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# DGT measurements for the evaluation of cadmium and lead flux and bioavailability in texturally different contaminated soils as function of time

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## Abstract

*Phytoavailable metals are referred to the fraction of total metals in soil that plants can easily uptake, potentially affecting environmental health and contributing to food chain. The DGT (Diffusive Gradients in Thin Films) time-based estimations are very effective for measurement of bioavailable Cd and Pb. The current study focused on the deployment time of DGT devices on two texturally different (sandy clay loam, SCL, and clay loam, CL) soils contaminated with Cd and Pb at the rate of 25 and 50 mg kg$^{-1}$, respectively. Deployment time for DGT devices was maintained as 2, 4, 8, 12, 24, and 48 hours, with three replications per soil. For the SCL soils S1, S2, and S3, the coefficients of determination ($R^2$) between $C_{DGT}$ of Cd and deployment time were 0.7153, 0.8221, and 0.7153, respectively. In the same soils, the $R^2$ values for Pb were lower, at 0.4422, 0.5030, and 0.4611, for S1, S2, and S3, respectively. While in the CL soil (S4), the $R^2$ value for Cd was 0.5544, while Pb showed a slightly higher $R^2$ of 0.5050. SCL soil showed a stronger linear trend for Cd than for Pb, while in CL soil*, *Pb exhibited the higher linearity compared to Cd. Cd consistently displayed higher $R^2$ values, indicating a more predictable decline of $C_{DGT}$ with deployment time, and 48-h deployment is adequate for Cd to attain quasi-equilibrium with the soil solution. Overall, Cd demonstrated clearer time-dependent trend across soil types, whereas Pb exhibited lower and more inconsistent correlations, reflecting its slower desorption kinetics and stronger soil binding.*

**Keywords:** DGT measurement, Deployment time, Bioavailability of Cd and Pb, Regression correlation

## Introduction

Contamination of agricultural soils with heavy metals (HMs) has been a persistent global issue for many years. These metals pose serious threats to environmental quality, food safety, and human health (Hou *et al.*, 2025; Sharafi and Salehi, 2025). In the suburbs of metropolitans, the deficiency of canal waters is supplemented with raw city effluents (Ali *et al.*, 2023). Among multiple heavy metals, agricultural soils are mainly receiving lead (Pb) and cadmium (Cd) through industrial wastewater discharge, smelting, atmospheric deposition, and sewage sludge (Angon *et al.*, 2024; Mitran *et al.*, 2024; Nieder and Benbi, 2024). Pb and Cd are of major concern because of their high toxicity, persistence, and ability to accumulate in plants and reaches the human body (Shaghaleh *et al.*, 2024; Huang *et al.*, 2025). The Cd and Pb exist as free ions and complexed species, with their chemical speciation regulating their mobility and ability to be absorbed by plants (Ondrasek *et al.*, 2020; Riaz *et al.*, 2021; Rahman *et al.*, 2024). Processes such as precipitation, adsorption, dissolution, desorption, and ligand complexation regulate the distribution and availability of metals (Bali *et al.*, 2020; Riaz *et al.*, 2021; Petruzzelli *et al.*, 2025). The harmful effects of Cd and Pb on plants depend heavily on their soil mobility and the extent to which they are absorbed by plants (Liu *et al.*, 2024; Rahman *et al.*, 2024).

Since the toxicity, bioavailability, and mobility of metals are dependent on their chemical forms rather than their total concentrations, analyzing metal speciation is essential for accurate environmental risk assessment (Laha *et al.*, 2024). Various methods have been proposed to assess contamination levels, exposure risks, and toxicological threats of metals (Ali *et al.*, 2023; Shi *et al.*, 2023). Primary approaches for assessing the availability of HMs in soil include methods of chemical extraction and soil solution analysis (Bai *et al.*, 2023). The reliability of soil solution and chemical extraction techniques, including sequential and single extractions, remains contested topic of contention. Dissolved organic matter commonly binds soil pore water with heavy metals for

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reducing their availability and leading to potential overestimation by the soil solution method (Kumkrong *et al.*, 2022; Forsyth *et al.*, 2024; Jadhav and Medyńska-Juraszek, 2025). Challenges such as trace metal concentrations, sewage irrigated contamination risks, and different textual characteristics of soils highlight the need for reliable, simple, and advanced methods to analyze trace element availability in soil. The DGT method provides time-integrated, in situ assessments of available metals in undisturbed soil (Fang *et al.*, 2024; Xie *et al.*, 2021). DGT measurements are highly effective technique to evaluate the bioavailability of various HMs, including Cu, Mn, Pb, Zn, Se, Cd, Sb, and As, as well as essential nutrients like S and P in soils (Zhu *et al.*, 2023; Senila and Kovacs, 2024).

The DGT operates without disturbing the soil or requiring chemical treatments; it preserves the natural speciation and mobility of metals, yielding a more accurate picture of the labile metal pool (Marrugo-Madrid *et al.*, 2021; Senila *et al.*, 2022). The DGT device itself consists of a binding resin (typically Chelex containing iminodiacetate functional groups), a diffusive hydrogel layer, and a 0.45 µm filter. Diffusion of metal ions to the binding phase occurs across the filter and gel layers, where they are immobilized and subsequently measured (Rougerie *et al.*, 2021; Lin and Pan, 2023). DGT effectively accumulates labile metal species from soil, showing strong correlations with metal concentrations taken up by plants (Bai *et al.*, 2023; Chen *et al.*, 2024). The DGT measurements have been extensively used to assess the bioavailability of different HMs in undisturbed soils, but most studies have been focused on single parameter of acidic to neutral soils (Zhang and Davison, 2015; Ma *et al.*, 2022). However, multiple soil textures, alkalinity, deployment time, and multiple HMs have not been examined under the same umbrella. The current study offers novel insights by comparing Pb and Cd availability in wastewater-irrigated, aged, and freshly spiked soils with distinct physicochemical properties at different deployment times. The objectives of the current study are (i) comparing Pb and Cd availability in aged-contaminated soils and spiked soils through DGT measurements; (ii) evaluating how soil texture influences DGT-derived metal fluxes and effective concentrations ($C_{DGT}$) in alkaline calcareous soils; and (iii) identifying the DGT deployment time that best reflects the available metal fractions in each soil type.

## Materials and Methods

### Soil sampling and analysis

Agricultural fields in the peri-urban regions of the Multan and Faisalabad districts in Punjab, which have been irrigated with untreated municipal wastewater for over 20 years, were selected for this study. Samples of composite soil were obtained from the upper 15 cm of the soil layer, placed in labeled polyethylene bags, and carried into the laboratory of the University of Agriculture, Faisalabad. The soil samples were kept in the open air to dry, followed by crushing to pass through a sieve with a 2 mm mesh. Labeled samples were put in plastic containers and kept at room temperature until they were analyzed. The available metal concentrations were initially measured by extracting the soil using an ammonium bicarbonate-diethylene triamine pentaacetic acid (AB-DTPA) solution buffered at pH 7.6 by Soltanpour (1985). Among different heavy metals, Pb and Cd were analyzed by running the samples extracted volumes on an Atomic Absorption Spectrophotometer AAS (Hitachi Z-8200 Polarized Zeeman model, the blank sample readings were 0.0001 $mg\ kg^{-1}$, recovery rate for Cd and Pb 95% and 93.5%, respectively, and the precision was up to 5-6% based on the coefficient of variation). Soil saturated paste was prepared for soil pH determination by the pH meter (Jenway 671P, UK). The extract obtained from a saturated soil paste was used to determine the soil's electrical conductivity (EC). The ratio of sodium adsorption (SAR) was measured according to the standardized procedures established by the US Salinity Lab Staff (1954) and those proposed by Page *et al.* (1982). The contents of calcium carbonate in the soil were measured by employing the calcimeter procedure (Moodie *et al.*, 1951).

### Spiking of soil with Cd and Pb

The prepared clay loam (CL) and sandy clay loam (SCL) soils sampled from Multan and Faisalabad districts were divided into four groups. Clay loam soil from Multan district was aged-contaminated, while sandy clay loam soil from Faisalabad was both spiked with Cd and Pb at 25 and 50 $mg\ kg^{-1}$ using $3CdSO_4 \cdot 8H_2O$ and $PbSO_4$, respectively, and aged-contaminated. Metal salts were evenly applied to the soil and thoroughly mixed. The spiked soils were kept in a wire house under controlled sunlight and aeration, maintaining temperatures of 25-30 °C and 70% relative humidity. The soils were stored in a shaded area for 60 days, with regular addition of distilled water to ensure uniform moisture maintenance, distribution, and stabilization. To minimize evaporation and allow the system to reach equilibrium, the soils were fully covered with plastic sheets during this period. After spiking, the homogeneity of metals in the soil was validated to ensure uniform distribution (ur Rehman *et al.*, 2020; Ali *et al.*, 2023). Following incubation, the soils were crushed once again, allowed to air dry, and then sieved through a sieve with a 2 mm mesh.

## Arrangement and deployment of DGT devices

The DGT units, comprising diffusive gels and Chelex, were sourced from England. The Chelax gel sheet was placed on a marble surface, rinsed with deionized water, and sterilized using ethanol. Circular discs, each 2.5 cm in diameter, were carefully cut from the sheet using a cutter by applying pressure and a twisting motion to ensure precision, then high-purity water was used to moisten the filter membrane. The Chelex gel was first positioned on the base with the resin side facing upward, followed sequentially by the layering of the diffusive gel and the placement of the filter membrane on top. Gentle lateral pressure was applied to firmly attach the cap to the base, ensuring a secure fit. DGT units were lightly hydrated using water and enclosed in sterile plastic bags for storage. Optimal membrane hydration was maintained by storing devices at 4 °C in a refrigerator.

The DGT deployment setup consisted of prewashed, air-dried plastic boxes, each filled with 1 kg of homogenized contaminated soil adjusted to 85% moisture. The deployment time for each DGT unit was recorded carefully at intervals of 2, 4, 8, 12, 24, and 48 hours. DGT units were retrieved after the designated exposure period with caution to prevent contact with the exposed window, followed by thorough rinsing using a pressurized stream of water from a wash bottle. After ensuring moisture retention, the DGT units were transferred to sterile plastic bags and stored for further analysis.

## Determination of Cd and Pb concentration in soil samples

Studies indicate that DGT techniques are most effective for measuring metals like Zn, Co, Ni, and Cu in both laboratory and field. Interpreting data for metals such as Cd and Pb is more complex. Therefore, although measurements were taken for various metals, the current focus is mainly on Zn, Co, Ni, Cu, and especially Pb and Cd. Stored DGT units were removed from the refrigerator without unsealing the plastic bags before placing them in the deployment boxes. Three DGT devices (T1, T2, and T3) were deployed in metal-contaminated soil. Initially, the soil was adequately moistened by irrigating it with deionized water until it resembled slurry. Sealed DGT units were opened and removed from the plastic bags, then four units were deployed in each box by pressing and rotating the device. Care must be taken to maintain a firm connection between the membrane filter and the soil. The temperature device was set up to monitor and determine the mean temperature during the DGT deployment time.

## DGT-based measurements of Pb and Cd

After the homogenization period (2-48 hours), the washed DGT devices were used to quantify metal adsorption. Pre-labeled tubes were weighed, filled with 2 mL of 1 M $HNO_3$, and weighed again to determine the exact volume. The DGT cap was opened using a screwdriver, and the gel layers and filter membrane were separated. The Chelex gel was gently rinsed with high-purity water and placed on filter paper to remove excess moisture. It was then transferred into the pre-weighed tube, ensuring it was fully submerged in the acid. After 24 hours, the gels were removed and the eluate was collected. The final solutions were diluted tenfold before analysis by AAS.

Metal concentrations (Pb and Cd) in elution solution (Ce) were measured by an atomic absorption spectrophotometer model Analytik Jena nova 350.

Metal concentration accumulated in resin membrane gel (M) was measured using the following equation:

$$M = Ce\ (VHNO_3 + Vrm)\ /\ fe$$

Here, Ce represents the concentration of metal ($\mu g\ L^{-1}$), $VHNO_3$ denotes the $HNO_3$ volume added to the membrane resin, Vrm represents membrane resin volume, commonly 0.15mL, and fe represents the metal-specific elution factor, typically 0.8 (Zhang and Davison, 1995). The DGT flux ($F_{DGT}$) was determined based on the metal mass accumulated (M), the surface area of the exposed gel (A) is 3.14 $cm^2$, and the deployment time was represented by t, as used in the equation:

$$F_{DGT} = M/(At)$$

The metal concentration at the soil-diffusive gel interface ($C_{DGT}$) was derived from the measured accumulated metal mass (M):

$$C_{DGT} = Mg\ /\ (DAt)$$

Here, the diffusion layer's total thickness is denoted by g (including the filter and diffusion gel, totaling 0.93 mm), and D stands for the diffusion coefficient of the metal in this layer.

## Statistical analysis

Basic descriptive statistics, analysis of linear regressions, and Pearson correlation coefficients were performed to evaluate the relationship between metal sorption on DGT devices and their deployment time. These statistical tools helped calculate trends, measure the strength of associations, and evaluate the time-based dynamics of Cd and Pb accumulation across different soil types.

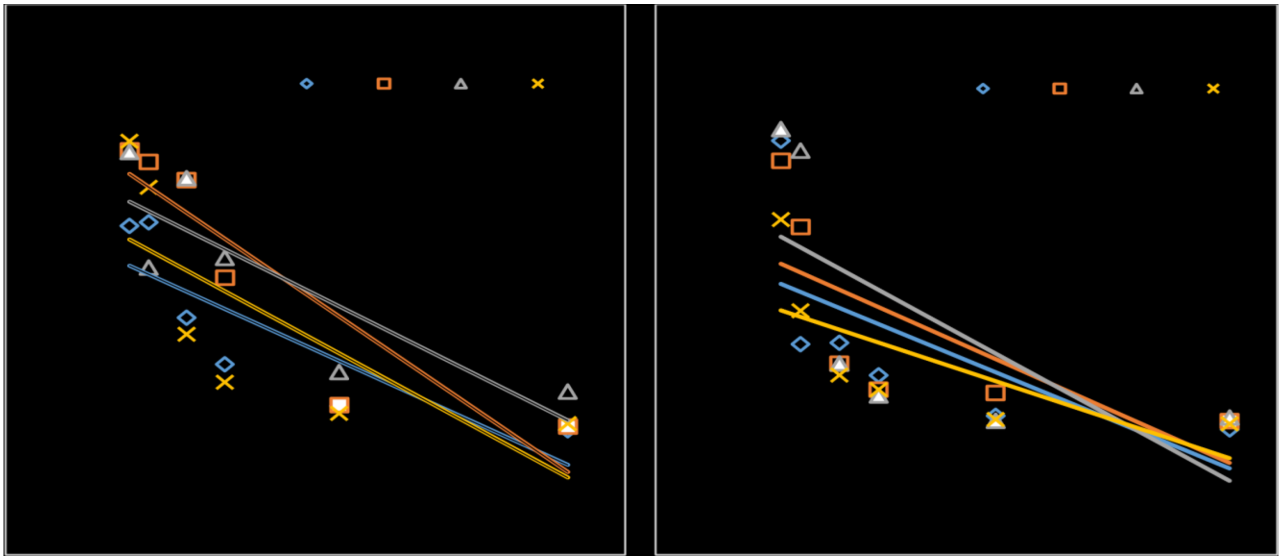

**Figure 1: Linear correlation analysis between deployment time and $C_{DGT}$ of Cd (A), and Pb (B) in different soils. $C_{DGT}$ representing the Cd concentration on DGT, S1 is the aged contaminated sandy clay loam soil, S2 is the spiked soil with Cd and Pb at 25 mg $kg^{-1}$, S3 is the spiked soil with Cd and Pb at 50 mg $kg^{-1}$, S4 is the aged contaminated clay loam soil, and the $R^2$ showing the coefficient of determination**

## Results

### Effect of deployment time on $C_{DGT}$ calculated from different soils

The soil available metal fraction represents the most important parameter in risk assessments of contaminated soil. The DGT-based estimation can be very effective, but plotting time-based metal adsorption on DGT is necessary to assess the time required for maximum accuracy for the sorption of metals, which is essential for precise measurement. The adsorption of labile metal concentrations on the DGT ($C_{DGT}$) is a time-dependent process that reaches a constant equilibrium. The metal ion concentration in solution, denoted by the $C_{DGT}$, reflects the proportion of metal released from the solid matrix that is still available for uptake by plants. DGT devices were placed in four different types of soil for varied periods of time, ranging from 2 to 48 hours (2, 4, 8, 12, 24, 48 hours) throughout the lab experiment. The DGT devices were used in each soil with three replications, and Pb and Cd concentrations were determined at the soil diffusive gel's (DGT) interface. Calculating the $C_{DGT}$ values for Cd and Pb over time shows that $C_{DGT}$ per unit time (seconds) linearly decreased with time, reaching equilibrium (Figures 1-3). A linear regression approach was applied to assess how strongly deployment time influences the concentration of metals accumulated on the DGT ($C_{DGT}$). For Cd, the coefficient of determination ($R^2$) between $C_{DGT}$ of Cd and deployment time in the soils S1, S2, S3, and S4 was 0.7153, 0.8221, 0.7153, and 0.5544, respectively, (Figure 1A). The maximum $R^2$ value was obtained for sandy clay loam soil SCL S2, which was artificially contaminated with Cd and Pb at 25 mg $kg^{-1}$ and irrigated with raw city effluents. At various deployment times, regression analysis between solution Cd and $C_{DGT}$ was developed to examine the dependence of $C_{DGT}$ on solution Cd concentration, evaluated in terms of $R^2$ with a threshold value of 0.75 (considered the criterion for good fitness of the model). The determination coefficients ($R^2$) for linear regression were found to be 0.4406, 0.5149, 0.9402, 0.9206, 0.9184, and 0.9538 at 2, 4, 8, 12, 24, and 48 hours of deployment, respectively (Figure 2). It is inferred that the deployment of DGT devices for 48 hours provided the maximum retention time for Cd on the DGT surface to achieve a concentration near equilibrium. For Pb, the coefficients of determination ($R^2$) between $C_{DGT}$ of Pb and deployment time in the soils S1, S2, S3, and S4 were 0.4422, 0.503, 0.4611, and 0.505 (Figure 1B). The maximum $R^2$ value was obtained for aged contaminated clay loam (CL) soil S4, which became contaminated as a result of prolonged irrigation with untreated raw city effluents. At various deployment times, regression analysis between solution Pb and $C_{DGT}$ was developed to examine the dependence of $C_{DGT}$ on solution Pb concentration and evaluated in terms of $R^2$ with a threshold value of 0.75 (considered a criterion for good fitness of the model). The $R^2$ values of 0.453, 0.5602, 0.0385, 0.6528, 0.4183, and 0.7581 were found for deployment times of 2, 4, 8, 12, 24, and 48 hours, with the final relation being the most significant (Figure 3). It is inferred that the deployment of DGT devices for 48 hours provided the maximum retention time for Pb on the DGT surface to achieve a concentration near equilibrium.

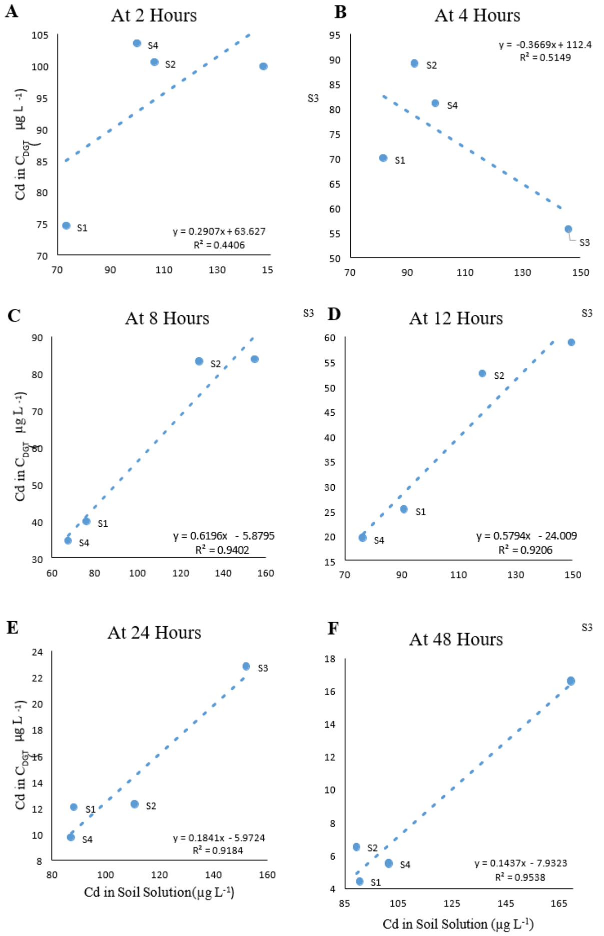

**Figure 2: Linear correlation analysis between the cadmium in soil solution and the $C_{DGT}$ of cadmium in the different soils at different deployment times. A, B, C, D, E, and F represents the deployment time at 2, 4, 8, 12, 24, and 48 hours, respectively. $C_{DGT}$ representing the Cd concentration on DGT, S1 is the aged contaminated sandy clay loam soil, S2 is the spiked sandy clay loam soil with Cd and Pb at 25 mg $kg^{-1}$, S3 is the spiked sandy clay loam soil with Cd and Pb at 50 mg $kg^{-1}$, S4 is the aged contaminated clay loam soil, and the $R^2$ showing the coefficient of determination**

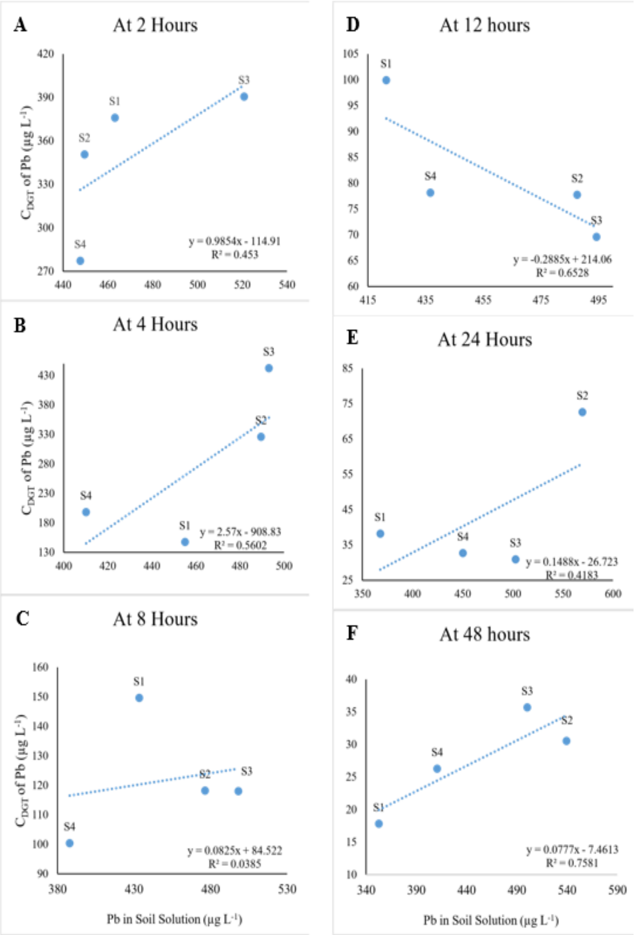

**Figure 3: Linear correlation analysis between the lead (Pb) in the soil solution and the $C_{DGT}$ of Pb in the different soils at different deployment times. A, B, C, D, E, and F represents the deployment time at 2, 4, 8, 12, 24, and 48 hours, respectively. $C_{DGT}$ representing the Pb concentration on DGT, S1 is the aged contaminated sandy clay loam soil, S2 is the spiked sandy clay loam soil with Cd and Pb at 25 mg $kg^{-1}$, S3 is the spiked sandy clay loam soil with Cd and Pb at 50 mg $kg^{-1}$, S4 is the aged contaminated clay loam soil, and the $R^2$ showing the coefficient of determination**

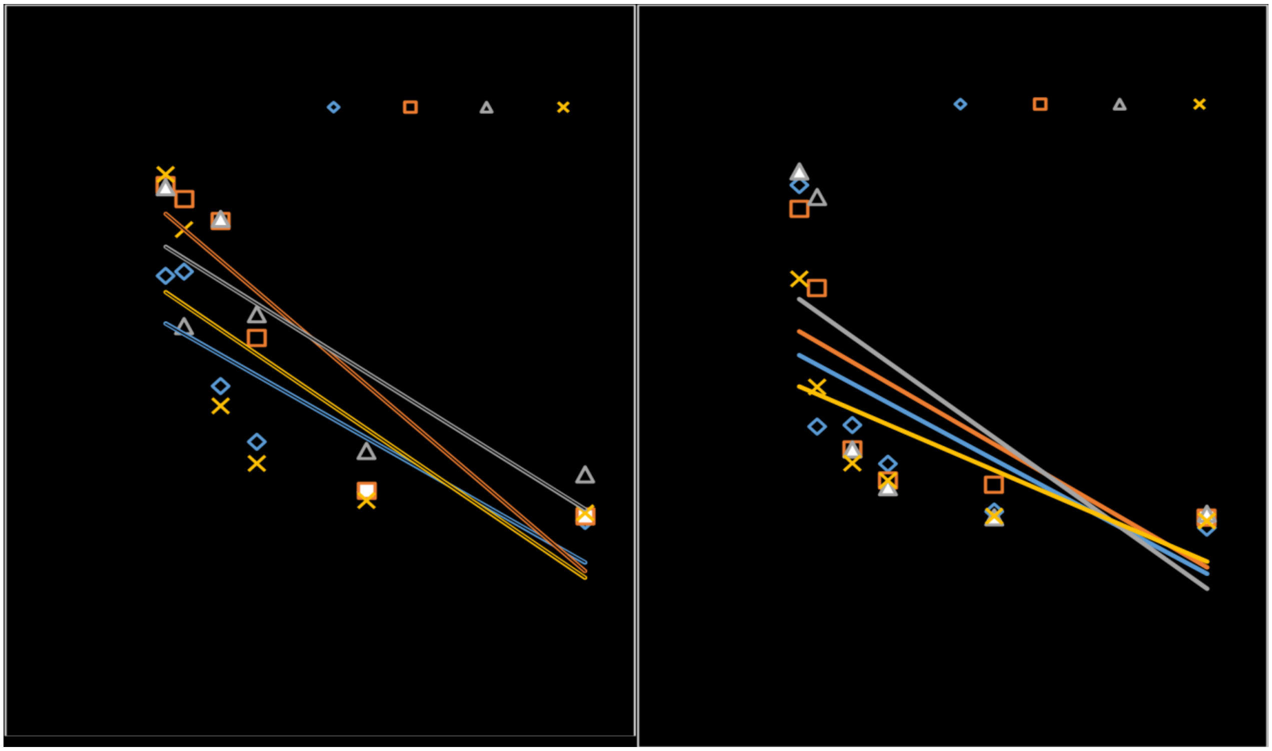

**Figure 4: Linear correlation analysis between deployment time and flux of Cd (A), and Pb (B) in different soils. S1 is the aged contaminated sandy clay loam soil, S2 is the spiked sandy clay loam soil with Cd and Pb at 25 mg kg$^{-1}$, S3 is the spiked sandy clay loam soil with Cd and Pb at 50 mg kg$^{-1}$, S4 is the aged contaminated clay loam soil, and R$^2$ showing the coefficient of determination.**

## Effect of deployment time on the flux of metals in different types of soils

The DGT method proved to be an efficient approach for analyzing the dynamic behavior and availability of metal ions in both water and soil sediments. In this technique, DGT adsorbs the dissolved metal ions from the soil solution, thereby influencing their desorption from the solid phase. The flux of metal ions depended on their concentration, diffusion speed, and the rate of metal replenishment into the solution phase from the solid phase of the soil. Metal replenishment is influenced by the readily available metal ions' concentration bound to the surfaces of soil particles and the kinetics at which metal ions are released into solution. The flux patterns of Cd and Pb changed over time in much the same way as their corresponding CDGT values. The fluxes of Cd and Pb reflected their transfer from the solid phase into the soil solution, indicating an increase in their labile forms. Figures 4-6 show that Cd flux was greatest at the start and gradually decreased over time. The results also show that flux was strongly influenced by deployment duration ($p < 0.001$). This might be because metal sorption attains equilibrium with increasing time.

Metal (Cd and Pb) adsorption flux on DGT devices was found to be in direct agreement with saturation time, as explained by regression analysis of flux and deployment time (Figures 4-6). For Cd, the coefficient of determination for S1, S2, S3, and S4 was found to be 0.7153, 0.8221, 0.7153, and 0.5544, respectively. Interactions between solution Cd concentration and Cd flux for 4 different soils at various deployment times were also plotted (Figures 4A). The R$^2$ values were found to be 0.4406, 0.5149, 0.9402, 0.9206, 0.9184, and 0.9538 for 2, 4, 8, 12, 24, and 48 hr, respectively. The strongest interaction was observed at the 48-hour deployment time, which may be attributed to the equilibrium attenuation. For Pb, the coefficient of determination for S1, S2, S3, and S4 was found to be 0.4422, 0.503, 0.4611, and 0.505, respectively. Interactions between the solution Pb concentration and Pb flux for 4 different soils at various deployment times were also plotted. The R$^2$ values were found to be 0.453, 0.5602, 0.0385, 0.6528, 0.4183, and 0.7581 for 2, 4, 8, 12, 24, and 48 hr, respectively. The strongest interaction was observed at the 48-hour deployment time, which may be attributed to the equilibrium attenuation.

## Effect of deployment times on distribution coefficient (R)

The gradual change in both Pb and Cd concentrations as measured by DGT over deployment times can offer valuable information about the adsorption capacity of DGT and the rate at which these metals are transferred into the solution phase of the soil from the solid phase. The distribution coefficient (R), calculated in this experiment, represents the ratio of the concentrations of Pb and Cd in the soil solution to those determined by DGT (Figures 7-9). This ratio offers an idea of equilibrium retention over time, with a linearly decreasing R indicating a relationship with time. For the Cd and Pb ratio-time regression, the $R^2$ values

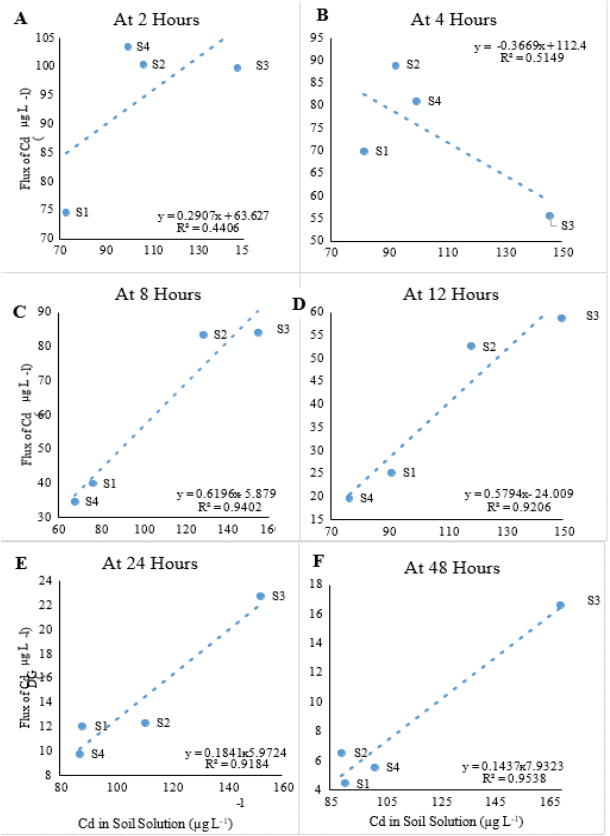

**Figure 5: Linear correlation analysis between the cadmium (Cd) in soil solution and the flux of Cd in the different soils at different deployment times. A, B, C, D, E, and F represents the deployment time at 2, 4, 8, 12, 24, and 48 hours respectively. S1 is the aged contaminated sandy clay loam soil, S2 is the spiked sandy clay loam soil with Cd and Pb at 25 mg $kg^{-1}$, S3 is the spiked sandy clay loam soil with Cd and Pb at 50 mg $kg^{-1}$, S4 is the aged contaminated clay loam soil, and the $R^2$ showing the coefficient of determination**

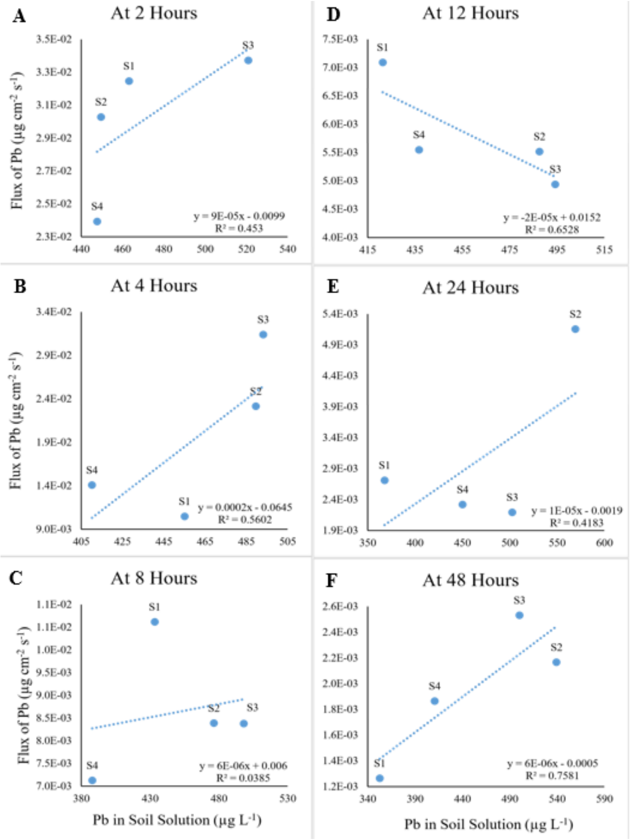

**Figure 6: Linear correlation analysis between the lead (Pb) in the soil solution and the flux of Pb in the different soils at different deployment times. A, B, C, D, E, and F represents the deployment time at 2, 4, 8, 12, 24, and 48 hours respectively. S1 is the aged contaminated sandy clay loam soil, S2 is the spiked sandy clay loam soil with Cd and Pb at 25 mg $kg^{-1}$, S3 is the spiked sandy clay loam soil with Cd and Pb at 50 mg $kg^{-1}$, S4 is the aged contaminated clay loam soil, and the $R^2$ showing the coefficient of determination**

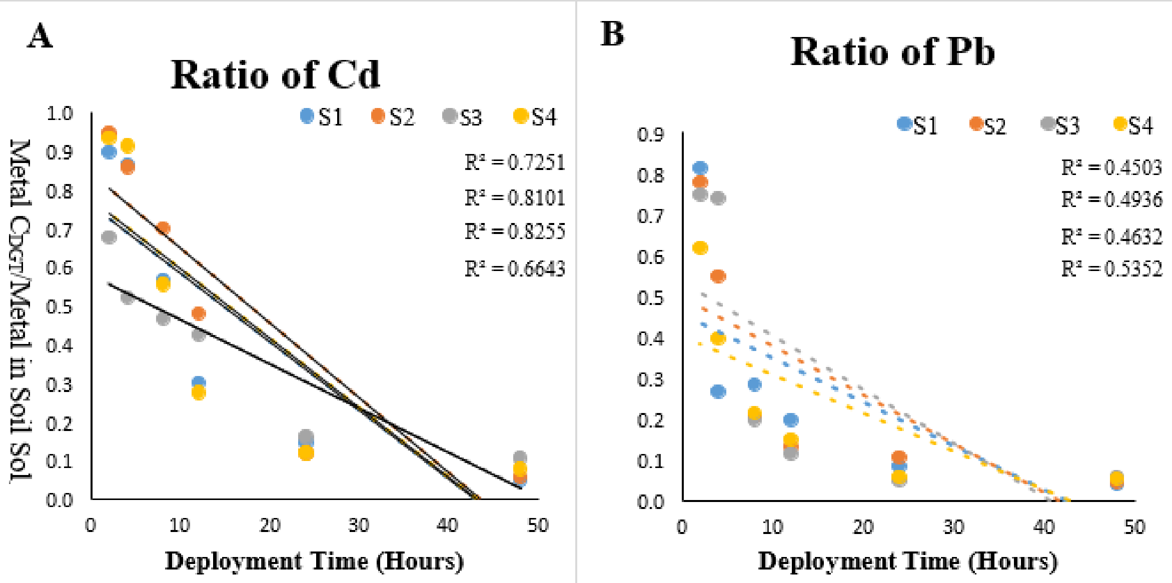

**Figure 7: Effect of deployment time on the distribution coefficient, R (ratio) on cadmium (A), and lead (B). S1 is the aged contaminated sandy clay loam soil, S2 is the spiked sandy clay loam soil with Cd and Pb at 25 mg kg$^{-1}$, S3 is the spiked sandy clay loam soil with Cd and Pb at 50 mg kg$^{-1}$, S4 is the aged contaminated clay loam soil, and the $R^2$ showing the coefficient of determination**

were 0.7251, 0.8101, 0.8255, 0.6643, and 0.4503, 0.4936, 0.4632, 0.5352 for S1, S2, S3, and S4, respectively. The correlation analysis demonstrated the significant relationship between the distribution coefficient and time, as shown by higher $R^2$ values across all soils, although results for Pb were inconsistent. The distribution coefficient was also correlated with soil solution concentrations at different deployment times. It was observed that the highest correlation occurred at 48 hours for Cd; however, the results for Pb were inconsistent as illustrated in Figures 9. The variability in Pb results for some parameters suggests extending the deployment time (more than 48 hours) for Pb, which could yield more promising results.

## Discussion

The deployment duration of DGT devices is crucial for accurately determining the concentrations of labile metals, since it affects the establishment of a steady-state diffusion and the resupply of metals from the solid phase to the solution phase. Deployment time in the current study affected $C_{DGT}$, fluxes, and distribution coefficients (R) of Cd and Pb in four soil types having different textures, contamination levels, and sources of contamination, including soils irrigated with sewage water, aged contaminated soils, and artificially spiked soils. Cd exhibited a linear decrease in $C_{DGT}$ per unit time with increased deployment time, reaching a state of equilibrium after 48 h, while for Pb, a weaker and not very consistent trend was observed, as illustrated in Figures 1-3.

Regression analysis indicated that the relations between solution Cd and $C_{DGT}$ after 48 h of deployment were characterized by strong positive correlations, $R^2 > 0.953$, whereas for Pb, a moderate correlation, $R^2 = 0.758$, reflected differences in the respective sorption kinetics and interactions of these metals with soil components. $C_{DGT}$ and metal flux decreased with increasing deployment time, showed that devices approached equilibration progressively as labile metal pools were depleted. Physicochemical properties and affinities for soil textures and contamination levels affect the kinetic dynamics of Cd and Pb.

Cadmium attained equilibrium faster than Pb, as shown by the higher values of $R^2$ between $C_{DGT}$ and deployment time (Fig. 1-3). Cd reaches equilibrium faster because of its higher solubility, smaller ionic radius of 0.97 Å, and weaker complexation with organic matter and soil colloids (Kubier *et al.*, 2019; Li *et al.*, 2024b). Cd exists mainly as free $Cd^{2+}$ or as weakly complexed species such as $CdCl^-$ and $CdSO_4$, allowing it to diffuse through the DGT diffusive layer more readily and to accumulate on the DGT surface (Kubier *et al.*, 2019; Fan *et al.*, 2022). In contrast, Pb may form stable inner-sphere complexes with oxygen-donor ligands in

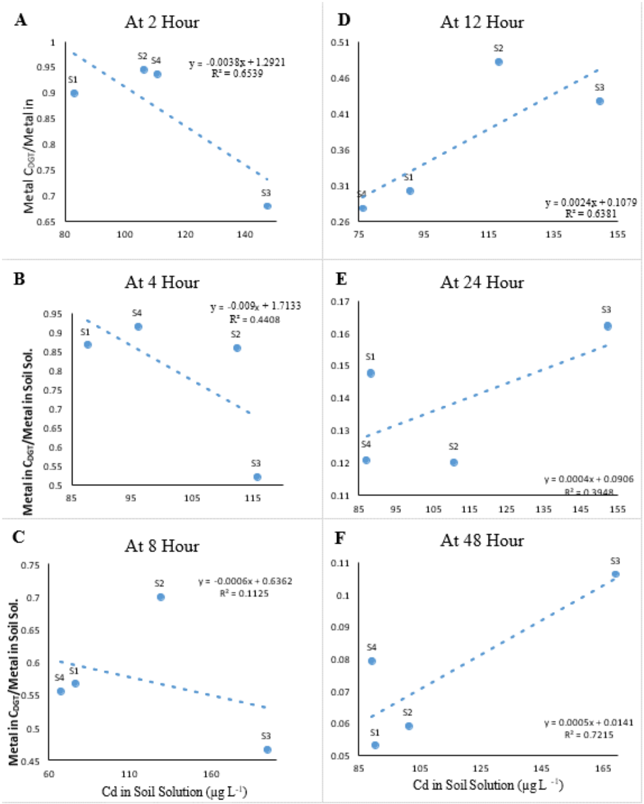

**Figure 8: Linear correlation analysis between the ratio of cadmium and concentration of cadmium in soil solution collected from different soils at different deployment times. A, B, C, D, E, and F represents the deployment time at 2, 4, 8, 12, 24, and 48 hours respectively. S1 is the aged contaminated sandy clay loam soil, S2 is the spiked sandy clay loam soil with Cd and Pb at 25 mg kg$^{-1}$, S3 is the spiked sandy clay loam soil with Cd and Pb at 50 mg kg$^{-1}$, S4 is the aged contaminated clay loam soil, and the R$^2$ showing the coefficient of determination**

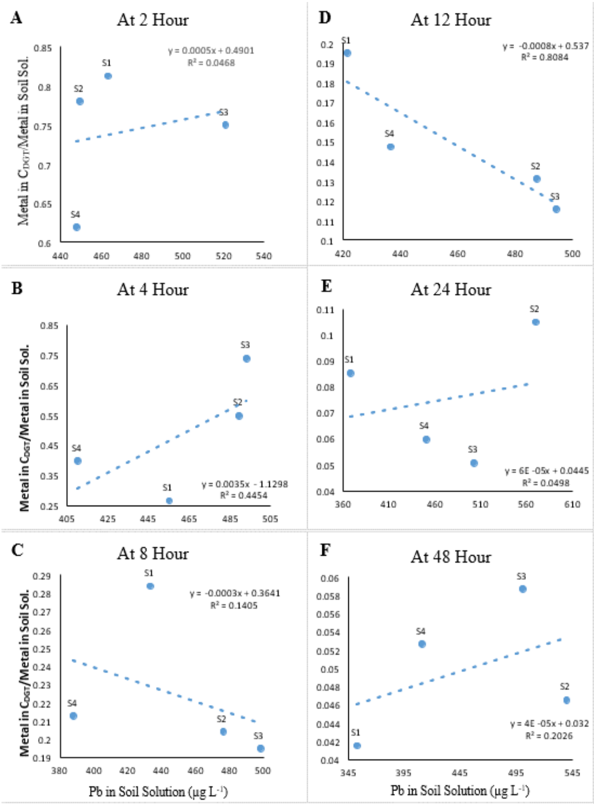

**Figure 9: Linear correlation analysis between the ratio of lead and concentration of lead in soil solution collected from different soils at different deployment times. A, B, C, D, E, and F represents the deployment time at 2, 4, 8, 12, 24, and 48 hours respectively. S1 is the aged contaminated sandy clay loam soil, S2 is the spiked sandy clay loam soil with Cd and Pb at 25 mg kg$^{-1}$, S3 is the spiked sandy clay loam soil with Cd and Pb at 50 mg kg$^{-1}$, S4 is the aged contaminated clay loam soil, and the R$^2$ showing the coefficient of determination**

organic matter and clay minerals because of its larger ionic radius of 1.19 Å and higher polarizing power (Refaey *et al.*, 2014; Du *et al.*, 2022; Steinberg and Hodge, 2022). This strong binding reduces $Pb^{2+}$ accumulation on the DGT device for the same deployment time by making it less mobile and thus moving more slowly from the solid phase into the soil solution (Zhu *et al.*, 2023). Another explanation for the deviating behaviors between Cd and Pb also considers their different geochemical characteristics and differing affinities for the soil solid phases. Cd is moderately mobile and forms weak complexes with soil organic matter and mineral oxides, allowing for its rapid desorption and replenishment into the soil solution (Degryse *et al.*, 2009; Mi *et al.*, 2018). Cd predominantly exists in exchangeable or carbonate-bound forms that are highly mobilizable when a DGT device serves as a sink. Consequently, Cd responds quickly to changes in the solution concentration and reaches its equilibrium over shorter deployment times (Ernstberger *et al.*, 2002; Degryse *et al.*, 2009). This pattern explains the steady decrease in CDGT per unit time and the high $R^2$ values observed after 48 hours, suggesting that the labile Cd fraction was efficiently taken up within this duration. Pb, on the other hand, has a significantly greater affinity for soil solid phases, forming strong inner-sphere complexes with organic matter, clay minerals, and Fe/Mn oxides (Aslam *et al.*, 2022; Mao *et al.*, 2022; Li *et al.*, 2024a). This strong binding led to decreased Pb solubility and delayed its release from the solid phase; thus, it could not easily diffuse towards the DGT surface, which caused the slow equilibration of Pb and weaker correlations between CDGT and solution concentrations during shorter deployments.

The physicochemical properties of the soils significantly affected DGT responses. Among the tested soils, the highest $R^2$ (0.8221) of CDGT versus deployment time was observed for Cd in S2 soil, with its moderate organic matter content and slightly low pH. The relatively low pH favors the desorption of Cd from the exchange sites and increases the dissolution of Cd-bearing minerals, leading to higher $Cd^{2+}$ concentrations in the solution (Wen *et al.*, 2023; Boostani *et al.*, 2024). On the other hand, S4 soil, which is characterized by higher pH, CEC, and organic matter content, showed a lower CDGT, or flux, of both metals, especially Pb. These factors contribute to forming stable Pb-organic and Pb-carbonate complexes, which decrease the available Pb pool for uptake by the DGT device (Senila and Kovacs, 2024; Cheng *et al.*, 2025). Shaghaleh *et al.* (2024) also reported that a higher CEC and organic carbon content in soils resulted in stronger retention of Pb than Cd, leading to reduced fluxes as measured by DGT.

The progressive decrease in metal flux over time reflects the depletion of readily exchangeable ions from the soil solution. The steep concentration gradients present at the beginning between the soil solution and the DGT surface drive rapid diffusion. However, this flux decreases as the system approaches a steady state (Luo *et al.*, 2014; Senila and Kovacs, 2024). These high $R^2$ values indicate that, until nearly reaching equilibrium, there was a continuous and very efficient resupply of Cd from the solid phase (Ernstberger *et al.*, 2005; Guo *et al.*, 2022). In contrast, the kinetic experiments revealed weaker correlations for Pb, and the approach to equilibrium was slower. This indicates that its resupply is constrained by diffusion and significant retention in the solid phase (Wagner *et al.*, 2022). In the aged contaminated clay loam soil (S4), the sequestration of Pb into more stable residual fractions over time has further reduced its mobility (Liang *et al.*, 2014; Qiu *et al.*, 2024). Temporal patterns of metal fluxes emphasize these mechanistic differences further. The highest fluxes of Cd and Pb occurred during the initial stages of deployment and progressively decreased as the approach toward equilibrium became stronger. This decrease reflects the progressive depletion of readily available ions in the soil solution and the growing influence of desorption kinetics on the replenishment of metals from the solid phase (Ridošková *et al.*, 2017) . Regression results showed that Cd displayed a stronger and more consistent linear decrease in flux across the soils, while Pb showed greater variability, reflecting its slower release from soil surfaces and lower lability (Ernstberger *et al.*, 2005; Ji *et al.*, 2021).

The distribution coefficient, R, represents the ratio of metal concentrations in the soil solution to those accumulated on the DGT. This coefficient provides valuable insights into the dynamics of metal partitioning. The gradual decline in R for Cd and Pb reflects progressive equilibration between the DGT and the soil solution. R decreased linearly for Cd with high coefficients of determination ($R^2 > 0.8255$) in several soils, indicating efficient replenishment of the soil solution and that steady-state equilibrium was approached rapidly. While R values of Pb were lower ($R^2 = 0.5352$) with weaker slopes related to deployment time, indicating much slower desorption from the solid phase, which is due to strong complexation with organic matter and oxide minerals. The variation in Pb R-values across the soils indicates that uniform equilibration likely requires more than 48 hours, reflecting its slower diffusion and desorption processes. These findings are consistent with earlier reports showing that Cd is predominantly held in more labile, easily exchangeable fractions, whereas Pb is largely tied to stable oxide- and organic-bound pools (Mi *et al.*, 2018; Cheng *et al.*, 2025). The slower equilibration of Pb implies that DGT deployment times longer than 48 hours are required to accurately determine its labile fraction, particularly in soils rich in clay and organic matter. Such time-dependent equilibration patterns for Pb were observed by Nolan *et al.* (2005), who, citing its low diffusivity and strong affinity for organic ligands, noted that concentrations measured by DGT are limited even for extended deployments.

The dissimilarities noted between soils highlight the crucial role played by soil characteristics and contamination backgrounds in determining the metal behavior. The highest correlation for Cd was detected in the case of S2 soil, which was sandy clay loam, artificially contaminated with Cd and Pb at 25 $mg\ kg^{-1}$. It can be assumed that such a type of soil would have a larger amount of newly introduced weakly bound Cd, which would be more readily transformed into the soil solution. In contrast, the highest correlation of Pb was found in the aged contaminated clay loam (S4) soil, possibly due to the gradual redistribution of Pb into oxide-bound and residual fractions with time. Soil texture, organic matter, oxide content, and contamination background are the key determinants in regulating the kinetics of metal release and the DGT response (Cheng *et al.*, 2025). High Fe/Mn oxide and humic substance soils have many adsorptions for Pb, which reduces its lability and makes it equilibrate more slowly than Cd (Chen *et al.*, 2021; Hu *et al.*, 2024). Mechanistically, the different behavior of Cd and Pb can be attributed to their respective binding energies and soil speciation. Cd, in most cases, is involved in outer-sphere complexation, where the ionic charges of the ions involved attract each other, resulting in a complex with high ion mobility. On the other hand, Pb is more likely to form inner-sphere complexes resulting from the sharing of electrons (covalent bond) with the groups of the organic matter or the oxides (Hao *et al.*, 2020; Steinberg and Hodge, 2022). The DGT method successfully demonstrates these kinetic disparities by indicating that the labile pool of Cd is mostly controlled by diffusion, whereas that of Pb is limited by desorption kinetics.

## Conclusions

The findings of this study emphasize the efficiency of the Diffusive Gradients in Thin Films (DGT) technique as a comprehensive and sensitive method for assessing the availability of Pb and Cd in soils. DGT measurements are influenced by time, soil texture, and the source of contamination, whether aged-contaminated or spiked soils. A decline in $C_{DGT}$ values over longer deployment periods indicates the reduction of the labile metal fraction and the approach to equilibrium. This highlights the importance of choosing appropriate deployment durations when interpreting DGT results. The present study suggests that a 48-hour deployment is adequate for Cd to attain quasi-equilibrium with the soil solution, as demonstrated by strong regressions and stable values of $C_{DGT}$. However, for Pb, longer deployment times were necessary due to its slower release rate and stronger binding onto soil constituents. Deployment times extending to more than 48 hours may yield more accurate measurements of Pb availability, especially in aged or organic-rich soils. These findings recommend the DGT application as a reliable predictive tool for metal mobility and availability, assisting environmental risk assessment and the sustainable management of contaminated agricultural soils. However, the deployment time for Pb measurement could be extended in future studies. Future research might include modeling the kinetic parameters that govern metal resupply and investigating how soil amendments or environmental changes influence the time-based dynamics of $C_{DGT}$ values.

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## Data and material availability

Data will be made available by the corresponding author on a reasonable request.

## Declarations

I affirm that this article has not been submitted to any other journal, and all the authors have consented to submit the manuscript.

## Conflict of interest

All the authors declare no conflict of interest regarding this paper.

## Authors' contribution

SR conceptualized and designed, material preparation, data collection, analysis, prediction of data, and first drafting. MZR supervised, conceptualized, designed, critically reviewed, and evaluated the manuscript. GM and SAA commented on the manuscript.

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