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Surfactant and rainfall influenced clodinafop-propargyl efficacy to control wild oat (*Avena ludoviciana* Durieu.)

Hossein Hammami, Mohammad Hassan Rashed Mohassel and Akbar Aliverdi *

Department of Agronomy, College of Agriculture, Ferdowsi University of Mashhad, Mashhad, Iran

*** Corresponding author: alahbareruca@gmail.com**

Abstract

A nonionic (Citogate) and cationic (Frigate) surfactant were evaluated for their efficacy to enhance clodinafop-propargyl performance and minimize rainfall effect in controlling wild oat (*Avena ludoviciana* Durieu.). Moreover, by using the capillary rise technique the surface tension of these surfactants or surfactants plus clodinafop-propargyl aqueous solution was determined. Lower and higher surface tension values were recorded with aqueous solution of Citogate and Frigate alone and along with clodinafop-propargyl, respectively. The critical micelle concentration of Citogate (0.15% *v/v*) was higher than of Frigate (0.1% *v/v*)**.** Both the tested surfactants minimized the rainfall effect and improved the performance of clodinafop-propargyl on wild oat. When Citogate was added to clodinafop-propargyl, herbicidal activity was higher than when Frigate was added, indicating that the surfactants potency to reduce surface tension of spray solution is a momentous factor in order to enhance clodinafop-propargyl performance. The data from rainfall treatment have confirmed this hypothesis, as it seems when Citogate was added to clodinafop-propargyl; rainfall adverse effect was lower which is presumably due to quick absorption of clodinafop-propargyl by wild oat leaves. In other words, clodinafop-propargyl infiltrated in short-term before it was washed off the wild oat leaf surface as a hypothesis.

Keywords: *Avena ludoviciana* Durieu.; ethylene oxide; rainfall; surface tension; surfactant.
Abbreviations: ED-effective dose; HLB-hydrophilic lipophilic balance; RP-relative potency.

Introduction

Clodinafop-propargyl is an herbicide that inhibits the enzyme acetyl coenzyme-A carboxylase and disrupts fatty acid biosynthesis in grasses such as wild oat (*Avena ludoviciana* Durieu.). In winter-based cropping systems, wild oat is economically the most important weed in Iran (Bijanzadeh et al., 2010). It reduces winter wheat yield by over 30% (Kazemi and Shimi, 2005). Due to its high effectiveness, clodinafop-propargyl at 64 g ai ha^{-1} is registered to control different species of wild oat in wheat and barley (Baghestani et al., 2008). Therefore, the usage of clodinafop-propargyl in Iran was increased from 30.6 tons to 1107.5 tons year^{-1} during 1994-2006 (Deihimfard et al., 2007) which could be qualified as a high-risk herbicide. Therefore, from the environmental and the economical point of view, optimizing the dosage of clodinafop-propargyl is important to reduce its possible side effects (Kudsk, 2008) and application costs. Previous studies have shown that the spray droplets slightly retained on the leaf surface and the majority of those droplets bounce off the leaf surface (Penner, 2000; Young and Hart, 1998). Therefore, improvement in the retention capacity of droplets could be an approach to increase the herbicide performance and to reduce its dosage (Kudsk, 2008). Water is the primary carrier for herbicide applications. It has been shown that the surface tension value of water is high (~73 mN m^{-1}) and it was slightly varied when an agrochemical was added (Sharma and Singh, 2000; Gauvrit and Lamrani, 2008). During the spraying process, the factors that determine the fate of the spray droplets included droplet size, impact velocity, leaf surface micro-morphology, and the dynamic surface tension of the spray solution (Sharma and Singh, 2000). In high surface tension, the spray droplets will bounce off and improper leaf coverage might occur (Hazen, 2000). It happens, particularly, on upright leaves of grasses such as wild oat (Penner, 2000). Previous study has shown that the spray droplets bouncing were diminished by decreasing the surface tension of droplets. This process led to reducing the contact angle and more spread of droplets on the leaf surface (Rashed-Mohassel et al., 2009). The latter results a more penetration of the active ingredient into the leaf (Sharma and Singh, 2000). Indeed, surfactants are an acceptable compound to enhance the penetrability of herbicide active ingredient in order to reduce herbicide dosage for controlling a weed (Penner, 2000). Moreover, the decrease in surface tension of spray droplets can indirectly overcome adverse application conditions such as rainfall (Smith et al., 1999; Kudsk, 2008). Rainfastness was defined as the time required after herbicide usage for adequate absorption to occur so that, herbicide activity not to be diminished by rainfall (Molin and Hirase, 2005). Although previous studies have shown that surfactants increased the performance (Sharma and Singh, 2000; Rashed-Mohassel et al., 2009) and decrease rainfastness of herbicides (Molin and Hirase, 2005; Young and Hart, 1998; Bariuan et al., 1999; Smith et al., 1999), an important question still arise whether each kind of surfactant will be compatible to increase efficacy of each kind of herbicide or not. Therefore, the objective of this study was assessing of two types of surfactants in (*i*) improving clodinafop-propargyl performance against wild oat and (*ii*) minimizing rainfall adverse effect against clodinafop-propargyl performance.

Materials and methods

Surface Tension Studies

The static surface tension of a range of concentrations 0.01, 0.05, 0.1, 0.15, 0.2 and 0.3% (*v/v*) of aqueous surfactant solution alone and formulated with clodinafop-propargyl were determined using the capillary rise technique (Vanhanen et al., 2008) with four replicates for each value at 25±1º C:

$$\text{(Eq.1)} \qquad \gamma = \frac{1}{2}\rho . g . r(h + \frac{r}{3})$$

Where r is the inner radius of the glass capillary tube (mm), g is the acceleration due to gravity which is 9.8 m sec^{-2}, ρ is density of solution (kg m^{-3}), h is the capillary rise (m) and γ is the surface tension (N m^{-1}) which was transformed to mN m^{-1} for analyzing. Of each solution, 50 ml was weighed to calculate density of solution by a laboratory scale:

$$\text{(Eq.2)} \qquad \rho = m / v$$

Where m is scale of solution (g) and v is volume of solution (g cm^{-3}). Then, the data was transformed to kg m^{-3} for placing into equation 1. The commercial clodinafop-propargyl was prepared at concentration corresponding to 64 g ai ha^{-1} applied at 200 L distilled water.

Plant Growth

Wild oat seeds were collected from plants from the field near Greenhouse of College of Agriculture, Ferdowsi University of Mashhad, Iran. After manual husking, seeds placed in 11 cm diameter glass petri dishes lined with a single layer of wathman no.1 filter paper. Ten ml of KNO_3 solution (2g L^{-1}) was added to each petri dish. Petri dishes were placed in a refrigerator at 4-5°C in the dark for two days and then transferred to an incubator with 20/10°C temperature in 45/65% relative humidity for a 16/8 h day/night for germination. After germination, the seedlings were sown in 2-L plastic pots filled with a silt loam soil (19.8% sand, 20.1% clay, 58% silt, 4.1% organic matter and a pH of 6.7) at 1cm depth. Pots were sub-irrigated every three days with tap water. At one leaf stage, the seedlings were thinned from approximately ten to five per pot and fertilized once with 30 ml of N:P:K (20:20:20) solution fertilizer at concentration of 3g of fertilizer L^{-1} of tap water.

Treatments

Wild oat plants were sprayed with herbicide at the four-leaf stage. Pots were distributed in a factorial arrangement based on completely randomized design with four replications. The bio-efficacy studies were repeated twice and gave a similar result. Hence, the results of one of the two will be reported. The experimental treatments were clodinafop-propargyl doses in six levels (0, 8, 16, 32, 48, and 64 g ai ha^{-1} of Topik$^{®}$ 8% EC, 80 g ai L^{-1} clodinafop-propargyl, Syngenta, Switzerland), two types of surfactant (Citogate (a nonionic surfactant, 100% alkylarylpolyglycol ether, Zarnegaran Pars Co., Iran) and Frigate (a cationic surfactant, 81.2% mixture of tallow amines ethoxylated long-chain fatty, ISK Biosciences Crop, Mentor, England)) and rainfall in two levels (with and without rainfall). Both surfactants were added at 0.2% (*v/v*). Treatments were applied using an overhead trolley sprayer equipped by 8002 flat fan nozzle tip delivering 200 L ha^{-1} at 200 kPa. For simulating rainfall treatment, 30 min after spraying, the plants were subjected using a rain simulator operated at an intensity of 50 mm for a 10 min period. Four weeks after spraying, the control and treated plants above-ground biomass from each pot (all of the plants in each pot) were harvested and weighed (fresh weigh), then, oven dried at 75°C for 48 h and reweighed (dry weigh).

Statistical Analyses

Since, fresh weight and dry weight data showed similar trend, only ED_{50} and ED_{95} doses based on dry weight date were included. Weight data of all the herbicide treatments (different surfactants and rain treatments) were subjected to non-linear regression analyses by using a logistic dose-response model (Kudsk and Mathiassen, 2004):

$$\text{(Eq.3)} \qquad U = C + \frac{D - C}{1 + \exp[b(\log(z) - \log(ED_{50}))]}$$

where, U_i is the plant response to the i^{th} herbicide treatment, z is the dose, D and C are the upper and lower limits of the curve respectively. ED_{50} denotes the required dose of herbicide to give 50% wild oat control and b is proportional to the slope of the curve around the ED_{50}. In equation 3, The ED_{50}-parameter can be replaced by any ED level, e.g. ED_{95}, which denotes the required dose of herbicide to give 95% weed control. The logistic response-dose model was fitted to the experimental data by the *Slide Write* software and then based on available information (Kudsk and Mathiassen, 2004); the relative potency (*RP*) value was used to describe horizontal displacement between the two response-dose curves:

$$\text{(Eq.4)} \qquad RP = ED_{50\text{clodinafop-propargyl commercial formulation}} \Big/ ED_{50x}$$

where, ED_{50x} is the ED_{50} of clodinafop-propargyl commercial formulation alone or in combination with (*i*) Citogate, (*ii*) Frigate, (*iii*) rainfall, (*iv*) Citogate and rainfall, (*v*) Frigate and rainfall. If the *RP*-value was equal 1, the addition of surfactant and the application of rainfall would not have any effect on herbicidal responses. While if the *RP*-value was bigger or smaller than 1, herbicide usage with treatments would be more or less potent than herbicide alone.

Results and Discussion

Surface Tension Studies

The distillated water surface tension was recorded 72.24 mN m^{-1} (Fig. 1a). All water–surfactant solutions had a significant lower surface tension than the corresponding values of water alone ($P < 0.01$). Citogate was more effective surfactant for reducing surface tension than Frigate. For Citogate, reduction of the surface tension was 53.4%, and for Frigate, it was 45.2% at 0.3% (*v/v*) (Fig. 1a). This finding was in agreement with the results of Aliverdi et al. (2009) who reported that the tested adjuvants decreased surface tension of clodinafop propargyl and tribenuron methyl spray solutions. High surface tension reducer potency of Citogate might be theoretically related to its chemical structure. The lower number of ethylene oxide units (CH_2CH_2O-) of Citogate (7) than Frigate (15) may resulted in such surface tension reduction (Hazen, 2000). The previous studies reported that the surface tension of water decreased with a decrease in ethylene oxide number (Sharma et al., 1996; Myers, 2006)

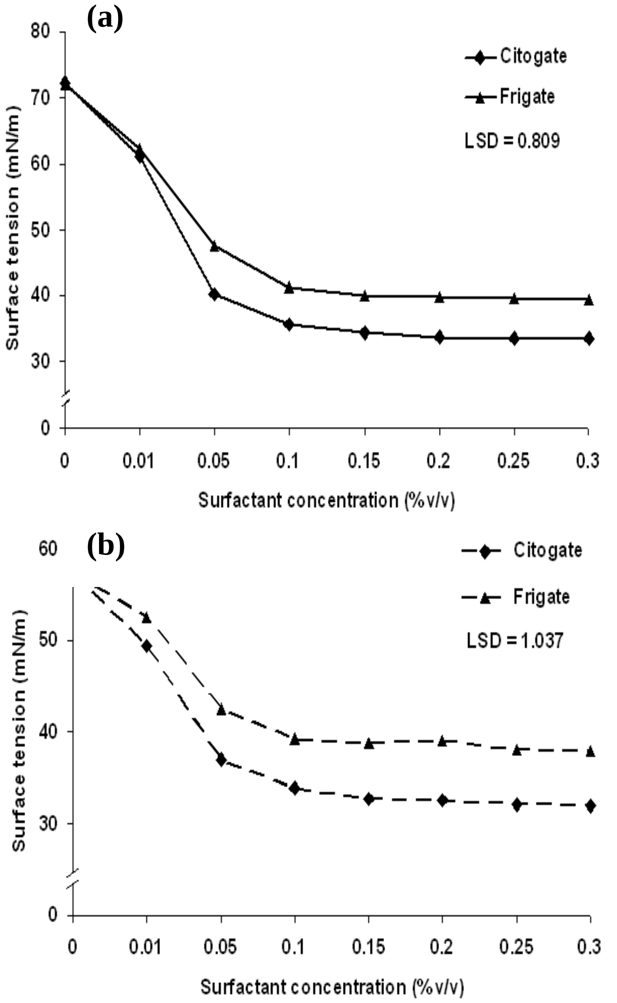

Fig 1. Influence of surfactant different concentrations on surface tension of distilled water (a) and clodinafop-propargyl solution (b) which was prepared at concentration corresponding to 64 g ai ha^{-1} applying at 200 L distilled water.

because each supernumerary ethylene oxide unit added to surfactant reduces the packing density of hydrophobic groups at water-surfactant interface which results in a less reduction in the surface tension of the solution (Myers, 2006). These findings were in agreement with the results of Stevens and Bukovac (1987) that high ethylene oxide surfactants have higher surface tension and contact angles than low ethylene oxide surfactants. Increasing concentrations of the surfactants up to 0.1% (*v/v*) for Frigate and 0.15% (*v/v*) for Citogate was resulted in a rapid and significant decrease in water surface tension value and thereafter there was no significant change. The concentrations of 0.15% for Citogate and 0.1% for Frigate are considered as critical micelle concentration. Based on Myers (2006), the critical micelle concentration is the concentration at which a surfactant forms micelles. Sun et al. (1996) also reported that the relationship of surfactant concentration and surface tension is theoretically a logistic distribution and reported that minimum surface tension occurred at concentrations of 0.1 to 0.5% (v/v) for the surfactants tested. Both the tested surfactants significantly decreased surface tension of clodinafop-propargyl solution ($P<0.01$) (Fig. 1b). Both the clodinafop-propargyl + surfactant solutions had significantly lower surface tension than the corresponding values of clodinafop-propargyl alone. Clodinafop-propargyl solution had a static surface tension of 57.39 mN m^{-1}, which was significantly less than the checked water (72.24 mN m^{-1}). Citogate was more effective to reduce the surface tension of clodinafop-propargyl solution than Frigate. Therefore, the reduction percent of the surface tension of clodinafop-propargyl solution was recorded 44.5% with Citogate and 33.8% with Frigate at 0.3% (*v/v*), respectively. The results indicated that with increasing the concentration of Frigate and Citogate up to 0.1% and 0.15% (*v/v*), respectively, a significant decrease in surface tension of clodinafop-propargyl solution was perceived and afterwards followed non-significant steady state. This finding was in agreement with the theory of Sun et al. (1996) that have shown above.

Bio-Efficacy Studies

The results from this study indicated that when clodinafop-propargyl was combined with each surfactant, wild oat control was remarkably increased. The ED_{50}- and ED_{95}-values of clodinafop-propargyl were remarkably decreased (Table 1) and the *RP*-value was considerably increased when both the surfactants were added to clodinafop-propargyl (Fig. 2). These results indicate an increase in the performance of clodinafop-propargyl. Therefore, the performance of 1 kg ha^{-1} clodinafop-propargyl, in the presence of Frigate and Citogate, was equals to 1.49 and 1.70 kg ha^{-1} clodinafop-propargyl alone, respectively (Fig. 2). Previous research findings showed that if surface tension reduces, the liquid sheet can more easily expand and lead to production of smaller droplets at atomization (Penner, 2000). This action will increase retention because of the lower impacting energy of the smaller droplets (Young and Hart, 1998). Tested surfactants decreased the surface tension value and improved efficacy of clodinafop-propargyl solution by bringing more active ingredient in contact with wild oat leaves. The results indicated that Citogate had superiority in enhancing the foliage activity of clodinafop-propargyl to Frigate (Table 1 and Fig. 2). These results can be attributed to chemical structure and HLB of the tested surfactants. As have shown above, in hydrophilic part, Frigate has ethylene oxide approximately twice as many as Citogate does. Previous studies have shown that absorption of hydrophobic herbicides (log *K*ow > 1) is generally increased by the addition of surfactants with low ethylene oxide content. In contrast, absorption of hydrophilic herbicides (log *K*ow < 1) is best with surfactants which own a higher content from ethylene oxide (Ramsey et al., 2005). Therefore, Citogate with low ethylene oxide content increased the performance of clodinafop-propargyl (log *K*ow = 3.9) higher than Frigate with high ethylene oxide content. Therefore, our results support the findings of Green (1999), who showed that increasing the number of ethylene oxide units makes the surfactant more hydrophilic and increase its HLB. In surface tension studies, we have also discovered that Citogate and Frigate have a lower (~8) and higher (>13) HLB, respectively. HLB can be estimated by observing solubility a surfactant in water with no dispersion = 1 to complete dispersion = 20 (Green, 1999). When Citogate and Frigate were added to distilled water gave a turbid and clear solution, respectively. In other words, Frigate is completely soluble in water. It can be attributed to its chemical structure. Myers (2006) stated that solubility of surfactant in water increases regularly as the number of ethylene oxide contents is increased from 3 to 16. Previous studies have shown that high HLB surfactants work best with hydrophilic herbicides (log *K*ow < 1), while, low HLB surfactants work best with

Table 1. The *ED*$_{50}$ and *ED*$_{95}$ (g ai ha^{-1}) of clodinafop-propargyl alone and in mixture with surfactants subjected to rainfall treatment on *Avena ludoviciana* Durieu.

Treatment	No Surfactant	Frigate	Citogate
		ED$_{50}$ (g ai ha^{-1}) ± SE	
Clodinafop	19.09 ± 1.21	12.84 ± 1.19	11.36 ± 0.76
Clodinafop + Rain	29.15 ± 0.98	24.79 ± 1.65	23.07 ± 1.03
		ED$_{95}$ (g ai ha^{-1}) ± SE	
Clodinafop	25.42 ± 1.54	31.64 ± 1.87	22.75 ± 1.09
Clodinafop + Rain	> 64	28.67 ± 1.10	34.11 ± 1.12

SE is standard errors ($P < 0.05$). Clodinafop-propargyl is shortened to clodinafop. Rainfall was exerted 30 min after treatment at intensity of 5 cm h^{-1} for a 10-min period.

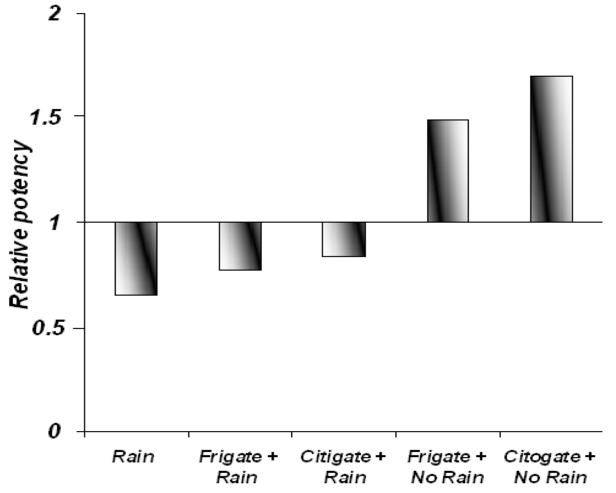

Fig 2. Relative potency values of clodinafop-propargyl plus Frigate and Citogate with and without rainfall on wild oat (*Avena ludoviciana* Durieu.). The *ED*$_{50}$-value of clodinafop-propargyl alone was specified 19.09 g ai ha^{-1}. Relative potency > 1 or < 1 indicate an increase or decrease in clodinafop-propargyl efficacy, respectively.

hydrophobic herbicides (log *K*ow > 1) (Green, 1999; Zimdahl, 2007). Whereas, log *K*ow for clodinafop-propargyl is 3.9, therefore, Citogate with low HLB increased the performance of clodinafop-propargyl higher than Frigate with high HLB. Nalewaja and Matysiak (1993), Nalewaja et al. (1996) and Green (1999) have also reported that the surfactants having a HLB-value of ~15 and of ~8 were optimal to enhance the performance of glyphosate (log *K*ow 236 < –3.4) and quizalofop-P ester (log *K*ow = 4.7), respectively. The results from this study indicated that the simulated rainfall increased remarkably the *ED50*- and *ED95*-values of clodinafop-propargyl (Table 1). The *ED*$_{50}$- and *ED*$_{95}$-value of clodinafop-propargyl increased from 19.09 g ha^{-1} to 29.15 g ha^{-1} for the former and from 25.42 g ha^{-1} to > 64 g ha^{-1} for the latter, respectively. In other words, from 1 kg ha^{-1} clodinafop-propargyl, 0.35 kg ha^{-1} (*RP*-value = 0.65) clodinafop-propargyl was washed off when rainfall was applied at 30 min after spraying (Fig. 2). Molin and Hirase (2005) found that glyphosate activity was reduced by 38% when simulated rain occurred within 30 min of treatment. Singh and Singh (2008) also reported that the surfactants improved rainfastness compared with glyphosate alone. According to our experiment data, Frigate and Citogate decreased adverse effects of rainfall on clodinafop-propargyl activity. As washing clodinafop-propargyl off wild oat leaves was minimized with Citogate better than with Frigate. The performance of 1 kg ha^{-1} clodinafop-propargyl in the presence of Frigate and Citogate, when rainfall was applied at 30 min after spraying, equals the performance of 0.77 and 0.83 kg ha^{-1} clodinafop-propargyl without rainfall, respectively (Fig. 2). The minimizing of rainfall adverse effect can be related to clodinafop-propargyl quicker absorption when the tested surfactants were added. Previous researches have shown that if surface tension is reduced, contact area (spreading) will be increased and caused to facilitate stomatal infiltration and promoted rainfastness for agrichemical (Penner, 2000; Molin and Hirase, 2005). Whereas, Citogate was an effective surfactant to decrease the surface tension of clodinafop-propargyl spray solution than Frigate (Fig. 1b), we may conclude that clodinafop-propargyl infiltrated in short-term before it was washed off the wild oat leaf surface.

Conclusion

Based on the results of this study; the following conclusions can be made: (*i*) by lowering the surface tension of spray solution with the surfactants Citogate and Frigate, the performance of clodinafop-propargyl has been highly improved to control wild oat, (*ii*) based on the tested of surfactants ability to lower the surface tension, Citogate > Frigate, and (*iii*) based on their ability to enhance the efficacy of clodinafop-propargyl under the simulated rainfall condition, Citogate > Frigate.

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