



Residual activity and sorption of tebuthiuron in different soils¹

Atividade residual e adsorção do tebuthiuron em diferentes solos

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HIGHLIGHTS:

The dynamics of tebuthiuron changes due to soil physical and chemical characteristics.

Tebuthiuron remains active for more than 360 days in sandy loam and sandy clay loam soils.

Soil clay and organic carbon contents are the main factors affecting tebuthiuron sorption.

ABSTRACT: Applications of tebuthiuron can increase the risks of environmental contamination and hinder the cultivation of sensitive species in succession. The objective of this work was to assess the residual activity and sorption of the herbicide tebuthiuron in soils with different physical and chemical attributes. The experiments were conducted in a completely randomized design with four replicates. The experiment for determining residual activity was conducted in a 2×7 factorial arrangement consisted of two herbicide doses (0.6 and 1.2 kg a.i. [active ingredient] ha^{-1}) and seven sowing times (0, 60, 120, 180, 240, 300, and 360 days after herbicide application) in sandy loam and sandy clay loam soils. The experiment for evaluating sorption was conducted in a sandy loam soil and two clay loam soils with applications of increasing tebuthiuron doses (0.025, 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.50, 0.60, 0.70, and 0.80 kg a.i. ha^{-1}). Regarding residual activity, the herbicide's effect of the decreased over time, with no toxicity detected 360 days after application (DAA), regardless of the soils and doses used. Sandy loam soils had lower sorption, resulting in lower shoot dry weight and plant height and in higher phytotoxicity 21 days after emergence. Residual activity was detected 360 DAA in the studied soils. Tebuthiuron sorption was higher in soils with higher organic carbon and clay contents.

Key words: herbicide dynamics, sugarcane, carryover, pesticide retention in soils

RESUMO: A aplicação de tebuthiuron pode aumentar os riscos de contaminação ambiental e impossibilitar o cultivo sucessivo de espécies sensíveis. Neste sentido objetivou-se a atividade residual e sorção do herbicida tebuthiuron em solos com diferentes atributos físico-químicos. Os experimentos foram realizados em Iturama, MG, Brasil, em 2021, em delineamento inteiramente casualizado com quatro repetições. O experimento para determinação da atividade residual foi montado em fatorial 2×7 , com os seguintes fatores: duas doses (0,6 e 1,2 kg i.a. [ingrediente ativo] ha^{-1}) e 0, 60, 120, 180, 240, 300 e 360 dias após a aplicação em solos franco-arenosos e franco-argilosos arenosos. No experimento de avaliação da sorção foram utilizados dois solos franco-arenosos e dois franco-argilosos, que receberam doses crescentes de tebuthiuron (0.025, 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.50, 0.60, 0.70 and 0.80 kg a.i. ha^{-1}). Por meio da matéria seca foi possível obter a relação de sorção. Com o passar do tempo, no estudo de atividade residual, foi observada diminuição do efeito do herbicida, não sendo detectada isenção de intoxicação após 360 dias da aplicação tanto nos solos quanto nas doses. Solos franco-arenosos apresentaram menor sorção, obtendo menor massa seca da parte aérea, altura e maior fitotoxicidade, aos 21 dias após a emergência. A atividade residual ainda foi detectada após 360 dias da aplicação nos solos estudados. A sorção de tebuthiuron foi maior em solos com maior teor de carbono orgânico e argila do solo.

Palavras-chave: dinâmica de herbicidas, cana-de-açúcar, carryover, retenção de pesticidas pelo solo



INTRODUCTION

The chemical control of weeds in sugarcane production relies primarily on herbicides for its efficiency and labor-saving benefits (Aekrathok et al., 2021). However, the prolonged presence of these chemicals in the environment can lead to adverse effects, including carryover (Rector et al., 2020) and environmental contamination (Santarossa et al., 2020). Sorption refers to the retention of herbicides in the soil, which is related to adsorption, absorption, and precipitation processes (Dorado & Almendros, 2021).

The persistence of herbicides refers to their ability to remain active in the environment, which is detected by the presence of residues over time after application (Gehrke et al., 2021). When pre-emergent herbicides such as tebuthiuron are applied to the soil, they can undergo retention, transport, and transformation processes (Peña et al., 2020). Tebuthiuron has a long half-life in the soil (exceeding 360 days), high water solubility (2500 mg L⁻¹ at 20 °C), and low soil retention (Koc = 80) (PPDB, 2007).

Sorption and persistence are intertwined and dependent on herbicide properties, climate, and soil characteristics (El-Saeid & Alghamdi, 2020). The dynamics of tebuthiuron in the soil is mainly affected by clay and organic matter contents (Mendes et al., 2021); lower retention is observed in soils with lower clay and organic matter contents (Souza et al., 2001).

Information on how the dynamics of the herbicide tebuthiuron in different soil types can help mitigate its harmful effects on subsequent crops and decrease environmental risks, particularly in terms of surface and groundwater contamination. Therefore, the objective of this work was to assess the residual activity and sorption of the herbicide tebuthiuron in soils with different physical and chemical attributes. The hypotheses tested were: i) the residual activity of tebuthiuron differs in soils with different physical and chemical characteristics, and ii) its sorption may be higher in soils with higher organic carbon and clay contents.

MATERIAL AND METHODS

Residual activity and sorption of tebuthiuron were assessed in two experiments conducted in 2021 in a greenhouse (19°43'20"S, 50°11'37' W, and mean altitude of 474 m). A completely randomized design with four replications was used in both experiments.

Experiment 1: Evaluation of residual activity of tebuthiuron in two different soils

Samples from the 0-20 cm layer of two soils with different physical and chemical attributes (Table 1) and textures (sandy loam, termed soil A; and sandy clay loam, termed soil B), free from herbicide residues, were collected in areas close to sugarcane fields.

The experiment was conducted in a 2 × 7 factorial arrangement consisted of two tebuthiuron doses (0.6 and 1.2 kg a.i. [active ingredient] ha⁻¹) and seven sowing times (0, 60, 120, 180, 240, 300 and 360 days after herbicide application). Four pots were kept without herbicide application for each sowing time and used as controls.

The herbicide was applied to 500 cm³ pots, using a CO₂-pressurized backpack sprayer coupled to a spray boom with four 110 02 fan tips. Each pot constituted a sampling unit.

Cucumber (*Cucumis sativus*) was used as a bioindicator species, as it is considered a bioindicator of the herbicide (Ferreira et al., 2021). Four cucumber seeds were sown in each pot at 0, 60, 120, 180, 240, 300, and 360 days after herbicide application; after emergence, the seedlings were thinned leaving three seedlings per pot. Following sowing, the pots received daily irrigation, keeping the soil moisture close to field capacity, which was previously determined using gravimetry; pots without herbicide application were also irrigated.

Phytotoxicity in the bioindicator species was assessed 21 days after emergence (DAE) at each sowing time. Visual symptoms were assessed using a scale of grades from 0% to 100%, referring to the percentage of injuries caused to plants by the herbicide, where 0% represents absence of injuries and 100% represents the death of the plant (SBCPD, 1995). The height of the bioindicator plants was measured at 21 DAE, using a ruler (mm), and shoots were collected, dried in an oven at 70 °C until constant weight, and weighed on a precision scale to determine the shoot dry matter.

Shoot dry matter and plant height were converted into percentages (%) relative to the control treatment.

The data were subjected to the Shapiro-Wilk normality test, Levene's homoscedasticity test, and analysis of variance ($p \leq 0.05$). Subsequently, regression equations were fitted using the software Sigmaplot 12.5.

Experiment 2: Evaluation of tebuthiuron sorption in different soils

Samples from the 0–20 cm layer of four soils with different physical and chemical characteristics (Table 2) were collected in areas with sugarcane crops. Each soil collection for physical and chemical characterization was carried out at four sampling points. Additionally, an inert substrate composed of washed sand (pH 7.0) was used (A). The textures of the soils used were: sandy loam (B and C) and clay (D and E).

Preliminary tests were conducted using increasing tebuthiuron rates to define rates capable of reducing the accumulated shoot dry matter of the bioindicator plants (*Cucumis sativus*) by 50% in each soil with different physical and chemical characteristics (data not shown). This initial evaluation was conducted to determine the rates at which the shoot dry matter of the bioindicator plant species would reduce by 50%. Rates were established for each soil (Table 3).

Table 1. Physical and chemical attributes of the soils used in the Experiment 1

Soil	pH CaCl ₂	¹ SOC (g dm ⁻³)	P (mg dm ⁻³)	K	Ca	Mg	H+Al	² CEC	Sand	Clay
									(mmol _c dm ⁻³)	
A ³	5.3	12.7	150	4.0	32	9	31	76.0	79	15.4
B ⁴	4.5	6.4	2	0.9	4	3	22	29.9	69.1	23.9

¹Soil organic carbon; ²cation exchange capacity; ³sandy loam; ⁴sandy clay loam

Table 2. Physical and chemical attributes of the soils used in Experiment 2

Soil	pH CaCl ₂	¹ SOC (g dm ⁻³)	P (mg dm ⁻³)	K	Ca	Mg (mmol _c dm ⁻³)	H+Al	² CEC	Sand	Clay (%)
C ³	5.3	12.7	150	4	32	9	31	76	79	15.4
D ⁴	5.3	19.4	6.6	12.1	11.7	20	39.6	62.4	16.8	58.6
E ⁴	4.9	13.2	5	5.5	6.4	11	54.5	68.7	24.5	63.6

¹Soil organic carbon; ²cation exchange capacity; ³sandy loam; ⁴clay

Table 3. Tebuthiuron doses (kg a.i. ha⁻¹) applied to each substrate

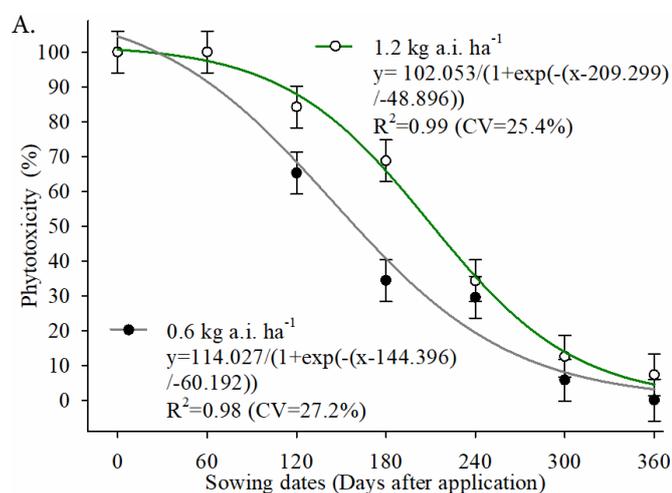
Sand (Soil A ¹)	Soil B ²	Soil C ²	Soil D ³	Soil E ³
Doses (kg a.i. ha ⁻¹)				
0.025	0.05	0.05	0.10	0.10
0.05	0.10	0.10	0.20	0.20
0.10	0.15	0.15	0.30	0.30
0.15	0.20	0.20	0.40	0.40
0.20	0.25	0.25	0.50	0.50
0.25	0.30	0.30	0.60	0.60
0.30	0.40	0.40	0.70	0.70
0.35	0.50	0.50	0.80	0.80

¹inert substrate composed of washed sand; ²sandy loam; ³clay

After filling the 500 cm³ pots with the five substrates, tebuthiuron rates were applied (as described in Experiment 1). Subsequently, seeds of the bioindicator species were sown. Phytotoxicity and plant height evaluations were conducted at 7, 14, and 21 DAE. Shoot dry matter was determined at 21 DAE, as described in Experiment 1.

The results of soil organic carbon (SOC), clay content, phytotoxicity, plant height, and shoot dry matter were used for principal component analysis (PCA). The rate-response referring to a 50% reduction in shoot dry matter of the bioindicator plant (C_{50}) was determined through shoot dry matter curves as a function of increasing tebuthiuron rates. The C_{50} data obtained for each substrate were used to calculate the sorption ratio (SR) of each soil in relation to the response obtained in sand (Souza et al., 1996), according to the following equation (Eq. 1):

$$SR = \frac{C_{50} \text{soil} - C_{50} \text{sand}}{C_{50} \text{sand}} \quad (1)$$



RESULTS AND DISCUSSION

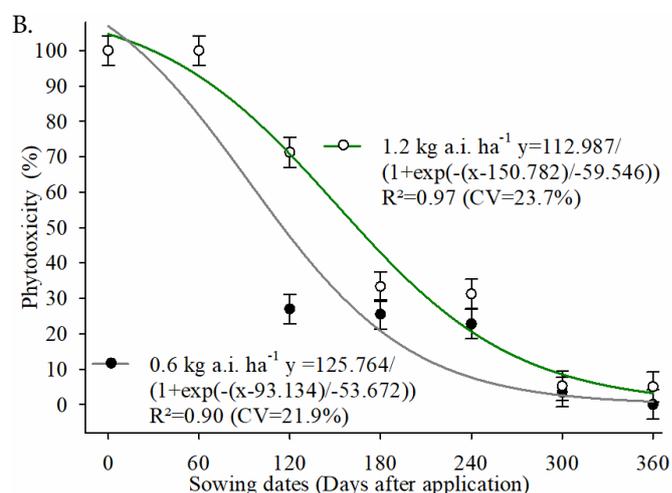
Experiment 1: Evaluation of residual activity of tebuthiuron in two different soils

The interaction between sowing time and herbicide dose (Figures 1A and B) was significant ($p \leq 0.05$) for phytotoxicity and shoot dry matter. The phytotoxicity in the bioindicator decreased over time in both soils, mainly at the lowest evaluated rate. Phytotoxicity in soil A (Figure 1A) at 60 days after application (DAA) was 91.51 and 97.45% for the rates of 0.6 and 1.2 kg a.i. ha⁻¹, respectively; it decreased to 3.09% (0.6 kg a.i. ha⁻¹) and 4.48% (1.2 kg a.i. ha⁻¹) at 360 DAA, due to the decreased residual effect. Phytotoxicity in Soil B (Figure 1B) at 60 DAA was 81.70 and 86.79% for the rates of 0.6 and 1.2 kg a.i. ha⁻¹, respectively, and reduced to 0% for both rates at 360 DAA.

The decrease in phytotoxicity as a function of DAA is due to the decrease in the herbicide's residual activity in the soils, i.e., the low availability of the molecule with phytotoxicity potential in the soil (Rose et al., 2016). Tebuthiuron sorption by SOC and clay (El-Saeid & Alghamdi, 2020; Pereira-Junior et al., 2015), combined with microbial degradation (Guimarães et al., 2022), results in a decreased availability of the molecule in the soil over time.

Plant height and shoot dry matter increased over time after herbicide application (Figures 1C and D). Sowing time had a significant effect on plant height. In soil A (Figure 1C), plant height reached 90.20% at 360 DAA compared to the control. In soil B (Figure 1D), plant height reached 90.22% at 300 DAA. According to Souza et al. (2001) and Duncan & Scifres (1983), soil carbon (C) and clay contents are the main factors affecting the adsorption of tebuthiuron in the soil.

Sowing on the same day of herbicide application did not result in seedling survival and shoot dry matter accumulation in both soils (Figures 1E and F), regardless of the applied herbicide rates. Shoot dry matter increased over time in both soils; significant



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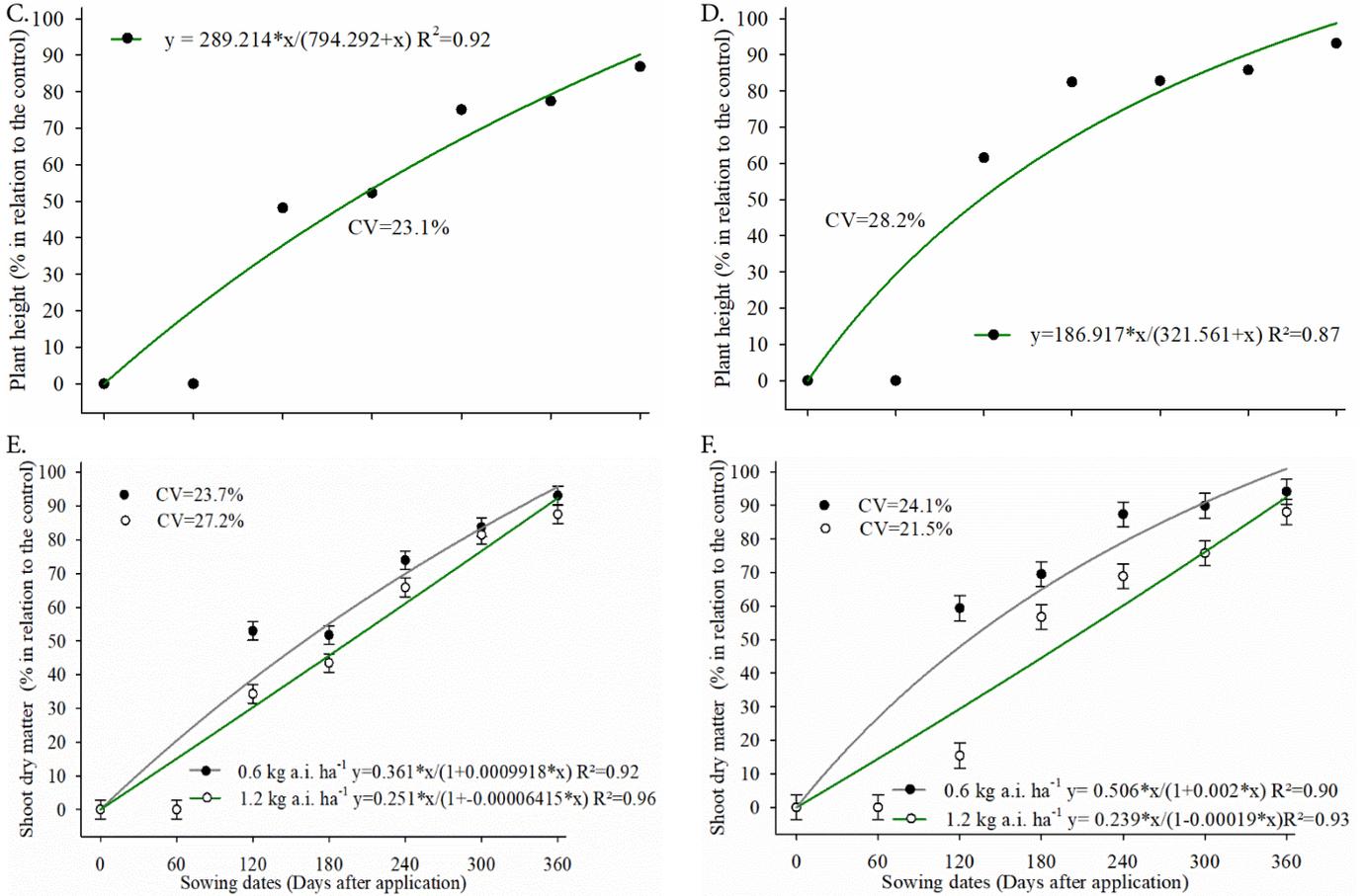


Figure 1. Phytotoxicity in a bioindicator species (*Cucumis sativus*) sown at different times in soils A (A) and B (B); plant height for the bioindicator species sown at different times in soils A (C) and B (D); shoot dry weight of the bioindicator species sown at different times in soils A (E) and B (F)

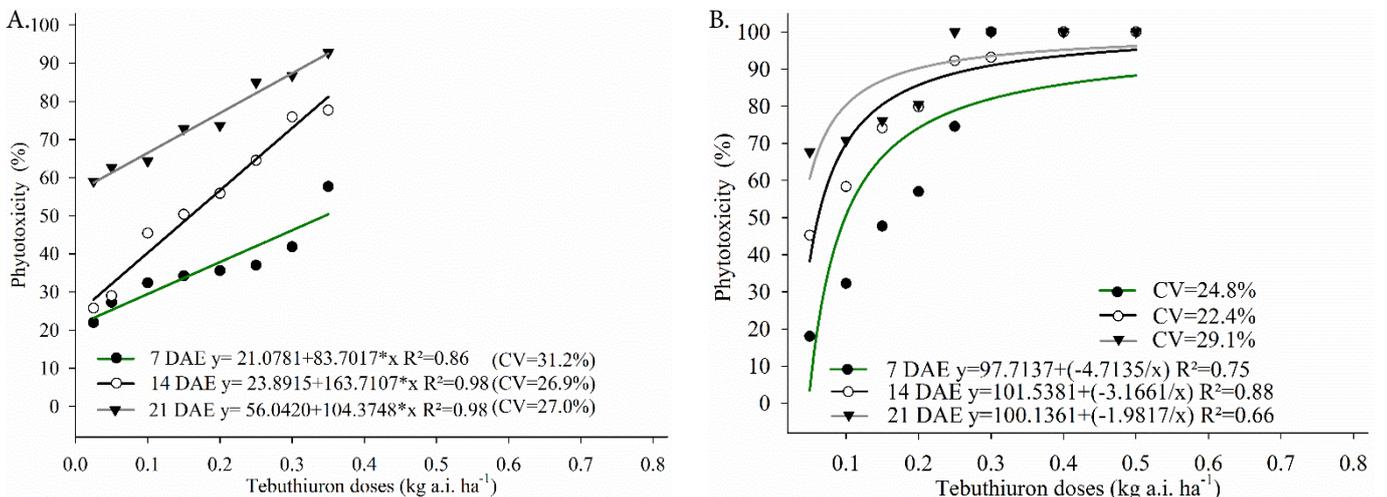
difference was found between herbicide rates from 120 to 240 DAA and up to 300 DAA in soils A and B. The soil C content affected the retention of tebuthiuron herbicide molecules in soil colloids (Souza et al., 2001). This dynamic can be explained by a slightly higher herbicide retention in soil A over time, as it has a higher C content (12.7 g dm⁻³ of SOC) than soil B (6.4 g dm⁻³).

Considering the analyzed variables of the bioindicator species, a significant decrease in the residual activity of the tebuthiuron molecule was found at 360 DAA. However,

biological activity was still observed, with shoot dry matter different from 100%, reaching 95.77 and 92.50% for the tebuthiuron rates of 0.6 and 1.2 kg a.i. ha⁻¹, respectively, in soil A, and 80.53% for the rate of 1.2 kg a.i. ha⁻¹ in soil B at 360 DAA.

Experiment 2: Evaluation of tebuthiuron sorption in different soils

The phytotoxicity caused by tebuthiuron (Figure 2) on *Cucumis sativus* plants increased ($p \leq 0.05$) as the herbicide



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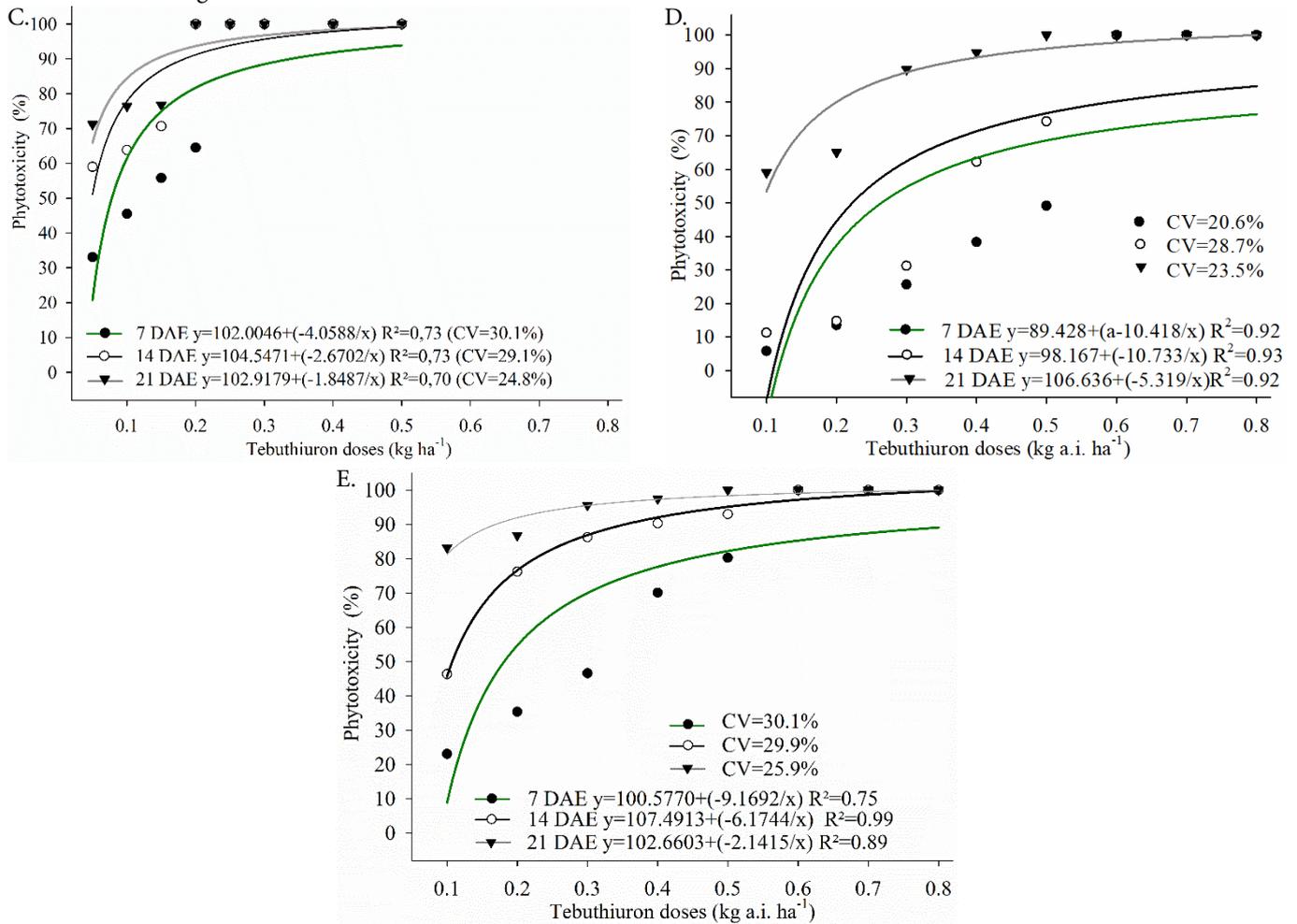
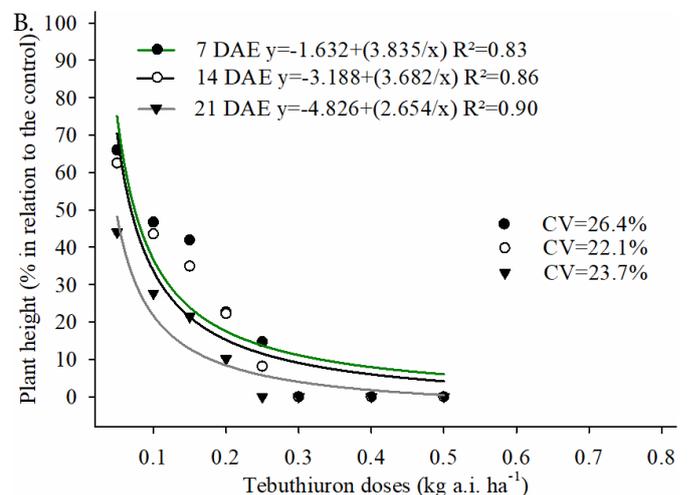
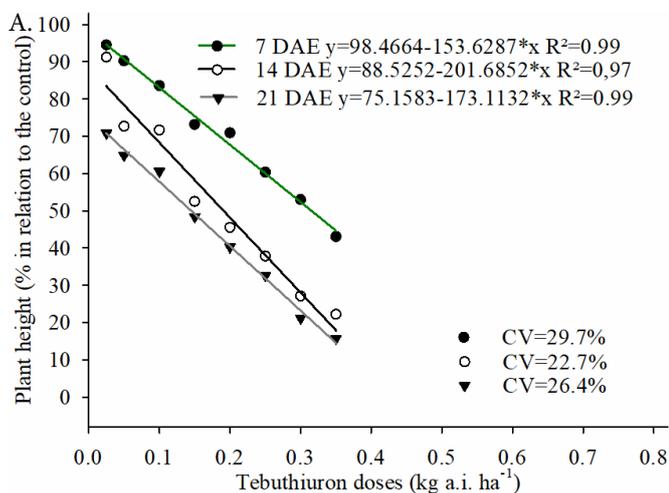


Figure 2. Phytotoxicity caused by application of tebuthiuron doses to a bioindicator species (*Cucumis sativus*) grown in sand (A), soil B (B), soil C (C), soil D (D), and soil E (E)

rates were increased, characterized by interveinal chlorosis starting at the edges towards the center of the leaves, followed by total necrosis. Symptoms increased over the evaluations at 7, 14, and 21 DAE in plants grown in all evaluated soils. The herbicide rate of 0.2 kg a.i. ha⁻¹ caused phytotoxicity of 93.67 and 80.04% at 21 DAE in the sandy loam soil (C) (Figures 2C) and clay soil (D) (Figure 2D), respectively.

Plant height ($p \leq 0.05$) decreased as the herbicide doses were increased for all substrates (Figure 3). The rate of 0.20 kg a.i. ha⁻¹ applied at 21 DAE to soils with higher clay and SOC contents (soils D and E) resulted in plant heights 37.91 and 37.52% relative to the control (Figures 3D and E), whereas the same dose applied at the same date to soils B and C resulted in 8.44 and 10.37%, respectively (Figures 3B and C).



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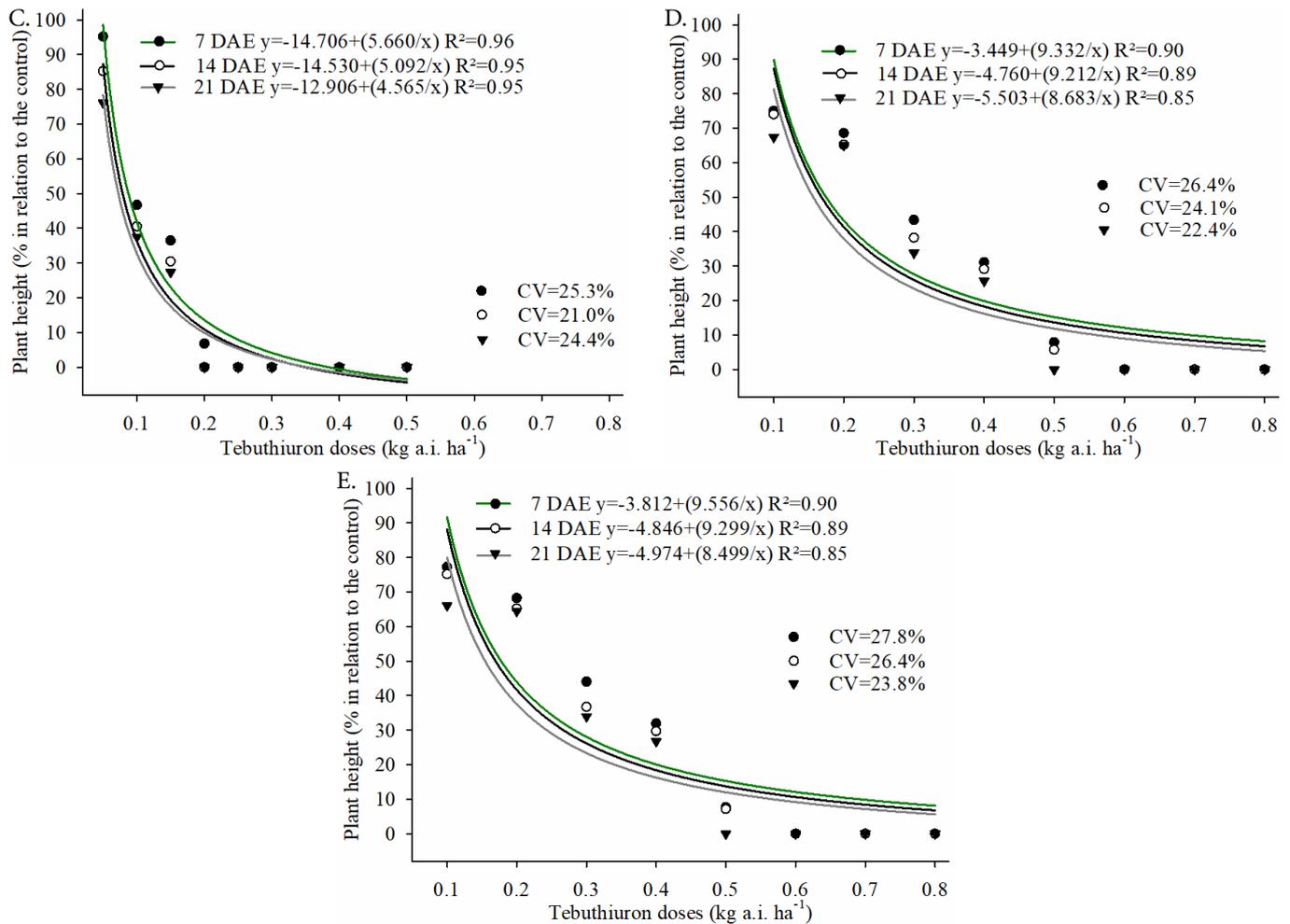


Figure 3. Plant height of a bioindicator species (*Cucumis sativus*) grown in sand (A), soil B (B), soil C (C), soil D (D), and soil E (E), as a function of tebuthiuron doses

PCA showed a clear distinction between the soils (Figure 4). The sandy loam soils (soils B and C) and clayey soils (D and E) were clearly separated by Axis 1, which represented 83% of the variation among the samples. The eigenvectors showed that soil physical and chemical characteristics strongly contributed to plant height and phytotoxicity. Phytotoxicity was positively

impacted by soil B (with low SOC and clay contents), while plant height was positively impacted by soil D (higher SOC and clay contents).

The different phytotoxicity and plant height between clayey and sandy soils denoted the distinct dynamics of tebuthiuron in soils with different characteristics. This is due to the enhanced adsorption of clay-rich soils with abundant soil organic carbon (Souza et al., 2001; Guimarães et al., 2022). Increasing tebuthiuron rates decreased aboveground biomass (shoot dry matter) ($p < 0.05$) in all soils (Figure 5). Soil D significantly affected shoot dry matter due to its higher sorption capacity for tebuthiuron (Table 4). Notably, soil D, which had a higher SOC content (19.4 g dm^{-3}) than the other soils and substantial clay content (58.6%) (Table 2), exhibited unique sorption characteristics, which is typical of non-ionic herbicides that rely on contact with the soil surface, primarily SOC and clay (Mendes et al., 2021).

The adsorption of tebuthiuron was more pronounced in soils with high clay and SOC contents, indicating that tebuthiuron has a greater affinity for these fractions (Souza et al., 2001). The sorption potential of tebuthiuron is dependent on the SOC content (Duncan & Scifres, 1983).

The dynamics of the herbicide tebuthiuron was different in the different soil physical and chemical conditions, and different sorption potentials were found in the studied soils (Table 4). Thus, considering the physical and chemical

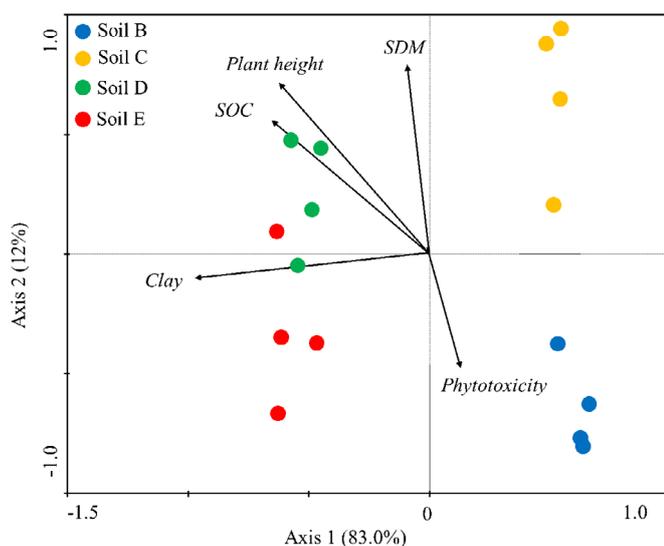


Figure 4. Distribution of plant height, phytotoxicity, shoot dry weight, soil clay, and soil organic carbon (SOC) by principal components analysis

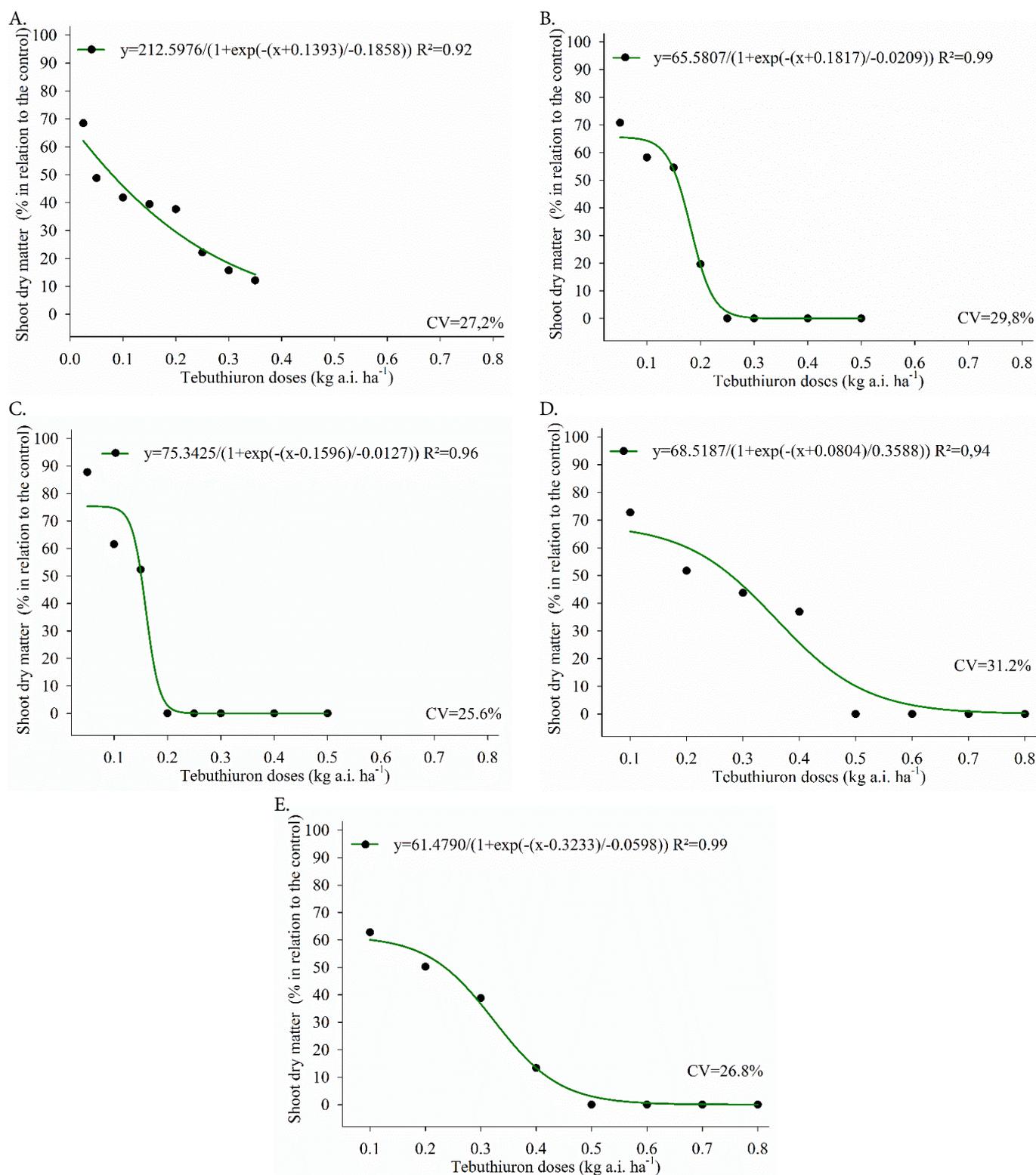


Figure 5. Shoot dry weight of a bioindicator species (*Cucumis sativus*) grown in sand (A.), soil B (B.), soil C (C.), soil D (D.), and soil E (E.) with application of increasing tebuthiuron doses

Table 4. Doses of the herbicide tebuthiuron (kg a.i. ha⁻¹) required to reduce the shoot dry weight accumulation in *Cucumis sativus* by 50% (C₅₀) and SR (sorption ratio) for different soils

Soil	C ₅₀	SR
Sand (A ¹)	0.08	-
B ²	0.16	0.86
C ²	0.15	0.79
D ³	0.28	2.25
E ³	0.24	1.78

¹inert substrate composed of washed sand; ²sandy loam; ³clay

attributes of each soil is essential for recommending or even restricting the application of tebuthiuron.

CONCLUSIONS

1. The residual activity of the herbicide tebuthiuron is low but still detectable 360 days after application at dose of 1.2 kg a.i. ha⁻¹ in soils with sandy loam and sandy clay loam textures.

2. Soil organic carbon and clay contents affected the sorption potential of the herbicide tebuthiuron. The increasing order of tebuthiuron sorption ratio in the evaluated soils is [respectively: clay (%); SOC (g dm⁻³): soil B [15.4; 12.7] < soil C [17.7; 8.7] < soil E [63.6; 13.2] < soil D [58.6; 19.4].

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