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Research Article

Hard-water antagonism of trifloxysulfuron as affected by time gap between the addition of adjuvant and herbicide to the tank

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Abstract: This study aimed to assess the effect of a time gap between adding adjuvant and herbicide to the tank on hard-water antagonism of trifloxysulfuron under greenhouse conditions. Treatments consisted of herbicide dose (0, 0.75, 1.5, 3, 6, and 12 g ha^{-1}), spray carrier (distilled water ± 600 mg $MgCl_2$, $CaCl_2$, and $FeCl_3$ L^{-1}), the order of adding adjuvant and herbicide to the spray carrier (herbicide alone, the addition of 20 g ammonium sulfate L^{-1} or 300 mg citric acid L^{-1} 15 min before, at the same time, and 15 min after adding trifloxysulfuron to the spray carrier). The dry matter of velvetleaf was regressed over the doses of trifloxysulfuron to obtain herbicide doses in which 50 and 90% velvetleaf control occur (ED_{50} and ED_{90}, respectively). The presence of Mg^{2+}, Ca^{2+}, and Fe^{3+} in the spray carrier increased the ED_{50} from 2.41 to 5.07, 7.65, and 9.78 g a.i. ha^{-1} and the ED_{90} from 5.88 to 16.56, 28.48, and 28.19 g a.i. ha^{-1}, respectively, indicating a hard-water antagonism of trifloxysulfuron as Fe^{3+} = Ca^{2+} > Mg^{2+}. Generally, the best order was adding adjuvants 15 min before adding trifloxysulfuron to the tank. It could completely overcome the hard-water antagonism of trifloxysulfuron. In contrast, the order of adding adjuvants 15 min after adding trifloxysulfuron to the tank had no significant effect on overcoming the hard-water antagonism of trifloxysulfuron, resulting in additional costs for farmers.

Keywords: ammonium sulfate, citric acid, spray water quality, velvetleaf

Introduction

In agricultural systems, weeds are the most severe concern of farmers. They compete with crops to obtain water, nutrients, and light, causing a significant decrease in crop yields. Many methods have been introduced to control them. However, since the discovery and development of herbicides in the 1940s, chemical control of weeds by herbicides has been an easy, effective, and affordable method (Daramola *et al*., 2022). The efficacy of herbicides against weeds is influenced by several factors: weed growth stage, herbicide formulation, weather conditions before, during, and after herbicide application, and herbicide application technology. In addition, since herbicides are carried by water, water quantity and quality, such as hardness, acidity, turbidity, and temperature, play an influential role in the efficacy of herbicides (Devkota *et al*., 2016a). Usually, farmers use the same water they use for

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irrigation to apply herbicides (Aliverdi *et al*., 2014). The quality of agricultural water is related to the amount of rainfall and the material of the ground bed. These two factors have caused the water quality to face challenges in dry regions of the world, such as Iran. Studies show that the unfavourable rainfall in Iran will intensify in the coming years (Moridi *et al*., 2017). Therefore, using low-quality water (hard and salty) to apply herbicides will be unavoidable.

The antagonism effect of the presence of cations of Na^+, K^+, Ca^{2+}, Mg^{2+}, Mn^{2+}, Zn^{2+}, and Fe^{3+} in hard water on the efficacy of weak acid herbicides that have an acid dissociation coefficient (pKa) < 7 has been reported. For example, glyphosate (Manuchehri *et al*., 2018), glufosinate (Devkota and Johnson, 2016); dicamba (Devkota and Johnson, 2020), 2,4-D (Schortgen and Patton, 2020), clethodim (Nandula *et al*., 2007), sethoxydim (Nalewaja *et al*., 1994), saflufenacil (Roskamp *et al*., 2013a), imazethapyr (Aliverdi *et al*., 2014), tembotrione (Zollinger *et al*., 2016), mesotrione (Devkota *et al*., 2016b), nicosulfuron (Bashashati *et al*., 2022), and sulfosulfuron (Aliverdi *et al*., 2020). The antagonism effect is due to the formation of herbicide-cation salt, which cannot be absorbed through the leaf cuticle, causing a decreased herbicide efficacy. In addition, the deposition of such salts into tanks causes a clogged nozzle or strainer (Daramola *et al*., 2022). Several methods have been suggested to reduce the antagonism effect of the presence of cationic salts in hard water on the efficacy of herbicides, which include reducing spray volume (Nalewaja *et al*., 1994), tank-mixing adjuvants such as citric acid (Travlos *et al*., 2017), ammonium sulfate (Devkota *et al*., 2016a), ammonium thiosulfate (Nurse *et al*., 2008), ammonium nitrate (Aliverdi *et al*., 2014), dipotassium phosphate (Zollinger *et al*., 2016), tetradiamine ethylene acetate (Bernards *et al*., 2005), glucoheptonate, and lignosulfonate (Bailey *et al*., 2002), and magnetizing hard water (Aliverdi *et al*., 2014). Meanwhile, farmers are more willing to use ammonium sulfate due to its availability.

Trifloxysulfuron belongs to group B/2 of herbicides that inhibit amino acid biosynthesis in treated plants, causing their death. It is a weak acid with a pKa value of 4.76 (EPA, 2008) and is used in cotton fields to control broadleaf weeds such as velvetleaf (*Abutilon theophrasti* Medic.). There is no report on the hard-water antagonism of trifloxysulfuron. As a result, this issue was the first objective of the present study. Moreover, in the literature and practice, the herbicide is immediately added to the tank after adding adjuvants. Therefore, there is no report on whether the sequence of the addition of adjuvant and herbicide to the tank containing hard water could affect the efficacy of trifloxysulfuron against velvetleaf. It was the second objective of the present study.

Materials and Methods

Velvetleaf seeds were collected from a field in Gorgan, Iran, in the summer of 2018 and stored in a cool, dry room. In summer 2020, they were hydro-primed in hot water at 60 °C for one hour (Ravlić *et al*., 2015). Ten hydro-primed seeds were planted at a depth of 0.5 cm in each 2-L brown plastic pot containing a clay loam soil with 0.4% organic matter and a pH of 7.2. They were taken out in the open air at Bu-Ali Sina University, Hamedan, Iran. The soil surface of pots was moistened at least once a day until the seedlings started to emerge. Thereafter, the plants were irrigated as required, thinned to five plants pot^{-1} at the one-leaf stage, and fertilized weekly with 30 ml of a solution containing 3 g L^{-1} of an N: P: K (20:20:20) fertilizer. During the growing of plants, outdoor environmental conditions were 25 ± 5 °C air temperature and 30% ± 10% relative humidity.

The experiment layout was a three-factor, completely randomized design (6 × 4 × 7) with four replications. The first factor was the dose of trifloxysulfuron-sodium (Envoke® %75 WG, Syngenta), including 0, 0.75, 1.5, 3, 6, and 12 g a.i. ha^{-1}. The second factor was the type of spray carrier, including distilled water with and without 600 mg $MgCl_2$, $CaCl_2$, and $FeCl_3$ L^{-1} (Penner, 2000). The third factor was the order of the addition of adjuvant and herbicide to the spray carrier, including herbicide alone, the addition of

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20 g ammonium sulfate L^{-1} or 300 mg citric acid L^{-1} 15 min before, at the same time, and 15 min after the addition of trifloxysulfuron to the spray carrier. Based on the herbicide label, a non-ionic surfactant (Trend-90®) at 0.1% v v^{-1} was added to the spray solutions. A portable spray test chamber with a constant nozzle travel speed was used to apply the treatments at 300 kPa spray pressure. It was equipped with Even Flat-Fan 11002. The shoots of plants were cut four weeks after treatment, kept inside an oven at 75 °C for 48 h, and then weighted to obtain the dry matter.

The data were analyzed based on the methodology used by Knezevic *et al*. (2007). Accordingly, a nonlinear regression analysis was conducted using the software R after installing the extension package *'drc'* version 3.5.1, in which the following four-parameter model, was applied to regress the dry matter of velvetleaf, *Y*, over the doses of trifloxysulfuron, *X*. A lack-of-fit test yielding a p-value > 0.05 confirmed that this model acceptably described data.

$$Y = C + \frac{D - C}{1 + \exp(B(\log X) - logE)}$$

The four parameters of the model above are D, C, B, and E, which were estimated using the function *'summary'*. *D* and *C* are the maximum and minimum asymptotes of *Y*, respectively; *E* is where *Y* is halfway between *D* and *C*, donating an effective dose for 50% velvetleaf control (ED_{50}); and *B* is the slope of the fitted nonlinear-regression line around *E*. By replacing the model, the ED_{90} denoting an effective dose for 90% velvetleaf control was obtained. The standard error of EDs was estimated at a 5% significance level and used to evaluate the inferences among the EDs (Ritz *et al*., 2015).

Results

The dose-response curves for velvetleaf dry weight to trifloxysulfuron applied by distilled water with and without Mg^{2+}, Ca^{2+}, and Fe^{3+} is shown in Fig. 1. The presence of Mg^{2+}, Ca^{2+}, and Fe^{3+} in the spray carrier increased the ED_{50} from 2.41 to 5.07, 7.65, and 9.78 g a.i. ha^{-1} and the ED_{90} from 5.88 to 16.56, 28.48, and 28.19 g a.i. ha^{-1}, respectively (Fig. 2), indicating a hard-water antagonism of trifloxysulfuron.

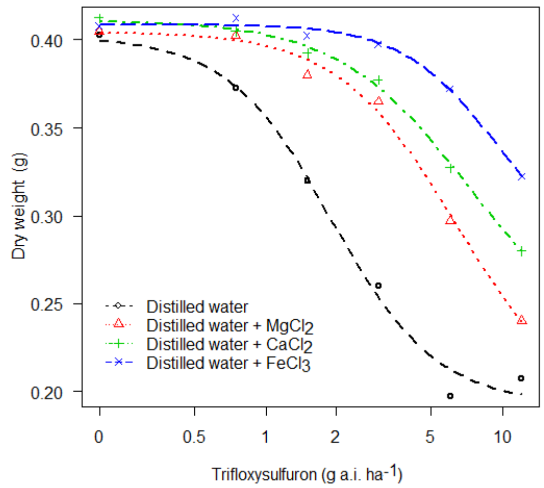

Figure 1 The dose-response curves for velvetleaf dry weight to trifloxysulfuron applied by distilled water with and without Mg^{2+}, Ca^{2+}, and Fe^{3+}.

When trifloxysulfuron was applied with distilled water, its efficacy was not affected by the order of the addition of ammonium sulfate and trifloxysulfuron to the spray carrier (Fig. 2). While it was affected by the order of the addition of ammonium sulfate and trifloxysulfuron to the spray carrier. Based on the ED_{50} values, the addition of citric acid 15 min before, at the same time, and 15 min after the addition of trifloxysulfuron to the spray carrier improved the efficacy of trifloxysulfuron by 2.4, 1.84, and 1.94-fold, respectively.

The results showed that the order of the addition of adjuvant and herbicide to the tank had a significant effect on the efficacy of trifloxysulfuron against velvetleaf (Fig. 2). The ED_{50} values obtained for the treatments of adding ammonium sulfate 15 min before, at the same time, and 15 min after adding trifloxysulfuron to the spray carrier containing Mg^{2+} were $2.67 \leq 3.52 < 4.77$ g a.i. ha^{-1}, respectively. The ED_{50} values obtained for the treatments of adding ammonium sulfate 15 min before, at the same time, and 15 min after adding trifloxysulfuron to the spray carrier containing Ca^{2+} were $2.49 < 3.47 < 8.74$ g a.i. ha^{-1}, respectively.

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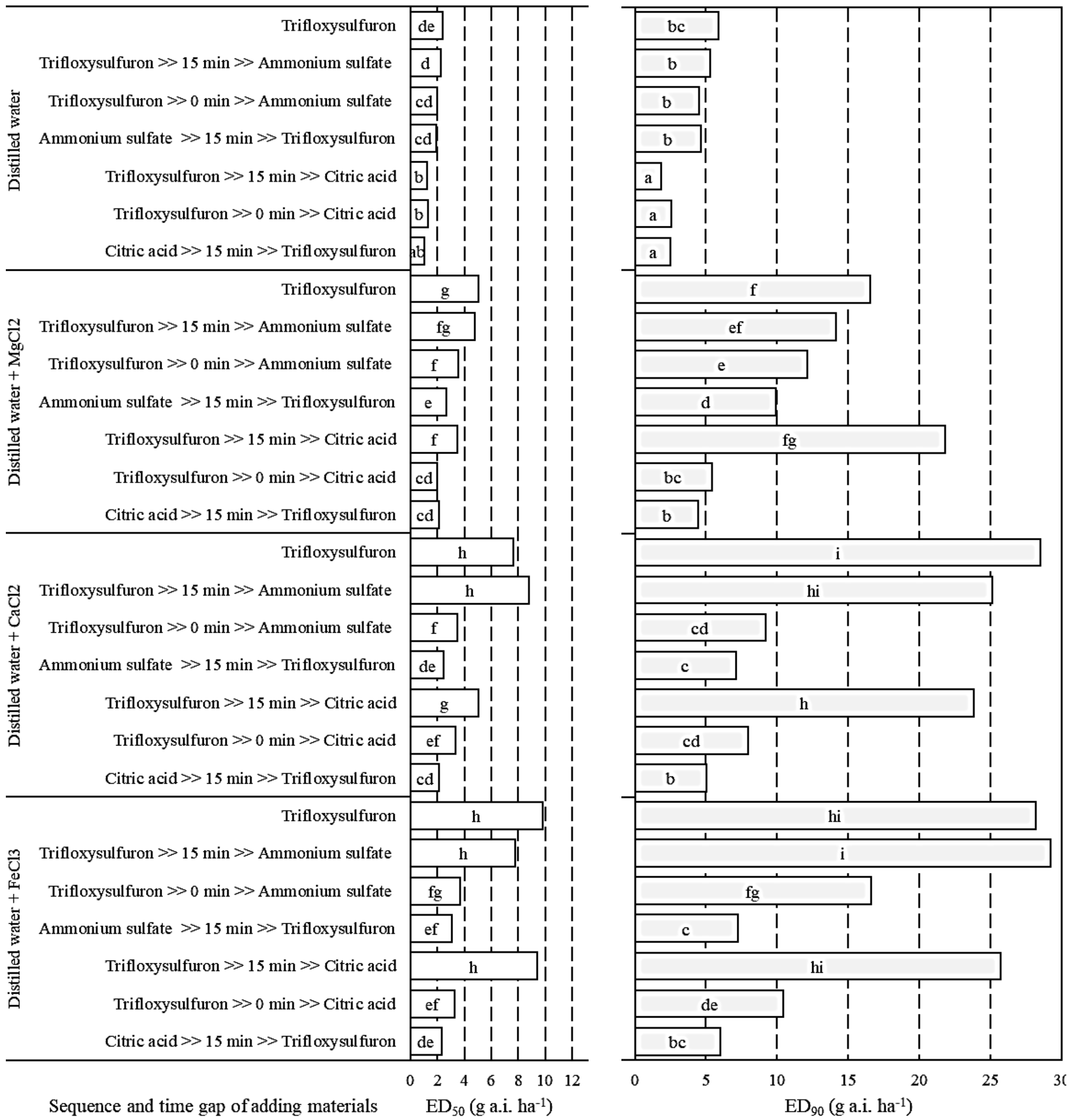

Figure 2 The effect of spray carrier, including distilled water with and without 600 mg $MgCl_2$, $CaCl_2$, and $FeCl_3$ L^{-1}, and the order of the addition of adjuvant and herbicide to the spray carrier, including without adjuvant, the addition of 20 g ammonium sulfate L^{-1} or 300 mg citric acid L^{-1} in 15 min before, at the same time, and 15 min after the addition of trifloxysulfuron to the spray carrier, on the dose of trifloxysulfuron (g a.i. ha^{-1}) required for 50 and 90% reduction in velvetleaf dry weight (ED_{50} and ED_{90}, respectively). In each ED, the values followed by the same letter are not different.

The ED_{50} values obtained for the treatments of adding ammonium sulfate 15 min before, at the same time, and 15 min after adding trifloxysulfuron to the spray carrier containing Fe^{3+} were $3.07 < 3.65 \leq 7.77$ g a.i. ha^{-1}, respectively. The ED_{50} values obtained for the treatments of adding citric acid 15 min before, at the same time, and 15 min after adding

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trifloxysulfuron to the spray carrier containing Mg^{2+} were 2.13 ≤ 1.99 < 3.45 g a.i. ha^{-1}, respectively. The ED_{50} values obtained for the treatments of adding citric acid 15 min before, at the same time, and 15 min after adding trifloxysulfuron to the spray carrier containing Ca^{2+} were 2.09 < 3.32 < 5.02 g a.i. ha^{-1}, respectively. The ED_{50} values obtained for the treatments of adding citric acid 15 min before, at the same time, and 15 min after adding trifloxysulfuron to the spray carrier containing Fe^{3+} were 2.29 ≤ 3.26 ≤ 9.38 g a.i. ha^{-1}, respectively. In general, the comparison of the values above with the ED_{50} value of control (trifloxysulfuron alone with distilled water; ED_{50} = 2.41 g a.i. ha^{-1}) shows that adding adjuvants 15 min after adding trifloxysulfuron to hard water has no significant effect to overcome the antagonism effect of Ca^{2+}, Mg^{2+}, and Fe^{3+} on the efficacy of trifloxysulfuron, except for adding citric acid 15 min after adding trifloxysulfuron to the spray carrier containing Fe^{3+}. When trifloxysulfuron is added to the hard water before ammonium sulfate or citric acid, the complex of herbicide-cation forms. Therefore, trifloxysulfuron loses its possibility to receive H^+ from citric acid or NH_4^+ from ammonium sulfate. For this reason, this sequence of adding chemicals to hard water is ineffective. In contrast, the comparison of the values above with the ED_{50} value of the control shows that adding adjuvants 15 min before adding trifloxysulfuron to hard water could completely overcome the antagonistic effect of Ca^{2+}, Mg^{2+}, and Fe^{3+} on the efficacy of trifloxysulfuron.

Discussion

The antagonistic effect of the cations could be ranked as follows: $Fe^{3+} = Ca^{2+} > Mg^{2+}$. Similarly, Daramola *et al*. (2022) reported that trivalent cations are more problematic than monovalent and bivalent cations. As mentioned above, many researchers reported the antagonistic effect of the cations on the efficacy of weak acid herbicides. As a weak acid herbicide, trifloxysulfuron is ionized into anion (with a negative charge) and cation (H^+) under alkaline conditions. The anion of trifloxysulfuron can bind with the cations in hard water. As a result, the complex of trifloxysulfuron-cation is formed, which cannot be absorbed through the leaf cuticle due to its low solubility in water, causing a reduced herbicide efficacy. Moreover, Hoffman *et al*. (2008) reported that the presence of Ca^{2+} in the spray carrier increases the size of the spray droplets. As a result, the retention of spray droplets on the leaf is reduced. They considered this phenomenon as the second reason for the hard-water antagonism of herbicides.

Many researchers have tried to understand the mechanism of improved efficacy of weak acid herbicides such as 2,4-D (Devkota and Johnson, 2019), chlorsulfuron (Fahl *et al*., 1995), glyphosate (Molin and Hirase, 2004), clopyralid, piclorem (Palma *et al*., 2015), bentazon (Liu, 2004), and nicosulfuron (Green and Cahill, 2003) by decreasing the pH of the spray solution. A decrease in pH makes a weak acid herbicide more permeable to pass through the cuticle - a process known as ion trapping. Under alkaline conditions, such a molecule is negatively charged and more hydrophilic. Under acidic conditions, it receives an H+ and becomes a molecule with no charge, which is more hydrophobic (Sterling, 1994). The water solubility of trifloxysulfuron has been measured up to 63 mg L^{-1} at pH = 5.06, 5016 mg L^{-1} at pH = 7.04, and 38 g L^{-1} at pH = 7.85 at 20 °C (EPA, 2008). By lowering the pH of the spray carrier by adding citric acid, trifloxysulfuron molecules become electrically uncharged (hydrophobic). Therefore, they can pass through the cuticle and cell membrane more easily. Inside the cell, where H^+ is relatively scarce due to its pumping out of the cell, the electrically uncharged trifloxysulfuron loses an H^+, and again, electrically charged trifloxysulfuron molecules (hydrophilic) are formed and are no longer able to leave the cell. In this way, a weak acid herbicide is trapped inside the cell or the phloem and transported efficiently (Sterling, 1994). Many studies have shown that adding ammonium sulfate to the spray solution improves the efficacy of weak acid herbicides (Izadi-Darbandi *et al*., 2019; Nurse *et al*., 2008; Zollinger *et al*., 2016;

Asif *et al*., 2019; Roskamp *et al*., 2013b; Aliverdi *et al*., 2020; Bashashati *et al*., 2022). By receiving NH_4^+ from ammonium sulfate, these herbicides become more hydrophobic, and their permeability is greater to pass through the cuticle and then the cell membrane. After passing, the NH_4^+ is separated from the herbicide molecule. Again, electrically charged herbicide molecules (hydrophilic) are formed, which can no longer leave the cell, resulting in increased herbicide efficacy. Nevertheless, it has been reported the addition of ammonium sulfate to the spray solution of some weak acid herbicides such as imazamethabenz is ineffective (Hsiao *et al*., 1996) and even flumetsulam has a negative effect (El-Metwally *et al*., 2010) on their efficiency.

When ammonium sulfate or citric acid is added to the hard water before trifloxysulfuron, the cations can receive H^+ from citric acid or SO_4^{2-} from ammonium sulfate. As a result, the hard water becomes soft water, and hard-water antagonism of trifloxysulfuron does not occur. In a previous study, Ramos *et al*. (2019) evaluated the effect of the sequence of adding surfactant and herbicide to soft water (not hard water) and reported that adding surfactant before adding herbicides (aminopyralid and fluroxypyr) caused better control of *Senna obtusifolia* than the opposite sequence.

The current study showed a hard-water antagonism of trifloxysulfuron, in which the antagonism effect of the cations ranked as follows: $Fe^{3+} = Ca^{2+} > Mg^{2+}$. For overcoming this antagonistic effect, the addition of ammonium sulfate or citric acid can be a way forward, but only by following this basic issue that they must be added to the tank at least 15 min before trifloxysulfuron. If done in reverse, no significant effect will be seen. It means weeds will not be controlled well, resulting in additional costs (herbicide + adjuvant + application) for farmers.

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کاهندگی آب‌سخت بر تری‌فلوکسی‌سولفورون تحت تأثیر فاصله زمانی بین افزودن مواد افزودنی و علف‌کش به مخزن

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چکیده: این مطالعه با هدف بررسی اثر فاصله زمانی بین افزودن ماده افزودنی و علف‌کش به مخزن بر کاهندگی آب‌سخت بر تری‌فلوکسی‌سولفورون در شرایط گلخانه انجام شد. تیمارها شامل دُز علف‌کش (۰، ۰/۷۵، ۱/۵، ۳، ۶ و ۱۲ ماده مؤثره گرم در هکتار)، حامل پاشش (آب مقطر با و بدون ۶۰۰ میلی‌گرم $MgCl_2$، $CaCl_2$ و $FeCl3$) و ترتیب افزودن ماده افزودنی و علف‌کش به حامل پاشش (علف‌کش به‌تنهایی، افزودن ۲۰ گرم سولفات آمونیوم یا ۳۰۰ میلی‌گرم اسید سیتریک در لیتر ۱۵ دقیقه قبل، هم‌زمان و ۱۵ دقیقه پس از افزودن تری‌فلوکسی‌سولفورون به حامل پاشش) بود. وزن خشک گاوپنبه روی دُزهای تری‌فلوکسی‌سولفورون برازش داده شد تا دُزهایی که در آن ۵۰ و ۹۰ درصد کنترل گاوپنبه رخ می‌دهد (به‌ترتیب ED_{50} و ED_{90}) برآورد شود. حضور Mg^{2+}،Ca^{2+} و Fe^{3+} در حامل پاشش به‌ترتیب سبب افزایش ED_{50} از ۲/۴۱ به ۵/۰۷، ۷/۶۵ و ۹/۷۸ و ED_{90} از ۵/۸۸ به ۱۶/۵۶، ۲۸/۴۸ و ۲۸/۱۹ گرم ماده مؤثره در هکتار شد که نشان‌دهنده کاهندگی آب‌سخت بر تری‌فلوکسی‌سولفورون به‌صورت $Fe^{3+} = Ca^{2+} > Mg^{2+}$ است. به‌طور کلی، بهترین ترتیب اضافه کردن مواد افزودنی ۱۵ دقیقه قبل از افزودن تری‌فلوکسی‌سولفورون به مخزن بود. این تیمار توانست کاهندگی آب سخت بر تری‌فلوکسی‌سولفورون را کاملاً رفع کند. در مقابل، ترتیب افزودن مواد افزودنی ۱۵ دقیقه پس از افزودن تری‌فلوکسی‌سولفورون به مخزن تأثیر معنی‌داری در رفع کاهندگی آب سخت بر تری‌فلوکسی‌سولفورون نداشت و در نتیجه می‌تواند هزینه‌های اضافی را متوجه کشاورزان سازد.

واژگان کلیدی: سولفات آمونیوم، اسید سیتریک، کیفیت آب سمپاشی، گاوپنبه

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