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Article

Leaching and Persistence of Sulfentrazone when Mixed with Adjuvants

Lixiviação e Persistência do Sulfentrazone em Função da Mistura com Adjuvantes

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ABSTRACT - The objective of this work was to study the influence of adjuvants on leaching and persistence of sulfentrazone in a Red-Yellow Ultisol. The soil was stored in PVC columns of 10 cm diameter and 50 cm length. The treatments were composed of sulfentrazone mixed with six adjuvants (Adesil®, Break-Thru®, Assist®, Hoefix®, Fera® and Nortox® Vegetable Oil), a treatment with sulfentrazone without any adjuvant and a treatment without the herbicide. In the treatments with sulfentrazone, the herbicide was applied at a rate of 1.0 kg ha^{-1} at the top of the columns, and 12 hours after application, the columns were subjected to simulated rainfall (60 mm). To confirm leaching of sulfentrazone, soil samples were collected every 5 cm up to 50 cm in each column and transferred to 300 cm^3 pots to conduct a bioassay using the species *Sorghum bicolor*. At 21 days after emergence (DAE) of sorghum, visual assessment of intoxication was performed and shoot dry matter percentage was determined. To determine the influence of adjuvants on persistence of sulfentrazone in the soil, new sorghum seeds were planted as soon as the sorghum plants from the first planting were cut; also, phytotoxicity scores were assigned and shoot dry matter was determined at 21 DAE. This procedure was repeated up to 173 days after herbicide application, when the plants showed no more visual symptoms of intoxication. The adjuvant Break-Thru® was effective at reducing leaching of sulfentrazone. The presence of adjuvants applied together with sulfentrazone did not influence the persistence of the herbicide in the study soil. Persistence of sulfentrazone lasted for 143 days.

Keywords: *Sorghum bicolor*, Red-Yellow Ultisol, phytotoxicity, environmental contamination.

RESUMO - Objetivou-se neste trabalho estudar a influência de adjuvantes na lixiviação e na persistência do sulfentrazone em um Argissolo Vermelho-Amarelo. O solo foi acondicionado em colunas de PVC de 10 cm de diâmetro por 50 cm de comprimento. Os tratamentos foram compostos pela combinação do herbicida sulfentrazone misturado a seis adjuvantes (Adesil®, Break-Thru®, Assist®, Hoefix®, Fera® e Óleo Vegetal Nortox®), um tratamento com sulfentrazone sem adjuvante e um tratamento sem herbicida. Nos tratamentos com sulfentrazone, o herbicida foi aplicado na dose de 1,0 kg ha^{-1} no topo das colunas, e, 12 horas após a aplicação, as colunas foram submetidas à chuva simulada (60 mm). Para confirmação da lixiviação do herbicida, em cada coluna, foram coletadas amostras de solo a cada 5 cm até 50 cm e transferidas para vasos de 300 cm^3, onde foi realizado o bioensaio utilizando a espécie Sorghum bicolor. Aos 21 dias após a emergência (DAE) do sorgo, foi avaliada a intoxicação visual e a matéria seca da parte aérea. Para determinação da influência dos adjuvantes na persistência do sulfentrazone no

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solo, assim que as plantas de sorgo do primeiro plantio foram cortadas, realizou-se o plantio de novas sementes de sorgo e, aos 21 DAE, foram dadas notas de fitotoxicidade e determinou-se a matéria seca da parte aérea. Esse procedimento foi repetido até 173 dias após a aplicação do herbicida, quando não foram mais observados sintomas de intoxicação nas plantas. O adjuvante Break-Thru® foi eficiente em reduzir a lixiviação do sulfentrazone. A presença dos adjuvantes aplicados junto ao herbicida não influenciou na persistência do sulfentrazone no solo estudado. A persistência do herbicida foi de 143 dias.

Palavras-chave: *Sorghum bicolor*, Argissolo Vermelho-Amarelo, fitotoxicidade, contaminação ambiental.

INTRODUCTION

One of the pre-emergent herbicides most frequently used in agricultural systems is sulfentrazone N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-methyl-5-oxo-1H-1,2,4-triazole-1-yl]phenyl]methanesulfonamide. This molecule is recommended for weed control in crops such as soybeans, sugar cane, coffee and eucalyptus (Rodrigues and Almeida, 2011). Low sorption of sulfentrazone (Passos et al., 2013; Freitas et al., 2014), together with its long persistence (White et al., 2010), facilitates its mobility in the soil profile (Monquero et al., 2010; Melo et al., 2010). As a result, there may be reduced effectiveness in weed control and contamination of the environment, especially of the water table.

Sulfentrazone is a weak acid with a dissociation constant (pK) of 6.56. It is classified as little volatile (vapor pressure of 1 x 10^{-9} mmHg to 25 ^{o}C), with low sorption in soil (K_{oc} 43) (Rodrigues and Almeida, 2011). Microbial degradation is considered to be the main form of dissipation of sulfentrazone. Water solubility changes on the basis of pH:110, 780 and 1,600 mg L^{-1} at pH 6.0, 7.0 and 7.5, respectively (Tomlin, 2011). Therefore, this herbicide may present high persistence and mobility in soil and aquatic systems (EPA, 2015), and possibly cause serious environmental problems, in addition to increasing the risk of intoxication of sensitive succeeding crops (carryover effect).

Passos et al. (2015), while evaluating leaching of sulfentrazone in different Brazilian soils, including Haplic Alfisol, Ultisol, and Entisol, detected the presence of the herbicide until the last section tested (30 cm). Braga et al. (2016) evaluated leaching of sulfentrazone in an Entisol (Quartzipsamment) and a Red-Yellow Oxisol, and detected the presence of the herbicide at a depth of 45 and 35 cm, respectively. Blanco et al. (2010) assessed potential carryover effects of sulfentrazone on beet grown after cultivation of sugar cane. They concluded that the herbicide persisted for 601 days after treatment (DAT) at the lowest rate (0.6 kg ha^{-1}) tested. For the rate of 1.2 kg ha^{-1}, the herbicide still persisted until the end of the trial, at 704 DAT.

The use of adjuvants mixed with herbicides in foliar applications (post-emergence) is a well-known procedure. The main function of these products is to optimize herbicide activity in plants, increasing contact area, adherence and penetration of the product in the leaves. This optimization promoted by the adjuvants occurs through solubilization of the cuticle present in the leaves of plants, which facilitates herbicide penetration through the stomata to reduce vapor pressure on the surface of guard cells. It also facilitates herbicide movement along the cell walls into the interior of leaves by decreasing interfacial tension, resulting in increased herbicide movement through intercellular spaces. These properties can be chemical, physical or biological (Bruce and Carey, 1996; Sharma et al., 1996). According to their functions, adjuvants may be classified as spray modifiers, e.g., dyes, drift retardants and thickeners; and activators, such as surfactants, vegetable oils, mineral oils and silicon derivatives (Monquero, 2014).

Application of adjuvants at pre-emergence can reduce production of satellite drops, improving spray distribution in the nozzles and herbicide deposition on the target (Bachega et al., 2009). There are few studies in the Brazilian and international literature describing the effects of adjuvants tank-mixed with pre-emergent herbicides (Kucharski and Sadowski, 2011). However, some studies have reported that adjuvants can reduce herbicide leaching along the soil profile (Reddy, 1993; Negrisoli et al., 2005; Bachega et al., 2009; Kucharski and Sadowski, 2011).

The use of techniques capable of reducing leaching of sulfentrazone may represent a major step forward to reduce the risk of environmental contamination by this compound. All things considered, the objective of this research was to evaluate the potential of adjuvants Adesil®, Break-Thru®, Assist®, Hoefix®, Fera® and Nortox® Vegetable Oil of reducing leaching of sulfentrazone in the soil, as well as the influence of these adjuvants on persistence of this herbicide.

MATERIAL AND METHODS

The experiment used samples of soil classified as Red-Yellow Ultisol (Embrapa, 2006) with sandy clay loam texture, collected at depths of 0 to 20 cm. These samples were characterized chemically and physically. The following characteristics were determined: pH in H_2O (6.51), Mehlich-1 extractable-P and K^+ (17.1 mg dm^{-3} and 104 mg dm^{-3}, respectively), levels of exchangeable Ca^{2+}, Mg^{2+} and Al^{3+} extracted in KCl 1 mol L^{-1} (4.12 $cmol_c$ dm^{-3}, 0.87 $cmol_c$ dm^{-3} and 0.0 $cmol_c$ dm^{-3}, respectively), H+Al in Calcium Acetate 0.5 mol L^{-1} - pH 7.0 (2.1 $cmol_c$ dm^{-3}) and OM (4.06 dag kg^{-1}). Based on the values determined in the soil analyses, the following parameters were calculated: effective and potential CEC (5.26 $cmol_c$ dm^{-3} and 7.36 $cmol_c$ dm^{-3}, respectively), total exchangeable bases (TEB) (5.26 $cmol_c$ dm^{-3}), aluminum saturation percentage (m) (0.0%) and base saturation percentage (V) (71.5%). Soil texture was determined according to Embrapa (1996), and the following values were found: coarse sand = 32 dag kg^{-1}; fine sand = 16 dag kg^{-1}; silt = 19 dag kg^{-1}; and clay = 33 dag kg^{-1}.

The soil samples were placed into polyvinyl chloride (PVC) columns of 10 cm in diameter and 50 cm in length, whose inside walls had been previously prepared and paraffin-embedded to reduce percolation of water. All the columns were marked and cut at every 5 cm, and they had a removable side cover.

The columns were saturated after being filled with the soil samples. At this stage, they were placed inside a container with water up to the level of 80% height for 48 hours, to moisten the samples from bottom to top, thereby avoiding the formation of air bubbles trapped in the pores. Afterwards, they were left to rest in an upright position for 72 hours for drainage of excess water, until reaching field capacity.

The treatments consisted of mixtures of sulfentrazone with six adjuvants: Adesil®, Break-Thru®, Assist®, Hoefix®, Fera® and Nortox® Vegetable Oil, applied at rates of 0.02%, 0.2%, 2%, 0.2%, 0.2% and 2% of the spray volume, respectively (the rates were based on manufacturers' instructions for the adjuvants); one treatment had sulfentrazone without adjuvant; and one treatment had no herbicide.

In the treatments with sulfentrazone, the herbicide was applied at a rate of 1,000 g ha^{-1} of the active ingredient on top of the columns, using a CO_2 pressurized sprayer, fitted with XR 110.02 tips, adjusted to apply a spray volume 150 L ha^{-1}. Twelve hours after herbicide application, with the columns still in the upright position, a single depth of 60 mm of rain was simulated for a period of three hours. Rain gauges were attached to the side walls of the columns for measurement of the rainfall applied.

After this step, the columns remained for another 72 hours in the upright position, and after that they were placed in a horizontal position. On that occasion, the columns were opened on the side and the soil was sectioned every 5 cm.

To confirm leaching of sulfentrazone in each treatment, soil samples were collected at depths of 0-5, 5-10, 10-15, 15-20, 20-25, 25-30, 30-35, 35-40, 40-45 and 45-50 cm. Each fraction was placed in pots without drainage holes with a capacity of 300 cm^3 in which the bioindicator species sorghum (*Sorghum bicolor*) was planted. This species was used as a bioindicator of sulfentrazone in the soil because it is highly sensitive to the herbicide (Faustino et al., 2015; Silva et al., 2016; Madalão et al., 2017). For this reason, 7 seeds were added to the soil, and three seedlings were left after thinning. Irrigation was performed daily, according to the water requirement of sorghum.

At 21 days after emergence (DAE), phytotoxicity scores were assigned, ranging from 0 to 100, in which 0 corresponds to absence of symptoms and 100 to plant death (ALAM, 1974). At this stage, all plants were cut flush to the soil surface and dried in a forced air circulation oven

(70 ± 2 °C to constant weight, to determine shoot dry matter weight of the plant. In each treatment, data on shoot dry matter (SDM) of sorghum (*Sorghum bicolor*) were transformed into shoot dry matter (SDM) percentage of sorghum in comparison to the control without herbicide.

To determine the influence of the adjuvants on persistence of sulfentrazone in the soil, new sorghum seeds were planted as soon as the sorghum plants from the first planting were cut; seven seeds were added to the soil while three seedlings were left after thinning. At 21 DAE, phytotoxicity scores were assigned and shoot dry matter was determined. This procedure was repeated until there were no observable symptoms of herbicide intoxication in the plants, which occurred in the sixth cycle of planting of the bioindicator species.

After collection and tabulation of data, bar graphs were designed with the means of each variable in each treatment. The data were analyzed using descriptive statistics.

RESULTS AND DISCUSSION

As shown in Figure 1, there was reduced gain of shoot dry matter of sorghum when it was grown at depths of 0-5 and 5-10 cm for the treatments control (sulfentrazone applied alone), sulfentrazone + Adesil®, sulfentrazone + Hoefix®, sulfentrazone + Fera® and sulfentrazone + Nortox® Vegetable Oil. This indicates that there was no interference of adjuvants on leaching of sulfentrazone. However, for the treatments sulfentrazone + Break-Thru® and sulfentrazone + Assist® (mineral oil), there was reduced gain of shoot dry matter of sorghum only when it was grown at a depth of 0-5 cm. This result is indicative that these adjuvants reduced leaching of sulfentrazone in the study soil.

Leaching of sulfentrazone depends on the sorption ability of the herbicide in the soil; the herbicide is usually trapped in the first layers of soils whose sorption is high (Tatarková et al., 2013; El-Nahhal and Hamdona, 2017). The main sources of sites of sorption in the soil, which involve specific mechanisms for acid herbicides, are Fe and Al oxides of the clay fraction, in addition to those associated with soil organic matter (Assis et al., 2011; Oliveira Jr. et al., 2013; Werner et al., 2013). In soils with a very small amount of these compounds, the herbicide may leach to deeper layers, reach the water table and spread more easily into the water cycle.

The study soil had high levels of organic matter and high cationic exchange capacity (CEC), which led the herbicide to be retained in the first layers rather than leach to the deeper layers. Passos et al. (2015) conducted a study on leaching of sulfentrazone in four typical Brazilian soils (Alfisol, Oxisol, Humic Inceptisol and Entisol) and concluded that leaching was lower in the Humic Inceptisol, which contained the highest levels of organic matter and the highest CEC.

Observation of phytotoxicity symptoms (Figure 2) in the sorghum plants grown in different layers of the leaching columns showed that in the treatment sulfentrazone + Break-Thru®, phytotoxicity symptoms were evident only in the 0-5 cm layer (around 97%), while in the other treatments, including the control, phytotoxicity symptoms were found in the 0-5 cm layer (between 96% and 99%), and in the 5-10 cm layer, around 15% to 34%. Thus, it is clear that the adjuvant Break-Thru® is effective at reducing leaching of sulfentrazone.

Contact time between a pesticide and the soil influences the extent of the sorption process. For an herbicide to express all of its sorption capacity, it has to remain in direct contact with the soil for as long as possible (Vereecken et al., 2011). Sulfentrazone is considered to be hydrophilic (k_{ow} = 9.8); it has great affinity with the liquid phase of the soil (soil solution) and, consequently, lower affinity with the solid phase of the soil. In liquids that contain polar molecules, such as water, there is great attraction between these molecules, thus generating a surface tension (Kissmann, 1998). Break-Thru® is an adjuvant classified as a surfactant (spreader), and the main action of these products is to reduce the surface tension of water. Possibly, when the surface tension of water was broken, there was increased contact of the herbicide with the solid phase of the soil, thereby facilitating the interactions of the herbicide with the colloidal particles and, consequently, interactions between the herbicide and the soil.

Assist® is an adjuvant classified as an additive, which acts directly on the cuticle of plants, dissolving fats and cell membranes and eliminating barriers that reduce herbicide absorption (Vargas and Roman, 2006). In addition, it also acts as a sticker-spreader; in this way, there may

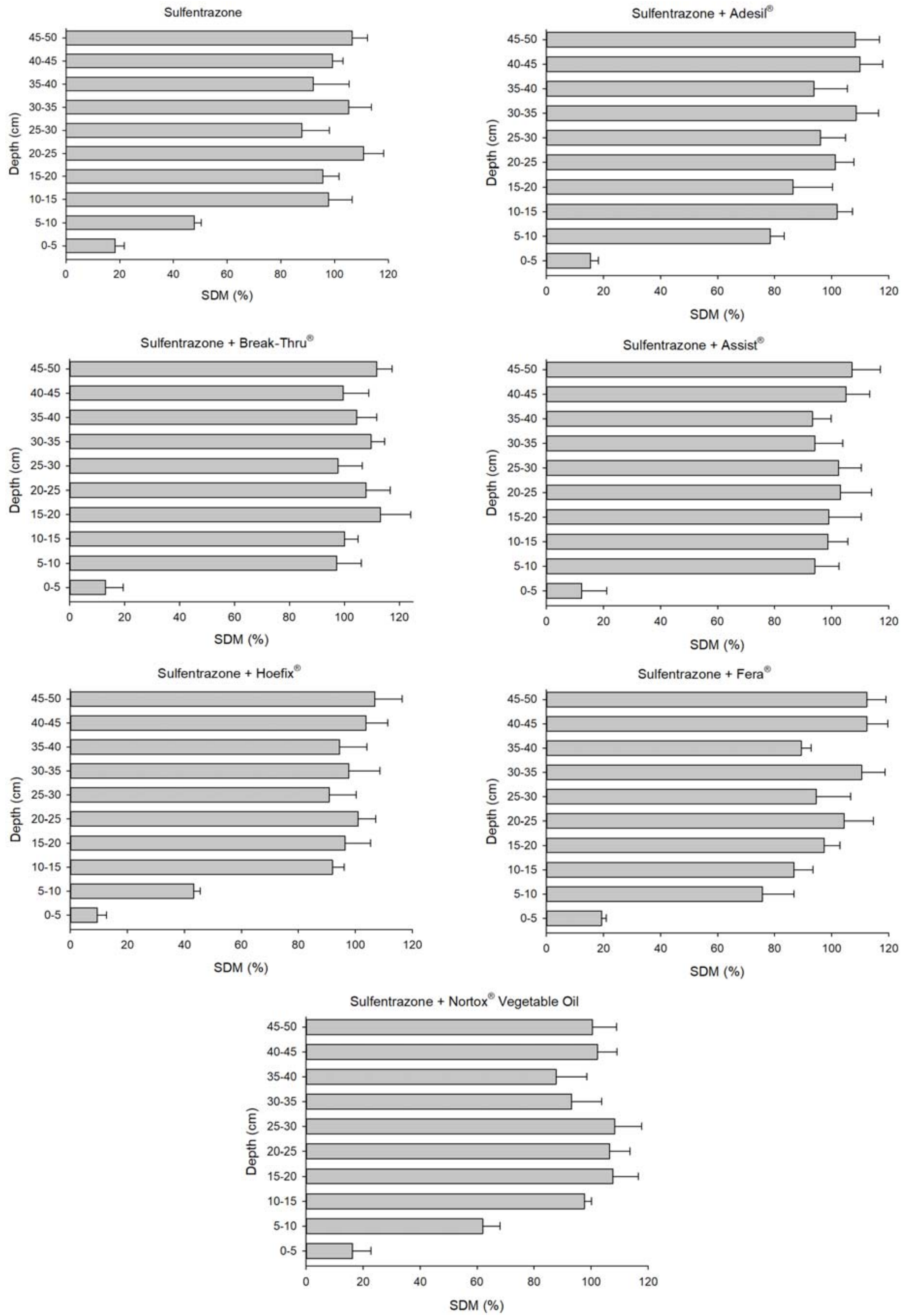

Figure 1 **-** Shoot dry matter (SDM) percentage of sorghum (*Sorghum bicolor*), in comparison to the control, grown at different depths, at 21 DAE, in soil contaminated with sulfentrazone, sulfentrazone + Adesil®, sulfentrazone + Break-Thru®, sulfentrazone + Assist®, sulfentrazone + Hoefix®, sulfentrazone + Fera® and sulfentrazone + Nortox Vegetable Oil®.

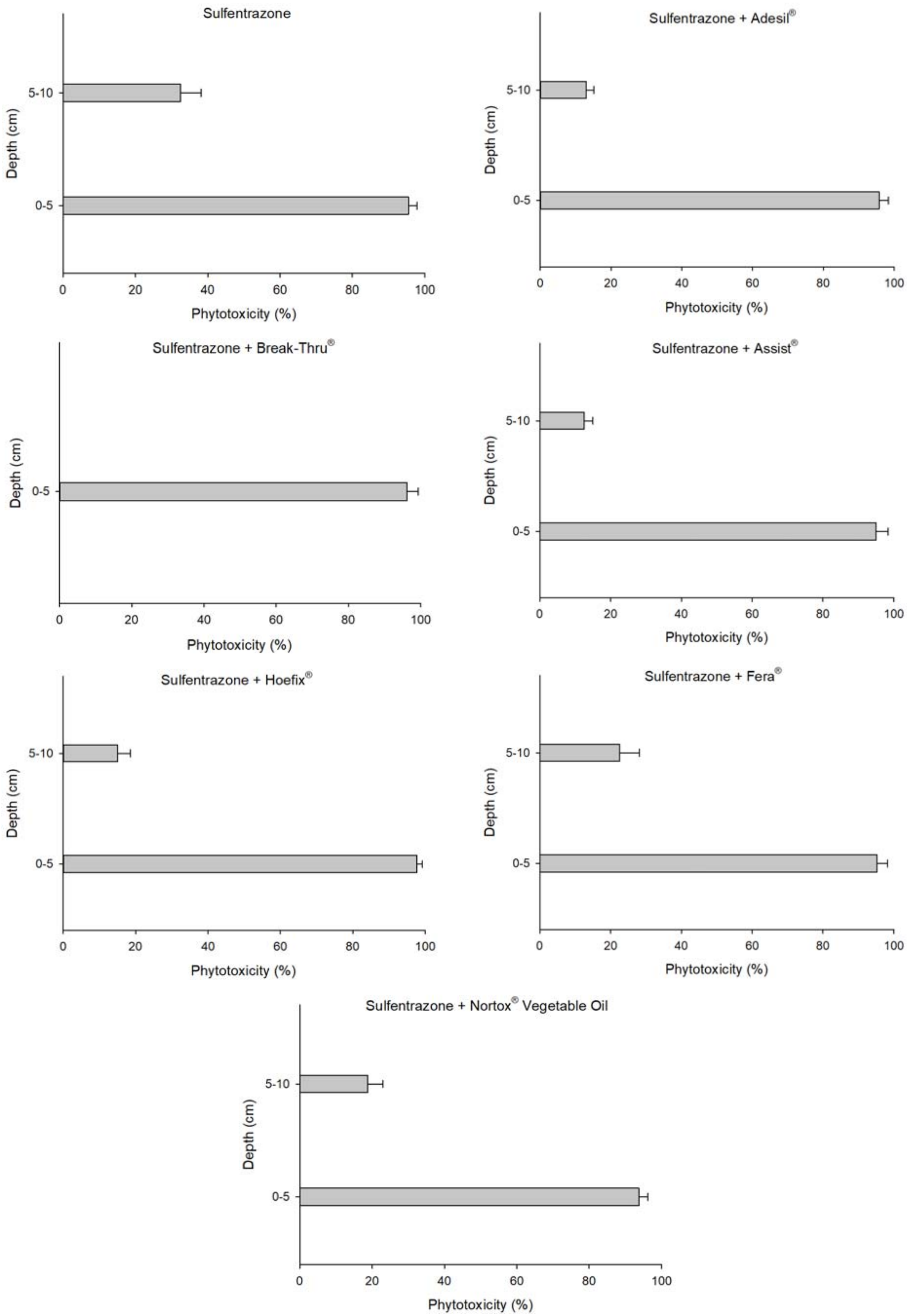

Figure 2 **-** Phytotoxicity in plants of *Sorghum bicolor* grown at different depths at 21 DAE in soil contaminated with sulfentrazone, sulfentrazone + Adesil®, sulfentrazone + Break-Thru®, sulfentrazone + Assist®, sulfentrazone + Hoefix®, sulfentrazone + Fera® and sulfentrazone + Nortox® Vegetable Oil.

have been an increase in the contact surface between the herbicide and the soil, thus reducing leaching. However, this adjuvant did not prevent the herbicide from leaching to the 5-10 cm layer, which was evidenced by intoxication symptoms in the plants.

Persistence of sulfentrazone lasted for 143 days after application. Importantly, this evaluation showed no reduction in shoot dry matter of sorghum in any of the treatments, but there were mild intoxication symptoms in the 0-5 cm layer (around 5% to 7%) in all treatments (Figures 3 and 4), indicating some residual activity of sulfentrazone. At 173 DAA, there was no phytotoxicity or reduction in shoot dry matter of sorghum in any of the treatments (Figure 5).

High persistence of sulfentrazone in this study (143 DAA) serves as a warning that more careful application of this herbicide is required in Brazilian soils. Although a high residual period of herbicides may be advantageous – because, in many cases, only one application is enough to control weeds during the total period of prevention of interference – if herbicides remain available in the soil beyond this period, there may be environmental contamination, especially water contamination, as well increased risk of carryover effects (Krutz et al., 2005; Dan et al., 2010). Ortiz Martinez et al. (2008), while studying sulfentrazone degradation in a Red-Yellow Ultisol, found that its half-life was 146.5 days.

The presence of adjuvants did not influence sulfentrazone persistence in the soil. Kucharski and Sadowski (2011) found that Break-Thru® and mineral oil not only reduced metolachlor leaching ÿþin soils, but also increased the residual period of the herbicide, because these products increased the availability of the herbicide in the environment.

Considering the study soil (Red-Yellow Ultisol), the adjuvant Break-Thru® was effective at reducing leaching of sulfentrazone in the deepest layers, as it was detected only in the 0 5 cm layer.

The adjuvant Assist® resulted in lower leaching of sulfentrazone, with loss of shoot dry matter of the bioindicator plant only in the 0-5 cm layer. However, there were intoxication symptoms in sorghum plants in the 5-10 cm layer, indicating that the adjuvant did not completely reduce herbicide leaching.

The presence of adjuvants applied together with the herbicide did not influence persistence of sulfentrazone in the study soil.

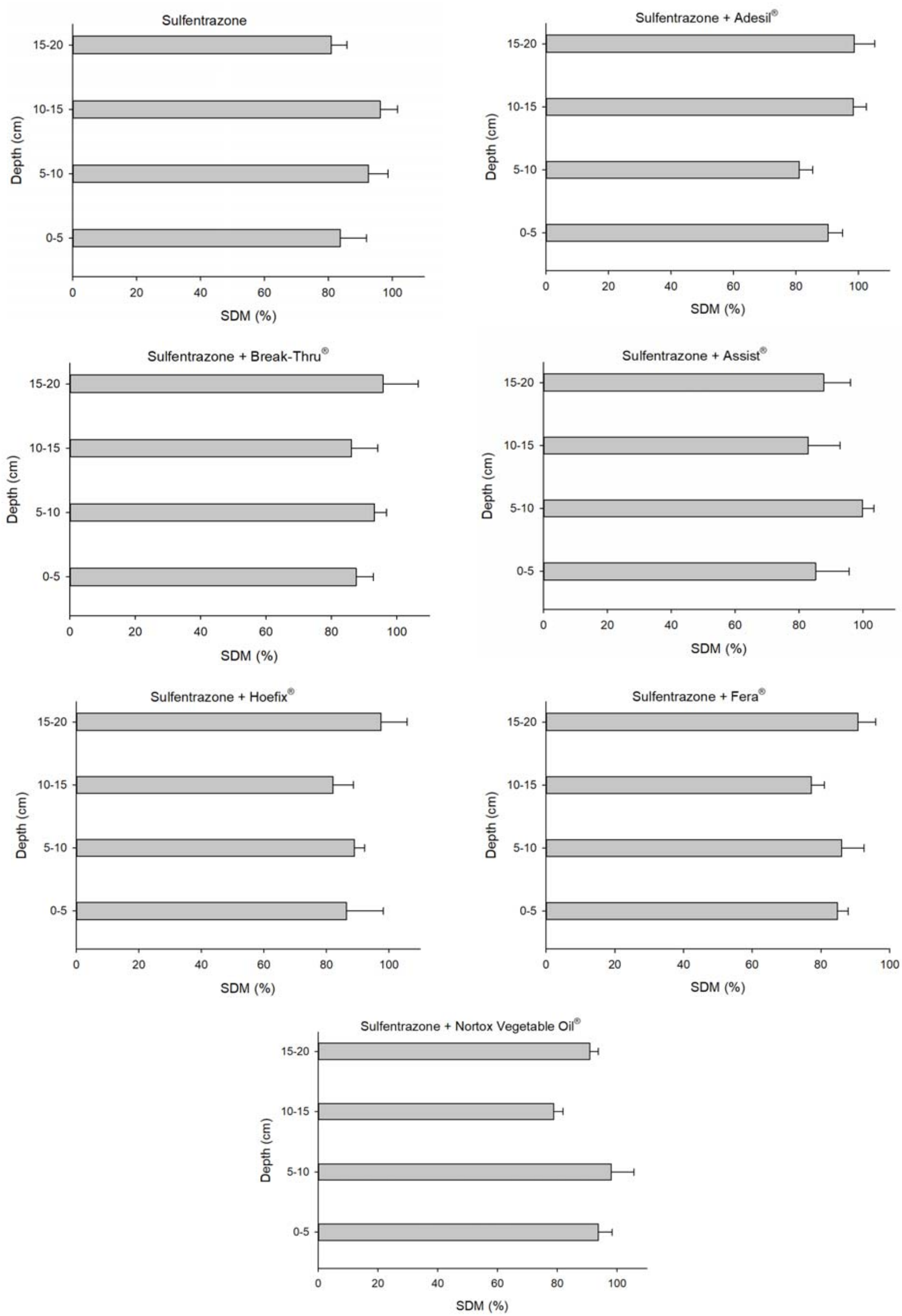

Figure 3 - Shoot dry matter (SDM) percentage of sorghum (*Sorghum bicolor*), in comparison to the control, grown in different depths, at 143 days after application in soil contaminated with sulfentrazone, sulfentrazone + Adesil®, sulfentrazone + Break-Thru®, sulfentrazone + Assist®, sulfentrazone + Hoefix®, sulfentrazone + Fera® and sulfentrazone + Nortox® Vegetable Oil.

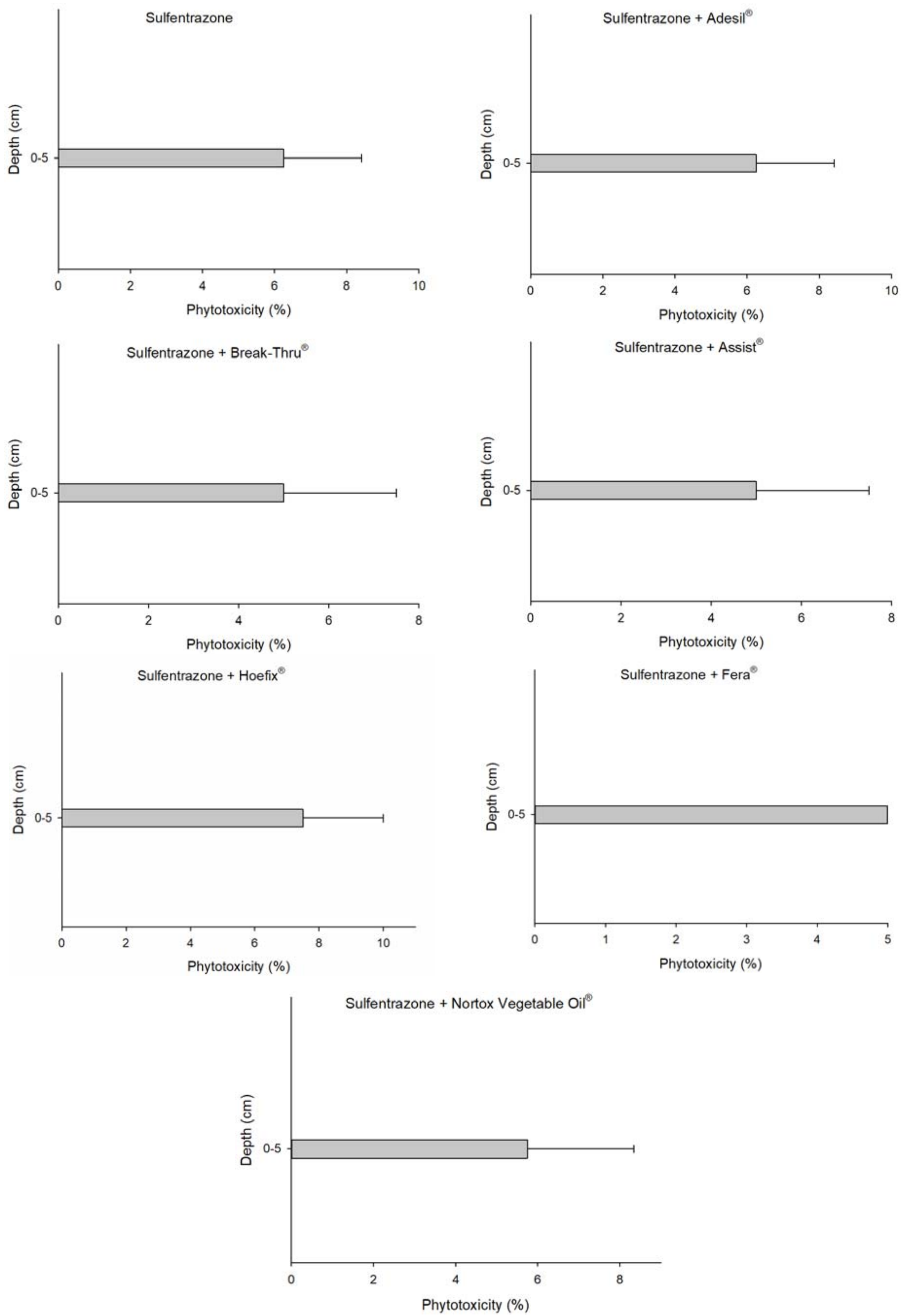

Figure 4 **-** Phytotoxicity in plants of *Sorghum bicolor* grown at different depths, at 143 days after application in soil contaminated with sulfentrazone, sulfentrazone + Adesil®, sulfentrazone + Break-Thru®, sulfentrazone + Assist®, sulfentrazone + Hoefix®, sulfentrazone + Fera® and sulfentrazone + Nortox® Vegetable Oil.

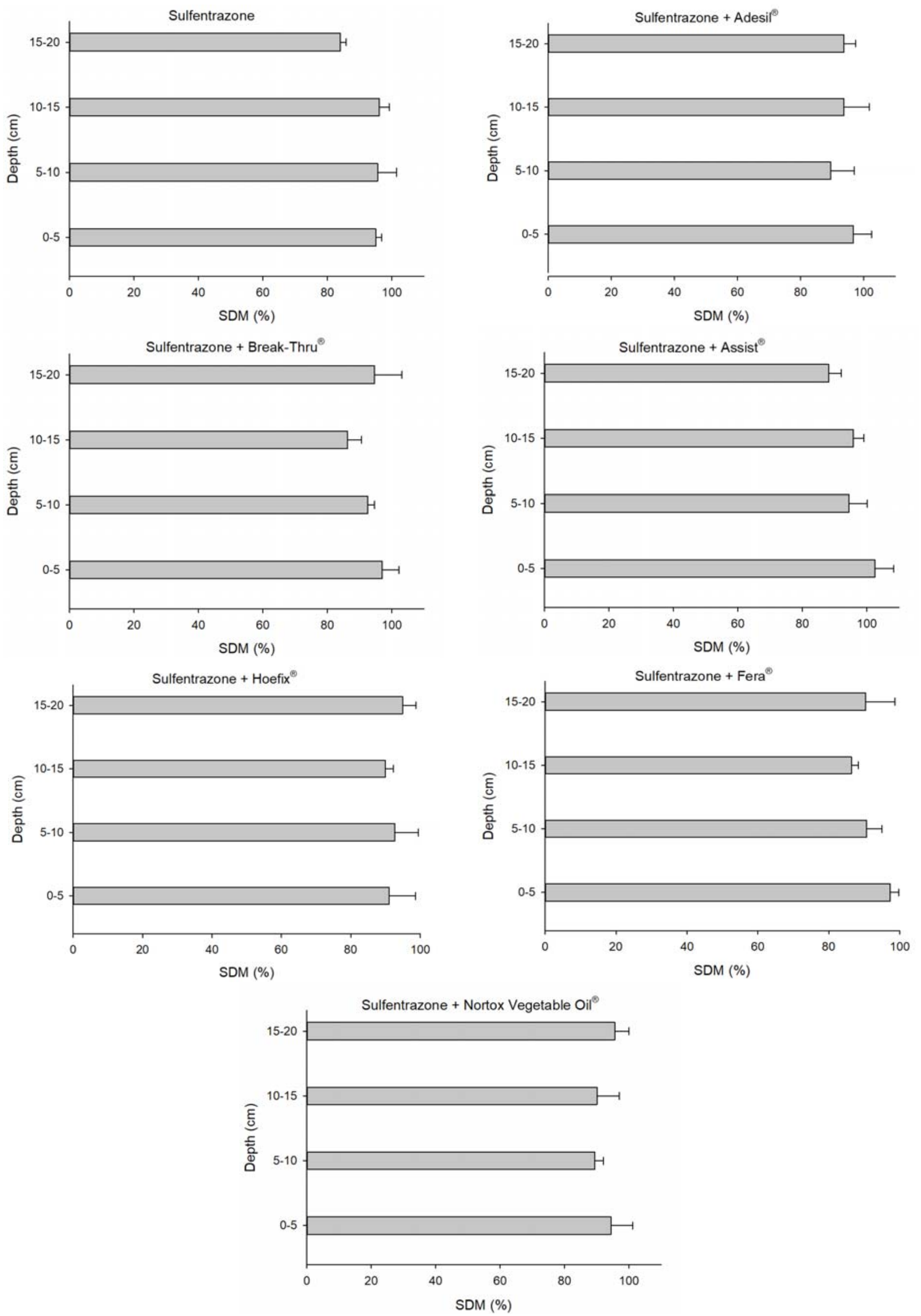

Figure 5 - Shoot dry matter (SDM) percentage of sorghum (*Sorghum bicolor*), in comparison to the control, grown in different depths, at 173 days after application in soil contaminated with sulfentrazone, sulfentrazone + Adesil®, sulfentrazone + Break-Thru®, sulfentrazone + Assist®, sulfentrazone + Hoefix®, sulfentrazone + Fera® and sulfentrazone + Nortox® Vegetable Oil.

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