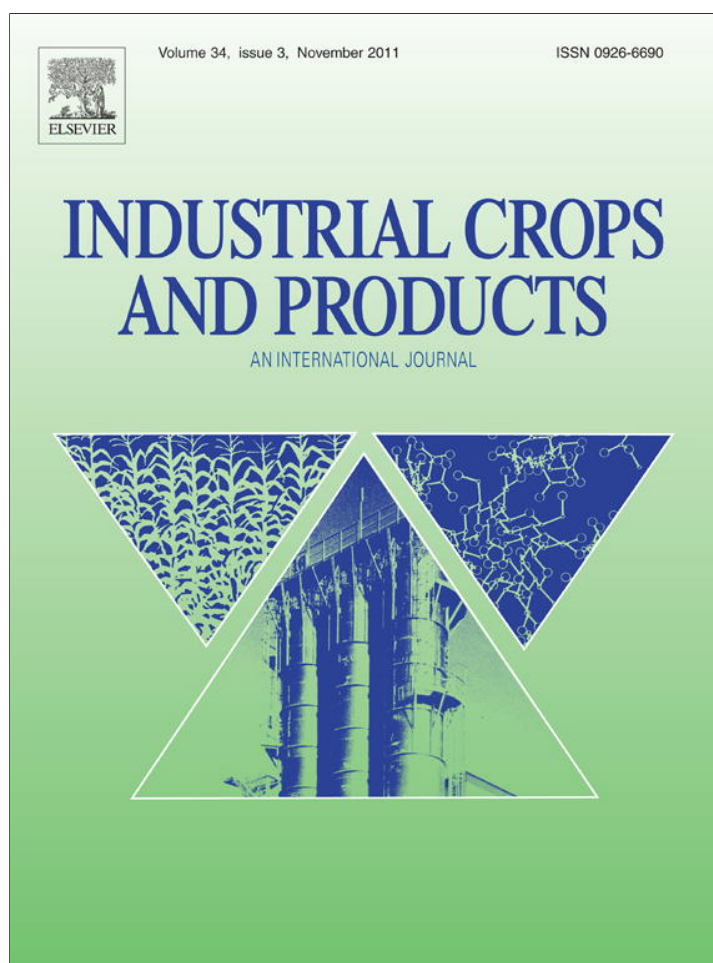


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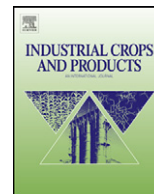
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Optimizing dosage of sethoxydim and fenoxaprop-*p*-ethyl with adjuvants to control wild oat

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ABSTRACT

Using adjuvants to optimize and increase the efficacy of herbicides is an acceptable manner to reduce herbicides undesirable impact on the environment. Therefore, to detect suitable adjuvants for sethoxydim or fenoxaprop-*p*-ethyl, two dose–response experiments were conducted separately. The treatments consisted of six doses of sethoxydim or fenoxaprop-*p*-ethyl with and without adjuvants of Frigate, Citogate and Adigor against wild oat (*Avena fatua* L.). Moreover, the surface tension of a range of concentrations of adjuvants and adjuvants + sethoxydim or fenoxaprop-*p*-ethyl aqueous solutions was determined. Lower and higher surface tension values were obtained with aqueous solution of Citogate and Frigate alone and with herbicides. When adjuvants were combined with herbicides, wild oat control was significantly increased and was more than when were used alone. Between evaluated herbicides, adjuvant receptivity for sethoxydim was higher than for fenoxaprop-*p*-ethyl. Among evaluated adjuvants, the addition of Frigate and Adigor had the lowest and the highest effect, respectively, on the performance of sethoxydim or fenoxaprop-*p*-ethyl in controlling wild oat, which supports the solubilizing nature of cuticular waxes by Adigor as a theory.

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1. Introduction

The history of agriculture has shown that humans strive to control weeds effectively at all times and they were able to discover various new methods for it (Rao, 2000). One of the most effective and modern methods to control a weed is the use of herbicides. Although there are other methods to control weeds, they rarely give the same degree of control as herbicides. Since the introduction of the phenoxy herbicides, in 1947, herbicides have been a tremendous contributor to world agriculture but may cause unfavorable effects on human and environment health (Kudsk, 2008).

Herbicides have become a key component of weed management programs in Iran. It has been reported that Iran arable lands have received approximately 11,100 tons of herbicides in 2006 (Baghestani et al., 2008). Previous reports have shown that if farmers did not have access to herbicides the overall income of the agriculture industry (Rao, 2000), and subsequently, the Gross National Product (Kudsk, 2008) would be reduced. The widespread use of herbicides due to leaching and runoff from agricultural land cause an herbicide contamination of agricultural and drinking water (Gawade et al., 2005). It stands to reason that there is an increasing focus on optimizing the dosage of herbicides. Herbicide

reduction programs have recently been enforced in many countries (Kudsk, 2008).

Fortunately, because of introducing and registering novel technologies into the herbicide industry, it has obtained an herbicide dosage reduction over the last four decades throughout the world (Jensen et al., 1998). For instance, during 1961–2004, the mean application rate of all registered herbicides in Iran has decreased from 5 to 0.54 kg ha⁻¹ (Deihimfard et al., 2007). Also, some initiatives have been taken to reduce herbicide dosage such as by spraying under favorable condition and weed growth early stages (Jordan, 1996; Pannacci and Covarelli, 2009).

Using adjuvants is one of the other acceptable manners to achieve this approach that can increase the performance of post-emergence herbicides through reducing surface tension (Kudsk, 2008). The decrease of the surface tension of the spray droplets can prevent bouncing off after impacting the leaves and allows for a low contact angle of the droplet on the leaf surface. The latter enhances spread and contact area of the drop on the leaf surface, which can allow more absorption of active ingredient (a.i.) by the leaves. Therefore, improving the penetrability of the a.i. is an implement not only to reduce the risk of side effects of herbicides but also user cost of them (Penner, 2000). Furthermore, adjuvants have been shown to delay crystallization of the a.i. on the leaf surface (Bunting et al., 2004), and to reduce the volatile and photodegradative properties of some herbicides which leads to more absorption of the a.i. of herbicides (Si et al., 2004). Moreover, some adjuvants break down

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quickly in the environment and do not have the risk of side effects (Penner, 2000). Adjuvants originate from renewable resources; as tallow amines (Frigate), a derivative of fatty tissue from sheep or cattle, contain a complex mixture of fatty acids including: linoleic, myristic, oleic, palmitic, and stearic (Brausch and Smith, 2007) and methylated seed oils (Adigor) are extracted from plant seeds that have been chemically modified (Hazen, 2000).

From a scientific, commercial, environmental, and farmer's point of view it is useful to be able to predict an adjuvant performance. Jensen et al. (1998) stated that each adjuvant is not able to increase absorption of all kinds of herbicides. Therefore, research is necessary to determine which adjuvant is most suitable with each herbicide. Thus, one of the major objectives of our research is to specify compatibility of adjuvants to increase herbicide performance.

2. Materials and methods

2.1. Surface tension

The static surface tension of aqueous solutions was measured by the capillary rise technique (Vanhanen et al., 2008) at $25 \pm 1^\circ\text{C}$ with four replications for each value, using the following equation:

$$\gamma = \frac{1}{2} \rho g r \left(h + \frac{r}{3} \right) \quad (1)$$

In this formula, r is the inner radius of the glass capillary tube (m) which was 0.5 mm, g is the acceleration due to gravity which is 9.8 m s^{-2} , ρ is density of liquid (kg m^{-3}), h is the capillary rise (m) and γ is the surface tension (Nm^{-1}) which turned to mN m^{-1} for analyzing. 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 adjuvant solution alone and formulated with sethoxydim or fenoxaprop-*p*-ethyl were determined. The static surface tension of aqueous solution of Frigate and Citogate alone have been already evaluated and reported by Aliverdi et al. (2009). The commercial sethoxydim or fenoxaprop-*p*-ethyl was prepared at a concentration corresponding to 375 g a.i. applied in 200 L distilled water for the former and 75 g a.i. applied in 200 L distilled water for the latter.

2.2. Plant material

Wild oat (*Avena fatua* L.) seeds were collected from plants nearby the Research Greenhouse of Collage of Agriculture in Ferdowsi University of Mashhad, Iran. They were dehulled by hand and placed in 11 cm diameter glass Petri dishes lined with a single layer of Wathman No. 1 filter paper. Ten milliliters of KNO_3 solution (2 g L^{-1}) was added to each Petri dish. Petri dishes were transferred to a refrigerator at $4\text{--}5^\circ\text{C}$ in the dark for 48 h, then, placed in an incubator at $20/10^\circ\text{C}$ in 45/65% relative humidity with a 16/8 h day/night, respectively, to germinate the seeds. After two days, the rootlets emerged from seeds; the seedlings were sown 1 cm deep in 2-L-plastic pots filled with a sterilized silt loam soil (19.8% sand, 20.1% clay, 58% silt, 4.1% organic matter and a pH of 6.7). The pots were placed in a greenhouse having a light/dark period of 16/8 h with $26\text{--}30/15\text{--}18^\circ\text{C}$ and 45/65% for relative humidity. To supplement natural sunlight and to extend the day length was used lamp light (400 W, Vialox® Nav®-T High-Pressure Sodium Vapor Lamp, Osram Co., Germany) with amount of illumination 2250 lux. The pots were subirrigated every three days with tap water. The seedlings, at one-leaf stage, were thinned from 10 to 4 per pot and fertilized once with 30 ml of a solution N:P:K (20:20:20) fertilizer at concentration of 3 g of fertilizer L^{-1} of tap water.

2.3. Treatments and chemicals

Sethoxydim at 0, 45, 94, 187, 281, and 375 g a.i. ha^{-1} (Nabos 12.5% EC, 125 g a.i. L^{-1} sethoxydim, BASF, Germany) and fenoxaprop-*p*-ethyl at 0, 9, 18, 37, 56, and 75 g a.i. ha^{-1} (Puma-Super 7.5% EW, 75 g a.i. L^{-1} fenoxaprop-*p*-ethyl, associated to safener of mefenpyr-diethyl 75 g a.i. L^{-1} , Aventis, France) were used in this study. The adjuvants included of (i) Citogate (a non-ionic surfactant, 100% alkyl aryl polyglycol ether, Zarnegar Pars, Iran) at 0.2% (v/v), (ii) Frigate (a cationic surfactant, 81.2% mixture of tallow amines ethoxylated long-chain fatty, ISK Biosciences Crop., Mentor, England) at 0.2% (v/v) and (iii) Adigor (a methylated seed oil, 44% methylated rapeseed oil, Syngenta, Switzerland) at 0.5% (v/v) were utilized. These concentrations were selected according to the label of the adjuvants. Two experiments were separately arranged in a completely randomized design with a factorial arrangement of treatments (herbicide dose by adjuvant application). There were four replications for both the treated and control plants. At the four-leaf stage, the plants were treated by using an overhead trolley sprayer equipped with a flat fan nozzle calibrated to deliver 200 L ha^{-1} at 200 kPa. Four weeks after treatment, biomass of experimental units (all of the plants in each pot) was harvested and oven dried at 75°C for 48 h and then weighed.

2.4. Statistical analysis

The response of wild oat foliage dry weight (U) on dose (z) was assumed to be by a log-logistic model (Streibig et al., 1993; Jensen et al., 1998; Pannacci and Covarelli, 2009):

$$U_{ij} = \frac{D - C}{1 + \exp[b_i(\log(z_{ij}) - \log(ED_{50(i)}))]} + C \quad (2)$$

where U_{ij} denotes the dry weight at the j th dose of the i th herbicide preparation (z_{ij}); D and C denote the upper and lower asymptote of dry weight at zero and at infinite doses and were assumed to be similar for all treatments within an experiment. $ED_{50(i)}$ denotes the required dose of herbicide, i , to give 50% weed control; and b_i is proportional to the slope of the curve around the $ED_{50(i)}$. The ED_{50} parameter in Eq. (2) can be replaced by any ED level (Pannacci and Covarelli, 2009), e.g. ED_{90} , which is of more practical relevance than the ED_{50} parameter when comparing herbicide preparations. The ED_{90} denotes the required dose of herbicide, i , to give 90% weed control. The logistic dose-response model was fitted to the experimental data, by the SlideWrite software (Computer Package Version 2.0. Advanced Graphics Software Inc., Encinitas, CA, USA). R parameter is the relative potency that is horizontal displacement between the two curves (Kudsk and Mathiassen, 2004). R indicates the ratio of the doses for commercial herbicide alone and commercial herbicide accompanied by adjuvant that give the same effect:

$$R = \frac{ED_{50A}}{ED_{50B}} \quad (3)$$

where ED_{50A} and ED_{50B} are the ED_{50} of herbicide A (without adjuvant) and B (with adjuvant), respectively.

If R was not different from 1.00, the addition of adjuvant would not have an effect on the responses of herbicide and when R was bigger or smaller than 1.00, the herbicide accompanied by adjuvant would be more or less potent than herbicide alone.

3. Results and discussion

3.1. Surface tension studies

The adjuvants of Citogate, Adigor and Frigate give superior performance in lowering surface tension of distilled water (Table 1).

Table 1
Influence of various concentrations of adjuvants on water surface tension.

Adjuvant concentration (v/v)	Surface tension (mN m^{-1}) \pm SD		
	Frigate	Citogate	Adigor
No adjuvant	72.24 \pm 0.32		
0.01%	57.79 \pm 0.47	61.10 \pm 0.22	59.38 \pm 0.23
0.05%	47.69 \pm 0.35	40.38 \pm 0.34	46.50 \pm 0.17
0.1%	42.36 \pm 0.16	35.58 \pm 0.29	38.42 \pm 0.10
0.15%	40.17 \pm 0.18	34.36 \pm 0.35	37.02 \pm 0.11
0.2%	39.79 \pm 0.21	33.86 \pm 0.20	36.65 \pm 0.31
0.25%	39.69 \pm 0.11	33.65 \pm 0.19	36.39 \pm 0.17
0.3%	39.53 \pm 0.26	33.64 \pm 0.23	35.98 \pm 0.16
LSD (0.01)		1.009	

All measurements were carried out at 25 ± 1 °C. Data of surface tension of Frigate and Citogate reported in this table have been already evaluated and reported by Aliverdi et al. (2009).

The water surface tension value decreased significantly as the concentration of Frigate and Adigor increased up to 0.1% ($P < 0.01$) and followed a steady state thereafter. While the water surface tension value decreased significantly with an increasing of Citogate concentration of up to 0.15%, and thereafter there was no a significant change (Table 1). The concentrations of 0.15% for Citogate and of 0.1% for Frigate and Adigor are considered to be the Critical Micelle Concentration (CMC). The CMC is the concentration at which an adjuvant forms micelles (Myers, 2006). Among evaluated adjuvants, the water surface tension value was the lowest for Citogate (33.64 mN m^{-1}) and the highest for Frigate (39.53 mN m^{-1}) solution (Table 1). This result may be related to the different adjuvants chemical structure. Frigate contains high ethylene oxide (15 units), while Citogate contains low ethylene oxide (7 units) in its chemical structure (Hazen, 2000). Water surface tension decreases with decreasing ethylene oxide number of surfactants (Sharma et al., 1996). Each supernumerary ethylene oxide unit added to surfactant reduces the packing density of hydrophobic groups at the water–surfactant interface, and therefore, results in a less reduction in the surface tension of the water (Myers, 2006).

The static surface tension of sethoxydim and fenoxaprop-p-ethyl solutions alone was 43.57 mN m^{-1} and 59.14 mN m^{-1} , respectively (Table 2). Among evaluated adjuvants, when Citogate and Frigate were added to each herbicide, the lowest and the highest values of surface tension at all concentrations were obtained, respectively ($P < 0.01$). According to data from this study, it is possible to rank the adjuvants in order of their performance in lowering surface tension with Citogate having the highest performance followed by Adigor and Frigate.

3.2. Bio-efficacy studies

The data from this study showed that, totally, the ED_{50} and ED_{90} values of sethoxydim or fenoxaprop-p-ethyl in the presence of the adjuvants decreased significantly (Table 3) and the R values were significantly higher than 1 (Fig. 1). This indicates a remarkable increase in the performance of the herbicides when the adjuvants were added to spray solution. The performance of 1 kg ha^{-1} sethoxydim, in the presence of adjuvants of Frigate, Citogate and Adigor, was equal the performance of 1.28, 1.43 and 1.49 kg ha^{-1} sethoxydim alone, respectively (Fig. 1a). In the other experiment, the performance of 1 kg ha^{-1} fenoxaprop-p-ethyl, in the presence of adjuvants of Frigate, Citogate and Adigor, was equal the performance of 1.19, 1.35 and 1.45 kg ha^{-1} fenoxaprop-p-ethyl alone, respectively (Fig. 1b).

These results indicated that, when sethoxydim or fenoxaprop-p-ethyl was combined with any one of the three adjuvants, wild oat control was significantly increased than when the herbicides were used alone. These effects may be related to improving retention of spray droplets on wild oat leaves. Penner (2000) reported that

the decrease of surface tension leads to the production of smaller droplets in the atomization process and, because of low energy in smaller droplets, the large number of droplets would remain on the leaf surface which can lead to increased performance of herbicide. Therefore, our study has clearly shown that by lowering the surface tension of spray solution with the adjuvants Citogate, Frigate or Adigor, the performance of sethoxydim or fenoxaprop-p-ethyl has been highly improved.

The results showed that, among evaluated adjuvants, Frigate and Adigor had the lowest and the highest effect on performance of sethoxydim and fenoxaprop-p-ethyl to control wild oat, respectively (Fig. 1). However, the application of all adjuvants leads to an increase in the performance of sethoxydim, which was higher

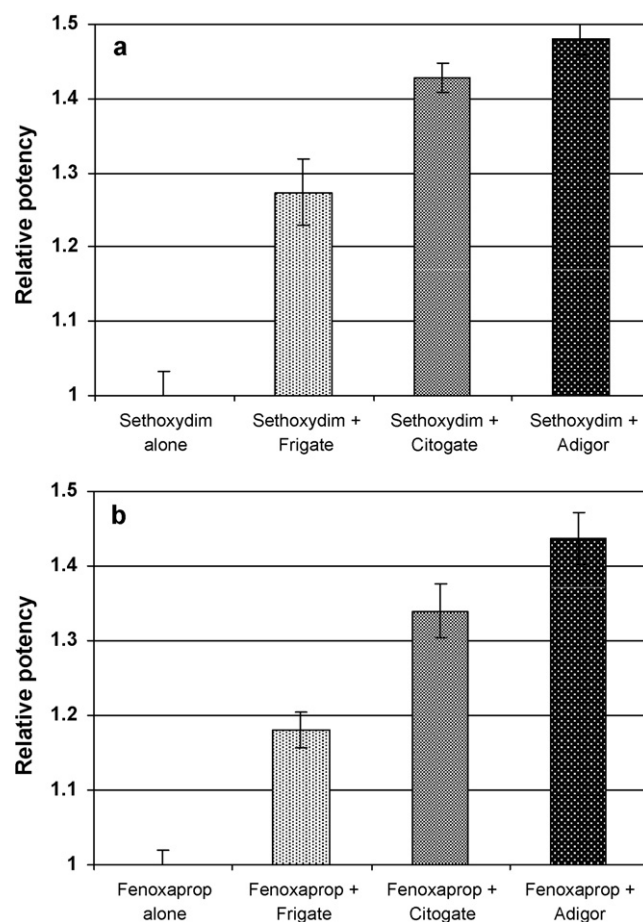


Fig. 1. Relative potency (R) values of sethoxydim (a) or fenoxaprop-p-ethyl (b) when combined with the adjuvants Frigate, Citogate, and Adigor on wild oat. Vertical bars are confidence intervals.

Table 2
Influence of various concentrations of adjuvants on the surface tension of sethoxydim or fenoxaprop-*p*-ethyl.

Adjuvant concentration (v/v)	Surface tension (mN m ⁻¹) ± SD					
	Sethoxydim ^a			Fenoxaprop- <i>p</i> -ethyl ^b		
	Frigate	Citogate	Adigor	Frigate	Citogate	Adigor
No adjuvant	43.57 ± 0.17			59.14 ± 0.15		
0.01%	39.56 ± 0.14	37.70 ± 0.27	40.60 ± 0.07	55.92 ± 0.26	52.43 ± 0.21	49.16 ± 0.22
0.05%	38.90 ± 0.17	32.95 ± 0.22	35.49 ± 0.18	41.77 ± 0.15	36.34 ± 0.05	38.88 ± 0.18
0.1%	38.35 ± 0.23	31.73 ± 0.12	33.83 ± 0.11	40.12 ± 0.12	34.33 ± 0.12	37.94 ± 0.08
0.15%	38.13 ± 0.26	31.49 ± 0.23	33.22 ± 0.14	40.11 ± 0.09	33.78 ± 0.19	37.44 ± 0.12
0.2%	37.49 ± 0.10	31.26 ± 0.05	32.89 ± 0.06	39.25 ± 0.10	33.58 ± 0.15	37.37 ± 0.13
0.25%	37.30 ± 0.13	30.96 ± 0.13	32.77 ± 0.05	38.71 ± 0.20	33.30 ± 0.20	37.34 ± 0.18
0.3%	37.32 ± 0.25	30.92 ± 0.17	32.74 ± 0.27	38.49 ± 0.12	32.59 ± 0.12	37.33 ± 0.06
LSD (0.01)		1.245			1.337	

^a 375 g a.i. sethoxydim.

^b 75 g a.i. fenoxaprop-*p*-ethyl per 200 L of distilled water.

Table 3
Estimated ED₅₀ and ED₉₀ of sethoxydim or fenoxaprop-*p*-ethyl alone or in the presence of various adjuvants in wild oat control.

	Treatment	ED ₅₀ (g a.i. ha ⁻¹) ± SD	ED ₉₀ (g a.i. ha ⁻¹) ± SD
1	Sethoxydim alone	93.78 ± 3.23	178.19 ± 3.40
	Sethoxydim + Citogate	65.67 ± 3.43	129.97 ± 3.71
	Sethoxydim + Frigate	73.62 ± 2.07	139.30 ± 2.25
	Sethoxydim + Adigor	63.39 ± 3.46	123.93 ± 3.14
2	Fenoxaprop- <i>p</i> -ethyl alone	16.88 ± 2.74	36.11 ± 2.94
	Fenoxaprop- <i>p</i> -ethyl + Citogate	12.60 ± 2.10	27.79 ± 2.14
	Fenoxaprop- <i>p</i> -ethyl + Frigate	14.31 ± 1.67	33.55 ± 1.71
	Fenoxaprop- <i>p</i> -ethyl + Adigor	11.75 ± 1.59	26.19 ± 1.62

Citogate and Frigate were added at 0.2% (v/v). Adigor was added at 0.5% (v/v).

than that of fenoxaprop-*p*-ethyl (Fig. 1). In other words, adjuvant receptivity for sethoxydim was higher than for fenoxaprop-*p*-ethyl. Among evaluated adjuvants, although Citogate had evinced a greater reduction on surface tension of herbicides solution (Table 2), Adigor (methylated rapeseed oil) had evinced a greater effect on performance of two herbicides (Table 3 and Fig. 1). Therefore, it seems that other factors of Adigor (except the decrease in surface tension) may contribute to its effectiveness.

Based on available information, surfactants (e.g. cationic Frigate and nonionic Citogate surfactants) are suitable to decrease surface tension, but they are not as effective as many methylated seed oils at increasing penetration (Penner, 2000; Sharma et al., 1996). Hazen (2000) reported that cuticular waxes can be plasticized, softened, or dissolved by methylated seed oils, allowing diffusion of a.i. to the more hydrophilic structures beneath, while cationic and non-ionic surfactants have no effect via softening or disruption of the cuticular waxes (Jensen et al., 1998). Therefore, an increase in the penetration of a.i. by softening or disrupting of the cuticular waxes is a more effective factor than a decrease in the surface tension of spray droplets (Sharma and Singh, 2000). Other studies have shown that methylated seed oils lead to better control with quinclorac (Zawierucha and Penner, 2001) and glyphosate (Collins and Helling, 2002) than cationic or nonionic surfactants.

The results indicated that the addition of Citogate had the higher influence than of Frigate on the performance of each of two herbicides against wild oat (Fig. 1). These results may be related to the different hydrophilic–lipophilic balance (HLB) between the adjuvants Citogate and Frigate. In surface tension studies, we have discovered that Citogate had low HLB (~8) so that when added to water gives opaque solutions. Unlike, Frigate had high HLB (>13) so that when added to water gives clear solution. HLB can be estimated by observing adjuvant dispersability in water with no dispersion = 1 to complete dispersion = 20 (Hess and Foy, 2000). High- and low-HLB adjuvants work best with water soluble herbicides (log *K*_{ow} < 1) and water insoluble herbicides (log *K*_{ow} > 1), respectively (Nalewaja et al., 1996; Zimdahl, 2007). Whereas, log *K*_{ow} for sethoxydim and

fenoxaprop-*p*-ethyl (1.56 and 4.58, respectively) is higher than 1, therefore, Citogate with a low-HLB increased the performance of this herbicides higher than Frigate with a high-HLB. Previous studies also have reported that, an adjuvant having a HLB value of ~15 and of ~8 were optimal to enhance performance of glyphosate (log *K*_{ow} = -3.4) and quizalofop-*p*-ester (log *K*_{ow} = 4.7), respectively (Nalewaja et al., 1996; Nalewaja and Matysiak, 1993). Our study results not only confirmed previous results but also showed that the relationship between HLB of adjuvant and log *K*_{ow} of herbicide is strongly relevant.

4. Conclusion

Herbicides dosage reduction is a research priority because of their bad visage. Fortunately, the use of adjuvants as a technique has become a key component to optimize the dosage of post-emergence herbicides. Based on the results; the following conclusions can be made: (i) by lowering the surface tension of spray solution with the adjuvants Citogate, Frigate or Adigor, the performance of sethoxydim or fenoxaprop-*p*-ethyl has been highly improved, (ii) the adjuvants Frigate, Citogate and Adigor increased the performance of sethoxydim or fenoxaprop-*p*-ethyl for controlling wild oat, (iii) adjuvant receptivity for sethoxydim was higher than for fenoxaprop-*p*-ethyl, (iv) it is possible to rank the adjuvants in order for their ability to lower the surface tension and enhance efficacy of sethoxydim or fenoxaprop-*p*-ethyl as Citogate > Adigor > Frigate for the former and Adigor > Citogate > Frigate for the later.

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