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Sorption of diuron and sulfentrazone on different substrates

Sorção de diuron e sulfentrazone em diferentes substratos

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The efficiency in pre-emergence weed control is linked to the understanding of sorption, whose process makes the herbicide molecule unavailable for absorption by plants, and only returns to the soil solution after desorption processes. Understanding herbicide sorption is crucial, especially when separating the use of substrates as a fixation base for plants, which can absorb greater or lesser amount of applied herbicides. Therefore, this study aimed to quantify the sorption of diuron and sulfentrazone herbicides on different substrates. Two experiments were carried out, the first was the control experiment, using washed sand substrate as control for the application of seven doses of the two proposed herbicides. In the second experiment, six doses of both herbicides were tested on six different substrates. The treatments were conducted in a greenhouse using polyethylene pots and cucumber with indicator plant. The characteristics evaluated were the determination of the I_{50} value of the test plant for each substrate as a function of the doses of each herbicide and the estimation of the amount of diuron and sulfentrazone sorbed in each substrate. Indian Black Earth showed the highest sorption capacity for diuron and sulfentrazone, followed by coconut fiber. The diuron doses for Indian Black Earth and Sand + Indian Black Earth were not effective in controlling the plant population. The substrates mixed with sand showed lower sorption capacity than the others, requiring greater care when using herbicides. The organic matter, pH and soil texture are factors that contributed to the sorption of herbicides.
Keywords: inhibition, phytotoxicity, organic matter.

A eficiência no controle em pré-emergência de plantas daninhas está ligada ao entendimento da sorção, cujo processo indisponibiliza a molécula do herbicida para absorção pelas plantas, e só retorna à solução do solo após processos de dessorção. Entender a sorção de herbicidas é crucial para utilização de substratos como base de fixação para as plantas, os quais podem sorver maior ou menor quantidade dos herbicidas aplicados. Portanto, esta pesquisa objetivou quantificar a sorção dos herbicidas diuron e sulfentrazone em diferentes substratos. Dois experimentos foram realizados, o primeiro foi o experimento controle, empregando o substrato areia lavada como testemunha para a aplicação de sete doses dos dois herbicidas propostos. No segundo experimento foram testadas seis doses dos dois herbicidas em seis substratos diferentes. Os tratamentos foram conduzidos em casa de vegetação usando potes de polietileno e pepino como planta indicadora. As características avaliadas foram I_{50} da planta-teste para cada substrato em função das doses de cada herbicida e a estimativa da quantidade de diuron e sulfentrazone sorvida em cada substrato. A terra preta de índio apresentou a maior capacidade de sorção para diuron e sulfentrazone, seguida pela fibra de coco. As doses de diuron para terra preta indiana e areia + terra preta indiana não foram eficazes no controle da população de plantas. Os substratos misturados com areia apresentaram menor capacidade de sorção que os demais, exigindo maior cuidado ao utilizar herbicidas. A matéria orgânica, o pH e a textura do solo são fatores que contribuíram para a sorção de herbicidas.
Palavras-chave: inibição, fitotoxicidade, matéria orgânica.

1. INTRODUCTION

The growing increase in agricultural activities and cultivated areas in Brazil has led to an increase in the use of pesticides, mainly of the herbicide class [1, 2]. The high use of herbicides is due to the appearance of weeds, which must be controlled at the appropriate time to avoid losses in crop productivity due to competition [3].

However, 60 to 70% of phytosanitary products applied in agricultural fields do not reach the target of interest, resulting in the deposition and accumulation in the soil [4], where they undergo physical, chemical and biological degradation and contaminate aquatic systems [5]. The indiscriminate use of herbicides has often caused environmental problems due to the scarcity of information on their dynamics in different soils, resulting in simple recommendations for different cultivation conditions [6]. Furthermore, the efficiency in pre-emergence weed control is directly or indirectly affected by interactions between soil attributes and herbicide components [7], which is still not well understood.

Understanding the interactions between herbicide and soil is essential for the safe use of this class of pesticide [8]. After being applied, the herbicide can be sorbed, leached or degraded by physical, chemical or biological processes, or even absorbed by plants [3]. Sorption refers to a general process, without any distinction between specific adsorption, absorption and precipitation processes [7]. When sorbed, the herbicide molecule becomes unavailable for uptake by weeds and only returns to the soil solution after desorption processes [9].

Among herbicides used for the pre-emergence weed control, Diuron is mainly indicated for pineapple, alfalfa, cotton, banana, cocoa, coffee, sugarcane, citrus, wheat crops, among others [2, 10]; and Sulfentrazone, which efficiently controls cyperaceae, mono and dicot species [11]. Diuron (3-(3,4-dichlorophenyl)-1,1-dimethylurea) is a molecule belonging to the chemical group of ureas, which acts on photosystem II by binding to protein D1 at the QB protein coupling site to interrupt the flow of electrons between photosystems II and I [12]. Diuron has low water solubility (35.6 mg L^{-1} at 20ºC), is slightly volatile and moderately persistent (half-life = 89 days) [13].

On the other hand, Sulfentrazone (N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-methyl-5-oxo-1H-1,2,4-triazol-1 -yl]phenyl]methanesulfonamide) belongs to the chemical group of triazolinones, which is a weak acid (pKa = 6.56) [14], and acts by inhibiting the protoporphyrinogen oxidase enzyme (PROTOX), responsible for the oxidation of protoporphyrinogen to protoporphyrin IX, in the biosynthesis of chlorophyll [15]. Sulfentrazone has water solubility dependent on soil pH, being 0.78 g L^{-1} at pH 7.0 [16], and has half-life of 121 days in sandy soils and 302 days in clayey soils [17].

However, there is lack of information about the sorption of these herbicides in different substrates in the soil. Substrates form the basis of root fixation to support the plant and have distinct and specific characteristics that can absorb greater or lesser amount of applied herbicides. Therefore, the aim of this study was to quantify the sorption of diuron and sulfentrazone herbicides in different substrates by means of bioassays in greenhouse.

2. MATERIAL AND METHODS

2.1 Collection and characterization of substrates

Sand, coconut fiber (FC) and loam (L) substrate samples were collected on properties of Procópio farms, FMI farm (located on Highway AM 10, km 113, 02º41'55.44"S; 59º25'53.66"W) and Panorama farm (located on Highway AM 10, km 86, 02º37'05.02"S; 59º40'53.87"W), both in Rio Preto da Eva-AM. Indian Black Earth (IBE) was collected in the Costa do Laranjal community (located on Highway AM 070, 03º07'21"S; 60º18'09.6"W) in Manacapuru-AM.

Coconut fiber, Argissolo loam and IBE were submitted to chemical analysis, performed at the Department of Soil Science, 'Luiz de Queiroz' School of Agriculture (ESALQ), Table 1.

Table 1. Chemical composition of Coconut Fiber, Loam and Indian Black Earth substrates.

Substrates	pH	P	K^+	Na^+	Ca^{2+}	Mg^{2+}	Al^{3+}	H+Al	SB	t
	H_2O	.... mg dm^{-3}		.... $cmol_c$ dm^{-3}						
FC	-	29.00	0.40	-	0.75	0.10	-	-	-	-
L	6.70	56.50	0.30	-	2.80	0.90	0.00	1.60	3.80	-
IBE	6.10	80.00	105.00	3.00	5.70	1.60	0.00	2.76	7.78	7.58
Substrates	T	V	OM	Zn	Fe	Mn	Cu	B	S	CTC
	$cmol_c$ dm^{-3}	….%....	g kg^{-1}	…………….... mg kg^{-1} ….…..….….						
FC	-	-	95.27	75.00	-	162.00	22.00	16.00	0.04	1.27
L	-	70.40	25.70	8.15	28.8	3.25	3.00	<0.15	<6.00	4.00
IBE	10.30	73.30	43.40	7.30	0.00	58.62	0.55	-	-	112.30

H+Al: potential acidity; SB: sum of bases; t: effective cation exchange capacity: T: potential cation exchange capacity; V: base saturation; OM: organic matter.

2.2 Sample preparation

Two experiments were carried out in greenhouse at the Faculty of Agricultural Sciences (FCA-UFAM). In the first experiment, coconut fiber, Argisol loam, IBE substrates and their mixtures with sand washed in hydrochloric acid solution (1 mol L^{-1}) in filtered water were used. Cucumber (*Cucumis sativus* L. var Aodai) was the indicator plant to evaluate the sorption of herbicides on substrates. In the second experiment, only sand was used as substrate, defining it as the control substrate, and cucumber as indicator plant to evaluate the effects of the herbicide on the plant.

In the experiment with substrates, the herbicide solution was applied with a graduated pipette, always from the lowest to the highest herbicide dose. Substrates were homogenized in polyethylene bags and placed in 500 ml plastic cups. Cucumber was sown after herbicide application, with 4 seeds per cup, at a depth of 1 cm. After seedling emergence, thinning was performed, keeping only two seedlings in each cup. Each cup consisted of an experimental unit.

The water content in the cups was maintained at approximately 60% of field capacity (246 ml kg of substrate), with daily weighing and replacement of water. The nutritional needs of plants were supplied by means of a modified nutrient solution of Hoagland and Arnon (1950) [18] provided in irrigation in all treatments on alternate days.

2.3 Experimental design

Treatments with substrates were installed in a completely randomized experimental design in a 2 x 6 x 6 factorial arrangement. The factors evaluated were: factor A: two herbicides (diuron and sulfentrazone), factor B: six doses of each herbicide: diuron (0; 250; 500; 1,000; 2,000 and 4,000 g ai ha^{-1}), sulfentrazone (0; 75; 150; 300; 600 and 1,200 g ai ha^{-1}) and Factor C: six substrates [Fiber coconut (FC); Loam (L); Indian Black Earth (IBE); Sand + Coconut Fiber (S+FC); Sand + Loam (S+L) and Sand + Indian Black Earth (S+IBE)], with four replicates, totaling 288 experimental units.

Treatment with washed sand was carried out in a completely randomized design, arranged in a 2 x 7 factorial scheme, with factor A: two herbicides (diuron and sulfentrazone), and factor B: seven doses of each herbicide (diuron 0; 2; 4; 8; 12; 16 and 20 g ai ha^{-1}) and sulfentrazone (0; 2; 4; 8; 12; 16 and 20 g ai ha^{-1}), with 4 replicates, totaling 56 experimental units.

2.4 Laboratory analyses

At 14 days after sowing (DAS), cucumber plants were cut and taken to drying in a forced ventilation oven at 65° C until constant weight, and subsequent weighing on a precision scale to determine the dry mass.

To calculate the lethal dose (I_{50}) value, the dry matter weight values obtained at dose zero as 100% growth and the values of the other treatments, in relation to 100% growth were considered. The diuron and sulfentrazone doses that resulted in 50% inhibition of the dry matter weight of cucumber plants were determined with graphs of diuron and sulfentrazone doses *versus* dry matter weight of cucumber seedlings, expressed as percentage, in relation to control.

The lethal dose (I_{50}), related to the degree of inactivation of the herbicide by the soil, was obtained by plotting the shoot dry weight of the bioindicator plant by the diuron dose applied to the soil. This index corresponds to the dose that inhibited the growth of 50% of the shoot dry weight of the bioindicator plant. The difference in I_{50} obtained between sand and each substrate was considered to be equal to the amount of herbicide inactivated by the soil.

2.5 Statistical analysis

Data from each sorption experiment were submitted to analysis of variance by the F test ($p \leq 0.05$) with the aid of the SISVAR 5.6 statistical software [19]. When significant, results were analyzed using regressions. For the selection of the regression equation, the significance of the F test, the value of the determination coefficient and the equation of best fit to the original data were considered, using the SigmaPlot 12.0 software (Systat Software Inc. San Jose, USA).

3. RESULTS AND DISCUSSION

To evaluate the sorption of herbicides on different substrates, it was first analyzed whether the doses applied were able to influence the dry matter weight of the indicator plant through analysis of variance (Table 2). For the substrate factor, the doses applied to the washed sand significantly influenced the dry matter weight of cucumber plants using both diuron and sulfentrazone herbicides. However, not all herbicide doses significantly affected the dry matter weight considering the substrates under study. This was the case with diuron doses for FC and A+IBE substrates, although the other substrates (L, IBE, S+FC, S+L and SAND) were significantly influenced by the applied doses. On the other hand, sulfentrazone doses were more effective and affected the dry matter weight of indicator plants in most substrates, with the only exception of S+FC.

Table 2. Analysis of variance (ANOVA) for shoot dry matter weight of cucumber seedlings sown in different substrates.

	F test values	
Source of Variation	Diuron	Sulfentrazone
FC	0.71^{ns}	3.34*
L	6.48**	9.90**
IBE	3.88*	7.95**
S+FC	5.22**	1.03^{ns}
S+L	22.26**	10.82**
S+IBE	1.74^{ns}	10.40**
SAND	21.19**	47.55**

Fiber coconut (FC); Loam (L); Indian Black Earth (IBE); Sand + Coconut Fiber (S+FC); Sand + Loam (S+L) and Sand + Indian Black Earth (S+IBE); **significant at 1% probability ($p < 0.01$); *significant at 5% probability ($0.01 \leq p < 0.05$); ^{ns} = not significant by the F test, ($p \geq 0.05$).

This greater ease with which a substrate can undergo alteration in the medium is due to the physicochemical characteristics of each substrate, especially those related to buffering. According to Rocha et al. (2013) [20], soils rich in organic matter (OM) have high retention capacity, which causes a decrease in the leaching potential and bioavailability of herbicides to plants and microorganisms. Thus, probably FC and S+IBE substrates would need higher doses to the point of being influenced by diuron doses, since diuron absorption occurs preferentially by roots and its translocation is via xylem [21].

For the washed sand substrate, the inhibitory dose of 50% of the dry mass of cucumber seedlings was higher for diuron than sulfentrazone, with values of 2.5 and 2.1 g ai ha^{-1}, respectively (Table 3).

Table 3. Sorption of herbicides by organic matter in different substrates for seedling production, Manaus, 2018.

Substrate	I_{50} (g ai ha^{-1})		SR		I_{50} inactivated (g ai ha^{-1})		OM (kg ha^{-1})	mg ai inactivated (kg^{-1} OM)	
	Diur.	Sulfen.	Diur.	Sulfen.	Diur.	Sulfen.		Diur.	Sulfen.
FC	1131.00	200.00	451.40	94.24	1128.5	197.90	190540	5.92	1.04
L	750.00	50.28	299.00	22.94	747.50	48.18	51400	14.54	0.94
IBE	-	400.00	-	189.48	-	397.90	86800	-	4.58
S+FC	260.00	25.00	103.00	10.90	257.50	22.90	-	-	-
S+L	125.00	15.00	49.00	6.14	122.50	12.90	-	-	-
S+IBE	-	90.00	-	41.86	-	87.90	-	-	-
SAND	2.50	2.10	-	-	-	-	-	-	-

Fiber coconut (FC); Loam (L); Indian Black Earth (IBE); Sand + Coconut Fiber (S+FC); Sand + Loam (S+L) and Sand + Indian Black Earth (S+IBE); Diuron (Diur.); Sulfentrazone (Sulfen.); Lethal dose (I_{50}); Sorption ratio (SR); and Organic matter (OM).

The growth behavior of the test plant in the control substrate under both herbicides was sigmoidal (Figure 1), with fitted regressions with determination coefficients above 92%. The herbicide applied to this material is free to be absorbed by cucumber seedlings. Therefore, even the lowest dose of both herbicides was enough to inhibit the growth of the test plant. Conversely, FC exhibited the highest resilience among substrates, requiring at least 1131.0 g a.i ha^{-1} of diuron to inhibit 50% the growth of test plants. As for sulfentrazone, IBE showed to be the least indicated substrate, as it showed the highest I_{50} value (400 g ai ha^{-1}) probably due to its high OM content (43.4 g kg^{-1}, Table 1).

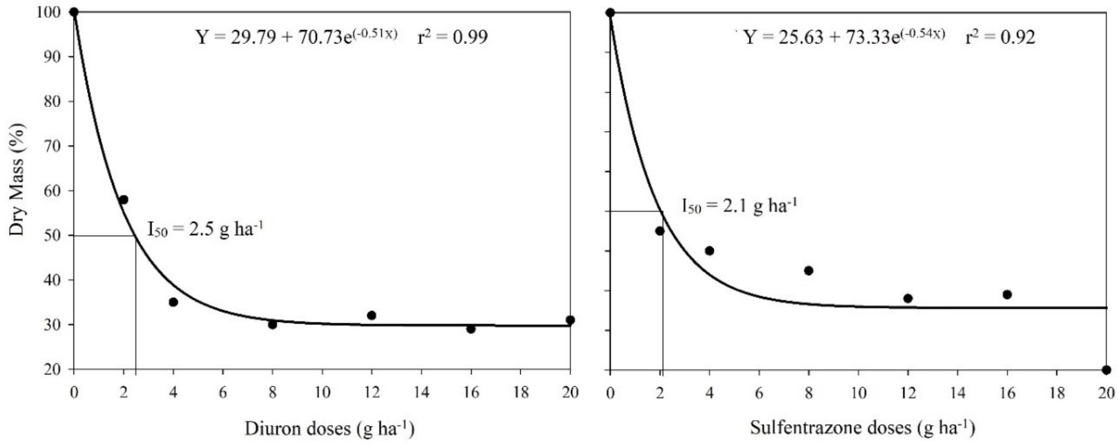

Figure 1. Regression and I_{50} value of diuron and sulfentrazone as a function of the shoot dry matter weight of cucumber seedlings in washed sand.

According to the pattern of the results found (Table 3), not only the amount of OM influences the retention of herbicides, but their physical and chemical characteristics are factors that must be taken into account. Although the absence of specific analyses on the physical and chemical fractionation of OM is a limiting factor in this research, this statement is based on the fact that each kilogram of OM from IBE was able to inactivate 4.58 mg of the sulfentrazone ai compared to FC, for example, which has 2 times more OM than IBE, but was sufficient to inactivate only 1.04 mg of the herbicide ai according to Melo et al. (2010) [22], for herbicides with high adsorption capacity, the higher the SOM content, the greater the herbicide adsorption and, therefore, the lower the leaching. However, leaching will be enhanced if the soil is sandy [23], compared to silty or clayey soils.

As for the experiment with diuron doses, regressions were obtained with determination coefficient above 94% for all substrates (Figure 2). S+L and S+FC substrates had the lowest I_{50} compared to the other substrates, with values of 125 g ha^{-1} and 260 g ha^{-1}, respectively, being the most suitable for the availability of this herbicide in the soil. The lower sorption of these substrates is probably associated with the use of sand in their composition, since among soils with different textures [24], sandy soils (Quartzarenic Neossolo) have the lowest sorption. On the other hand, IBE and S+IBE are substrates with high diuron sorption capacity and not even the highest dose could inhibit 50% of the test plant.

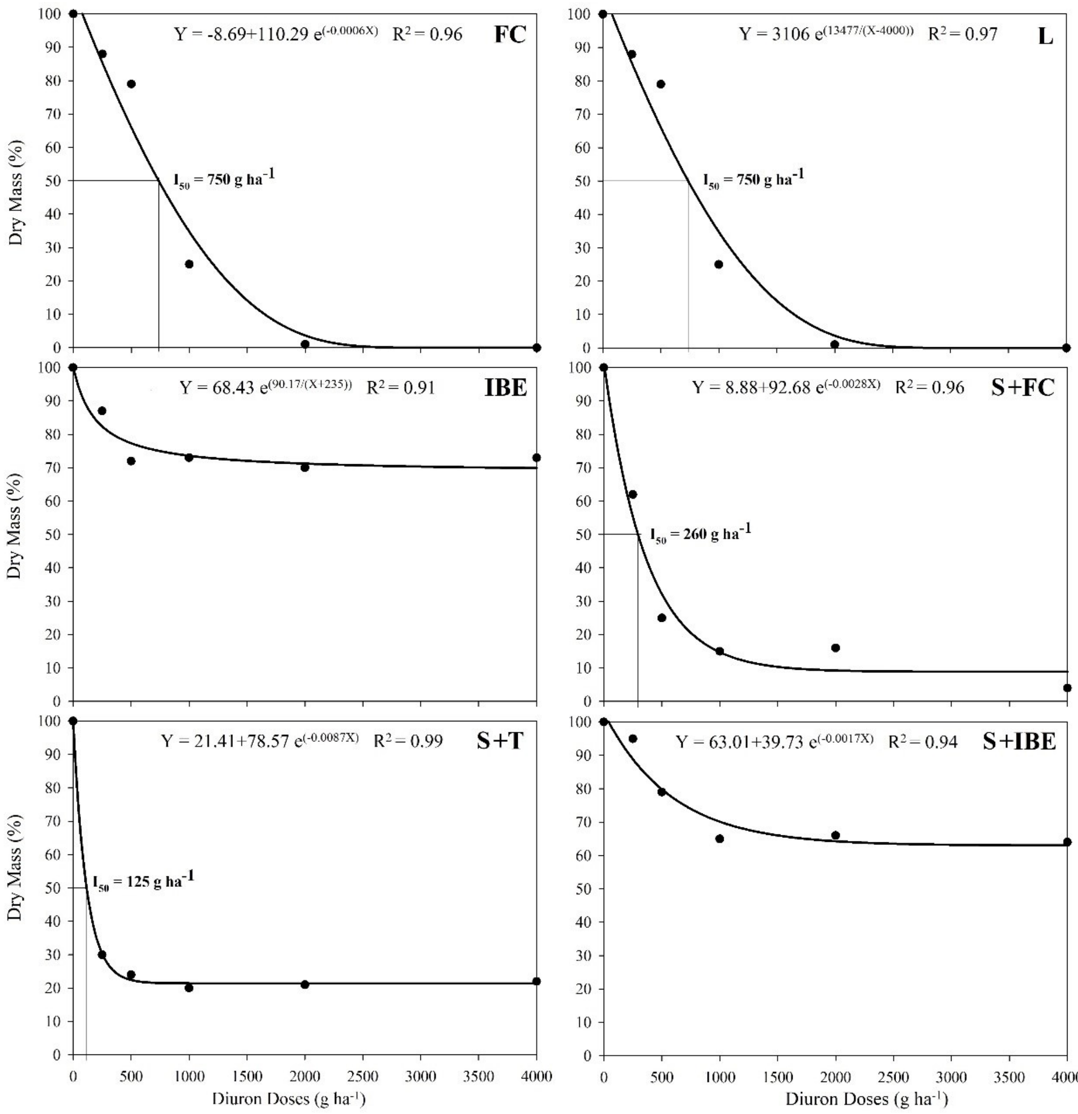

Figure 2. Regression of diuron as a function of shoot dry matter weight of cucumber seedlings in different substrates. Fiber coconut (FC); Loam (L); Indian Black Earth (IBE); Sand + Coconut Fiber (S+FC); Sand + Loam (S+L) and Sand + Indian Black Earth (S+IBE); and Lethal dose (I_{50}).

The treatments with sulfentrazone presented I50 values ranging from 15 g ha^{-1} to 400 g ha^{-1}, for the S+L and IBE substrates respectively, with the lowest values being observed in the substrates with sand (S+FC, S+L and S+IBE) (Figure 3). Therefore, greater care is recommended when applying sulfentrazone for pre-emergent control, in substrates with higher sand contents, aiming to reduce the risk of contamination. Despite the fact that the herbicide is a Protox inhibitor, capable of selectively controlling broadleaf weeds [25, 26].

However, the results show that IBE and also FC, if used alone, have a high capacity to sorb sulfentrazone, requiring doses of 200 and 400 g ha-1 respectively, to control 50% of population (Figure 3). This higher sorption capacity is probably due to the high OM content of these substrates due to the high specific surface and adsorption sites for herbicide particles [27]. In conventional cultivation situations, the sorption process is dependent on the soil OM content and pH [15], and soil classes such as Neosols may present higher I_{50} compared to Argisols as long as pH and OM are high.

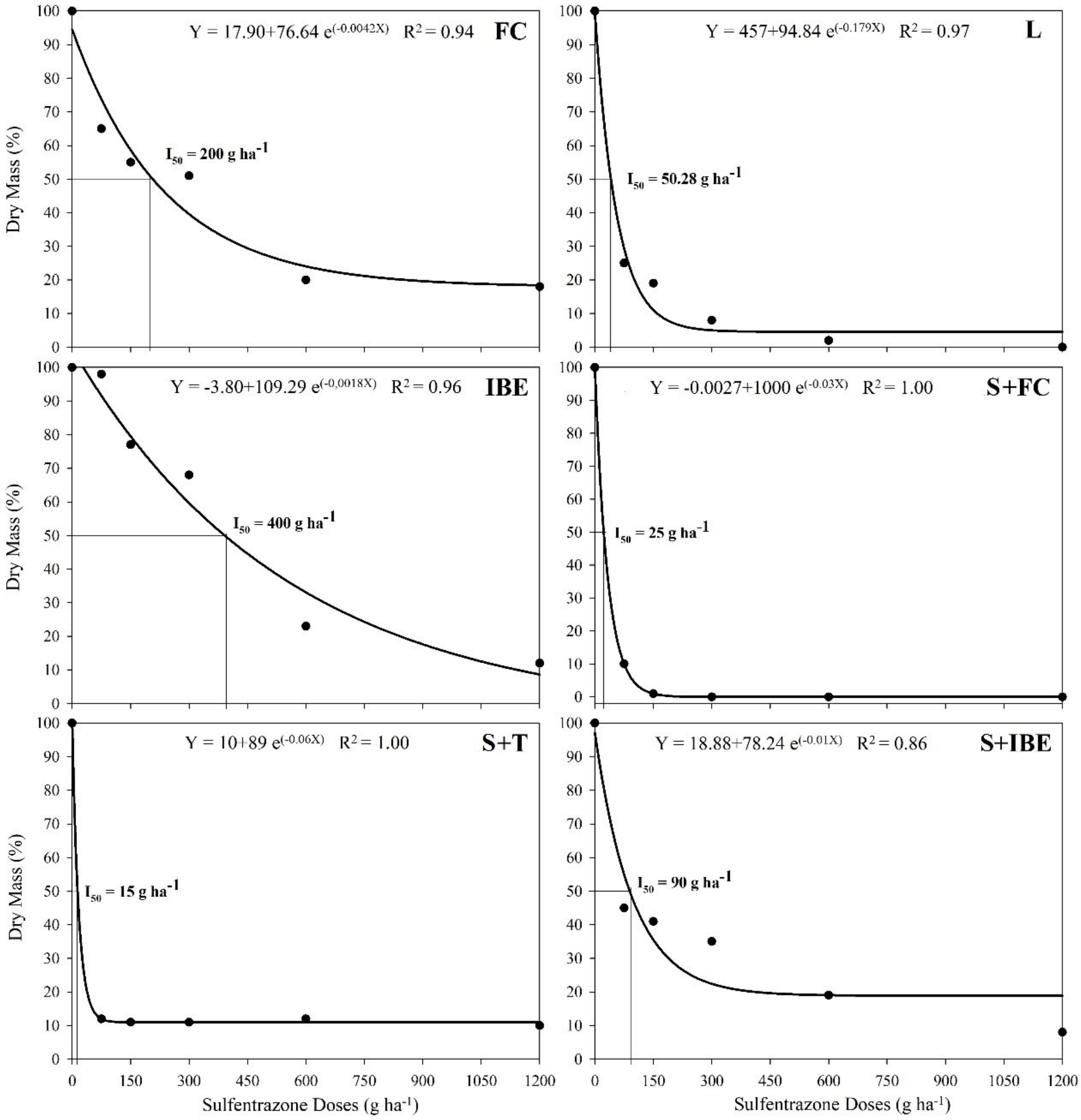

Figure 3. Regression and I_{50} value of sulfentrazone as a function of shoot dry matter weight of cucumber seedlings in different substrates. Fiber coconut (FC); Loam (L); Indian Black Earth (IBE); Sand + Coconut Fiber (S+FC); Sand + Loam (S+L) and Sand + Indian Black Earth (S+IBE); and Lethal dose (I_{50}).

The phytotoxic effects on cucumber seedlings for diuron and sulfentrazone in the sand substrate are observed from the dose of 2 g ia ha^{-1}. Symptoms were characterized by the bleaching of leaf tissues followed by necrosis with reduced dry matter and plant death as doses increased (Figure 4). According to Dayan et al. (2007) [28], such visual symptoms are due to the mechanism of action of these herbicides. According to Marchi et al. (2008) [25] and Oliveira Junior (2011) [26], the action of sulfentrazone is to inhibit protoporphyrin oxidase, which causes the death of plants when they come into contact with soil treated with this herbicide. According to Oliveira Junior (2011) [26], the action of diuron is to inhibit photosystem II, which blocks the electron transport chain, interrupting CO_2 fixation and ATP and NADPH production, with consequent appearance of interveinal and leaf edge chlorosis due to chlorophyll photooxidation.

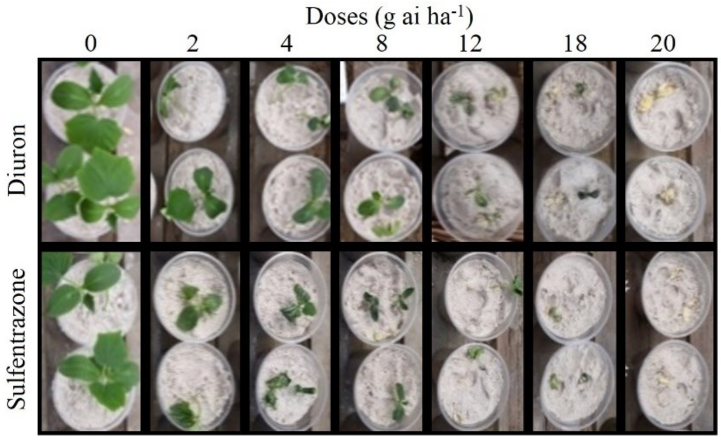

Figure 4. Phytotoxicity of cucumber under different diuron and sulfentrazone doses in washed sand substrate.

For diuron doses on different substrates, phytotoxicity symptoms are observed from dose of 1000 g ai ha^{-1} for FC and L substrates. For S+FC and S+L, symptoms are observed from dose of 250 g ai ha^{-1}. For the IBE substrate, no phytotoxicity symptoms were observed, and only for A+IBE at dose of 4000 g ai ha^{-1} that interveinal chlorosis was observed (Figure 5). At sulfentrazone dose of 75 g ai ha^{-1}, plants showed phytotoxicity symptoms in L, S+FC and S+L substrates. For FC and S+IBE substrates, phytotoxicity was observed at dose of 150 g ai ha^{-1}. For Indian Black Earth, symptoms were observed at dose of 300 g ai ha^{-1}.

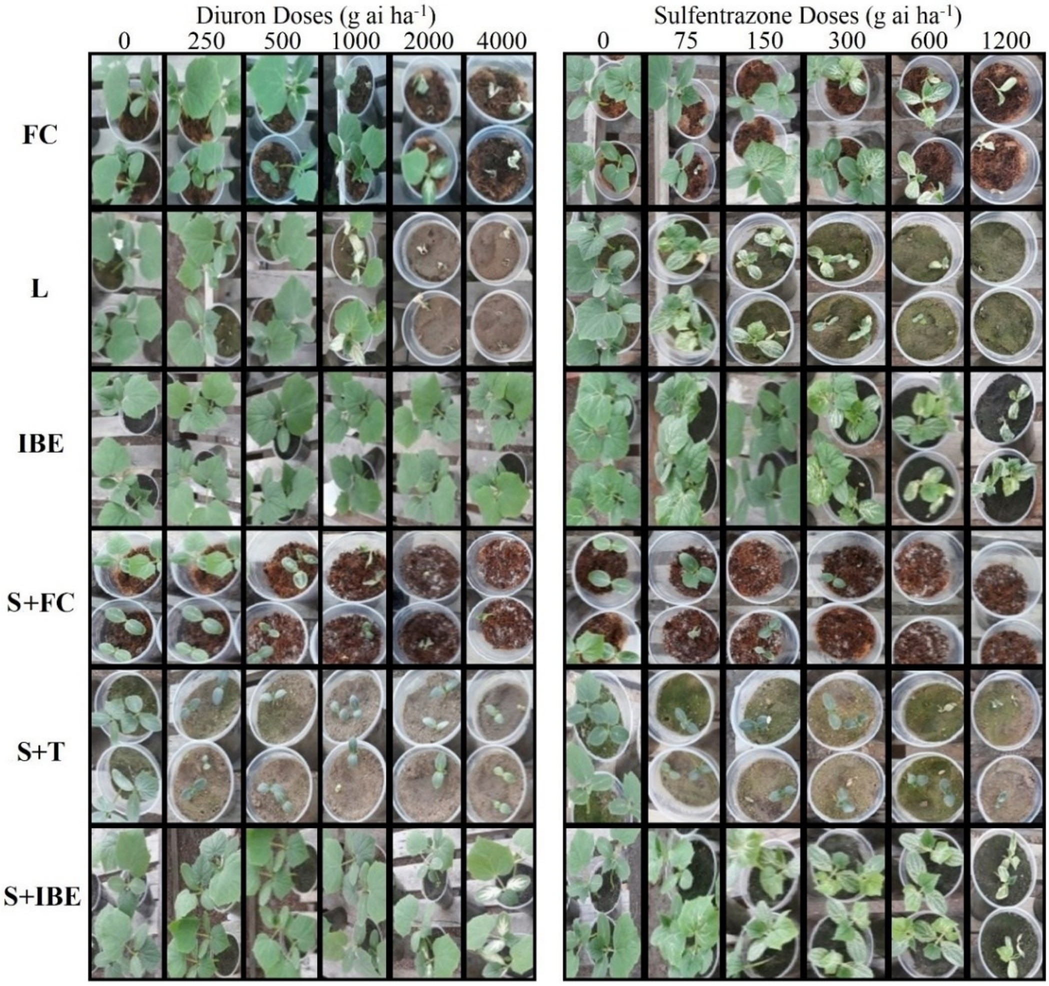

Figure 5. Phytotoxicity of cucumber under different diuron and sulfentrazone doses on different substrates. Fiber coconut (FC); Loam (L); Indian Black Earth (IBE); Sand + Coconut Fiber (S+FC); Sand + Loam (S+L) and Sand + Indian Black Earth (S+IBE).

Considering the control of specific plants using pre-emergent herbicides, Sulfentrazone had a greater control capacity than Diuron, reaching I50 in all substrates used. Lower doses are necessary in relation to Diuron. However, more care is needed in substrates containing high levels of sand, so as not to contaminate the environment due to excessive dosage. The IBE substrate showed high sorption capacity, often making the application of herbicides unfeasible. Therefore, it is necessary to seek other ways to control weeds in it.

4. CONCLUSIONS

Indian Black Earth showed the highest sorption capacity for diuron and sulfentrazone, followed by coconut fiber. The diuron doses for Indian Black Earth and Sand + Indian Black Earth were not effective in controlling the plant population.

The substrates mixed with sand showed lower sorption capacity than the others, requiring greater care when using herbicides.

The organic matter, pH and soil texture are factors that contributed to the sorption of herbicides. However, studies are needed to evaluate the isolated effects of these on the sorption of diuron and sulfentrazone.

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