both compounds. The least MRI represented an acidic, mineral-rich soil with low desorption and positive hysteresis, indicating a very low mobility risk. These findings indicate that the primary determining factors of the mobility risk of GLY and AMPA in tropical soils are soil mineralogy, pH, and texture, which are integrated into a single model by HCI. The MRI framework provides a more collective and mechanistically grounded tool for understanding the mobility risk of GLY and AMPA in a given site than existing indices.

Declarations

All authors have read, understood, and complied as applicable with the statement on "Ethical responsibilities of Authors" as found in the Instructions for Authors.

All authors contributed to conducting the study.

D A T W K Dissanayake – Funding Acquisition, Conceptualisation, Project administration, Formal data analysis, Statistical analysis, Writing-original draft, and editing.

P Ranasinghe – Project Administration, Statistical Analysis, Review, editing, and supervision

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Competing Interest

The authors have no relevant financial or non-financial interests to disclose.

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Supplementary material

Additional heatmaps and data tables are given in the supplementary material.

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