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Optimizing diquat efficacy with the use of adjuvants

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Abstract Optimizing diquat efficacy with the use of adjuvants may broaden the spectrum of weed control, but relevant research towards this direction is limited. Field and greenhouse experiments were conducted to evaluate the effect of diquat applied alone and with six commercial adjuvants (surfactants and oil-based adjuvants) on various weed species. Diquat effect was evaluated in two field experiments on natural populations of common lambsquarters (*Chenopodium album* L.), prostrate knotweed (*Polygonum aviculare* L.) and burning nettle (*Urtica urens* L.) along with two greenhouse trials on rigid ryegrass (*Lolium rigidum* L.). In field or greenhouse experiments, all the adjuvants significantly increased the control of *C. album, P. aviculare*, and *L. rigidum*, from 48, 42 and 7%, up to 82, 74 and 67%, respectively, in terms of fresh weight reduction, but to a different extent for each adjuvant. *U. urens* was totally (100%) controlled in terms of visual estimation either with diquat or with diquat plus any adjuvant. The differences in the effect of diquat applied with adjuvants mainly depended on the weed species examined and they were not proportional to the surface tension reduction of the spray solution caused by the adjuvants. Overall, the surfactants and the oil-based adjuvants examined in this study considerably enhanced the effect of diquat; this can broaden the spectrum of weed control against broadleaf and grass weeds in orchards and non-crop areas. The results are discussed in relation with the classification of the adjuvants.

Keywords Bipyridylium herbicides · Efficacy · Surfactants · Oil-based adjuvants

Introduction

Diquat is a non-selective contact herbicide for the control of broadleaf and grass weeds in non-crop and aquatic areas (U.S. EPA 1995). As water soluble dibromide, it is used in orchards and vineyards (Menendez and Bastida 2004) by strict spot spraying at a rate of 400–1000 g ai ha^{-1} (Tomlin 2000). Rates depend on weed growth stage at application. Diquat is applied up to 500 L ha^{-1} spray volume at low pressure such that of 100 kPa (for a knapsack sprayer) to avoid air drift and contact of the spray solution with the foliage and other green parts of trees (Anonymous 2007). This herbicide is also used to facilitate crop harvest by desiccating weeds, accelerating crop drying, and reducing seed moisture content (De Souza Lacerda et al. 2005). Additionally, it is used as an aquatic herbicide (Clayton and Matheson 2010) to control algae, submersed and floating aquatic weeds in ponds, lakes, and drainage ditches (Ahrens 1994).

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To increase herbicide efficacy on weeds, foliar-applied herbicides are often used in combination with adjuvants to improve foliar coverage, spray retention on the foliage, and absorption by leaf surfaces (Kudsk and Streibig 1993). Adjuvants are classified as those that modify the physical characteristics of the spray solution and those that enhance the biological efficacy of a pesticide (Hazen 2000). Adjuvants can modify the physical characteristics of the spray solution by adjusting the pH, preventing or decreasing the formation of foam, increasing the spray deposition on leaf surface, enhancing the target coverage, providing herbicide rainfastness, or increasing the drying time of the aqueous spray deposit; moreover, they can enhance the biological efficacy of a pesticide by increasing its absorption and activity through dissolving the cuticular leaf waxes or via infiltrating the leaf stomata (Hazen 2000; Penner 2000). Adjuvants can overcome impediments caused by adverse environmental conditions and may contribute to decreasing variability in herbicide performance, an aspect that needs much more attention for reducing herbicide inputs (Kudsk 2002).

As the bipyridylium herbicides rely strongly on contact for their foliar activity, good coverage of the leaf surfaces is essential for optimum herbicide performance. For these herbicides, it is important that the leaves and stems are thoroughly wetted for efficient activity, particularly when treating dense and tall vegetation. The addition of adjuvants in the spray solution could enhance diquat efficacy and broaden the spectrum of weed control. This is was evident in some previous studies (Gilreath and Gilreath 1989; Menendez and Bastida 2004; Gitsopoulos et al. 2014). In the market there are numerous adjuvants under different classification, such as surfactants that reduce the surface tension and improve spreading, sticking and herbicide uptake as well as crop oil adjuvants that also reduce the surface tension, improve leaf spreading, herbicide penetration and uptake or decrease crystallization of the active ingredient (Miller and Westra 1996; Zhang et al. 2013). Vegetable oils, such as rapeseed oil, improve herbicide adhesion and uptake on plants and protect herbicide spray from rain, evaporation, dew and sunlight. Organosilicone surfactants reduce the surface tension to a greater extent compared to other adjuvants and allow the spray solution to run into stomata on the leaves (Knoche 1994). Considering the emphasis given on herbicide efficacy, the use of adjuvants is a great concern. Non-ionic surfactants are normally recommended for diquat (Puri et al. 2008). However, information concerning the effect of adjuvants of different classification on diquat activity remains limited. Optimizing diquat efficacy with the use of adjuvants may broaden the spectrum of weed control; however, to our knowledge relevant research towards this direction is limited. Thus, the objective of this study was to evaluate the effect of six commercially available adjuvants of different classification on diquat efficacy against broadleaf and grass weeds under field and greenhouse conditions.

Materials and methods

Field experiments

A field experiment was conducted in April 2013 and repeated in April 2015 in an experimental field of the Institute of Plant Breeding and Genetic Resources in northern Greece. The physicochemical characteristics of the soil were clay 6.8%, silt 32%, sand 61.2%, organic matter 1.85%, $CaCO_3$ 3.5%, pH 7.7 and cation exchange capacity (CEC) 3.5 meq 100 g^{-1} soil. The field was naturally infested by common lambsquarters (*Chenopodium album* L.), prostrate knotweed (*Polygonum aviculare* L.) and burning nettle (*Urtica urens* L.) as the dominant weed species; these weed species were present in patches in all field plots, whereas other species, such as common poppy (*Papaver rhoeas* L.) and brome grass (*Bromus* spp.), were not present in all plots and for this reason they were not examined in the present study. Both experiments were arranged in a randomized complete block design with four replicates; each plot size was 3-m long and 4-m wide with an adjacent non-treated plot served as control.

C. album and *U. urens* plants were at a mean height of 15 to 20 cm by the time of herbicide application. *P. aviculare* plants had stems about 20 cm long. Herbicides were applied on 21 April 2013 and 27 April 2015. Herbicide treatments consisted of foliar applications of diquat at 0.9 kg ai ha^{-1} in a soluble liquid (SL) formulation alone and in mixtures with each of the six commercially available adjuvants: a) isodecyl-alcohol-ethoxylate (non-ionic surfactant), b) paraffinic oil (crop oil concentrate), c) blend of fatty acid esters plus alkoxylated alcohols-phosphate esters (non-ionic wetting agent), d) rapeseed oil (vegetable oil), e) polyether-polymethylsiloxane-copolymer (organosilicone surfactant), and f) methylated rapeseed oil (methylated vegetable oil). The trade names and the rates of the adjuvants are presented in Table 1. Diquat rate was within the recommended dose range of the product label. Herbicide solutions were applied with a hand-held AZO portable 2.4-m-wide boom field plot sprayer, fitted with AI XR Teejet 11002 nozzles and calibrated to deliver 500 L ha^{-1} at a pressure of 215 kPa. The high carrier volume was applied to ensure a thorough coverage of the weed foliage as indicated in the product label (Anonymous 2007). Weather parameters 4 days before, after and the day of herbicide applications are shown in Table 2.

Diquat effect was estimated by recording plant fresh weight, as typically used in assessments of herbicide efficacy among other continuous variables, according to common international standards (EPPO 2007, 2012). When complete control (dead plants) was evident, no fresh weight was measured but visual estimation of weed control was performed on a scale 0–100%, where 0% indicated no control and 100% indicated dead plants. For fresh weight measurements, 10 plants of each weed species, considered as the minimum representative number for weed control estimation, were randomly selected from different patches within the center of each plot, cut at ground level and the total fresh weight for each weed species was recorded separately. Then the fresh weight plant^{-1} was calculated and used for data analysis. Fresh weight recordings were conducted at 10 DAT. An analysis of variance (ANOVA) was conducted to test the effect of diquat solutions on weed species. Before the ANOVA, weed control percentages (%) of visual estimation were arcsine transformed, whereas fresh weight data were log(x)-transformed to normalize variance. Non-transformed data of percentages of visual estimation showed no differences with the arcsine transformed percentages in mean separation, therefore original means were used for ANOVA. Since there was no interaction effect between treatments and years, visual estimation and fresh weight data were pooled over years. Original means of visual estimation for *U. urens* control and plant fresh weight for *C. album* and *P. aviculare* are presented (Table 3). Means of all data were separated using Fishers' protected LSD test at 5% level of significance.

Table 2 Mean temperature and rainfall before, after and the day of herbicide applications for year 2013 and year 2015

Days from application	Year 2013[a]		Year 2015[b]	
	Temperature (°C)	Rainfall (mm)	Temperature (°C)	Rainfall (mm)
−4	15.7	0.0	15.2	0.0
−3	14.7	0.0	14.2	0.0
−2	14.0	0.0	13.6	0.0
−1	14.7	0.0	15.8	0.0
0	16.4	0.0	15.3	0.0
+1	17.0	0.0	15.5	0.0
+2	16.9	0.0	17.6	0.0
+3	18.4	0.0	18.8	0.0
+4	19.6	0.0	18.0	0.0

[a] http://meteosearch.meteo.gr/data/thessaloniki/2013-04.txt

[b] http://meteosearch.meteo.gr/data/thessaloniki/2015-04.txt

Greenhouse experiments

A pot experiment was conducted twice (two runs) with a week interval under greenhouse conditions in March 2016. Seeds of rigid ryegrass (*Lolium rigidum* L.) were planted in 0.65-L cylindrical

Table 1 Classification, content, trade names and rates of the adjuvants

Classification	Content	Trade name	Rates
Non-ionic surfactant	Isodecyl-alcohol-ethoxylate 90%	TREND®	100 mL/hL
Paraffinic oil	Paraffinic oil 60%	ATPLUS®	1000 mL/hL
Non-ionic agent	Mixture of fatty acid esters (37.5%) and alkoxylated alcohols-phoshate esters (22.5%)	DASH HC®	1000 mL/ha
Vegetable oil	Rapeseed oil 86.4%	CODACIDE®	2000 mL/ha
Organosilicone surfactant	Polyether-polymethylsiloxane-copolymer 100%	BREAKTHRU S240®	20 mL/hL
Vegetable oil	Methylated rapeseed oil 47%	ADIGOR®	1%

Table 3 Effect[a] of diquat applied alone and with various adjuvants on *Chenopodium. album*, *Polygonum aviculare* and *Urtica urens*

Treatment	*C. album* Fresh weight (g/plant[b])	*P. aviculare*	*U. urens* % control[c]
Diquat	12.0 b[#]	21.5 a	100 a
Diquat+ Isodecyl-alcohol-ethoxylate	6.6 cd	10.9 b	100 a
Diquat + Paraffinic oil	6.0 cd	12.5 b	100 a
Diquat + Mixture of fatty acid esters and alkoxylated alcohols-phoshate esters	5.3 d	14.2 b	100 a
Diquat + Rapeseed oil	5.9 d	13.5 b	100 a
Diquat + Polyether-polymethylsiloxane-copolymer	7.6 c	11.5 b	100 a
Diquat + Methylated rapeseed oil	6.2 cd	7.7 c	100 a
Untreated control	20.6 a	23.2 a	0

[a] Data are means of two experiments

[b] Log (x) transformation for *C. album* and *P. aviculare* was used for the statistical analysis. Original data are presented

[c] On a scale 0% (no control) to 100% (dead plants) of visual estimation. Untreated control values were excluded from the statistical analysis

[#] Means followed by same letter do not significantly differ at 5% level of significance

plastic pots filled with sieved soil. The soil used was collected from the field of the experiments described above. The mean temperature under greenhouse conditions was 14 °C, ranging from 8 to 24 °C and pots were watered once daily throughout the experiments. Plants grew up normally without experiencing any particular environmental stress conditions in terms of temperature, nutrient and water requirements. One week after emergence, plants were thinned to 10 plants per pot to obtain a uniform population in all pots. The same treatments described in the field experiments were applied outdoors with the same boom sprayer when plants were at the growth stage of three to four leaves. After herbicide treatments pots were transferred back to the greenhouse bench and remained there till the end of experiment. A randomized complete block design was used with five replications. Non-sprayed pots were used as control. The effect of the adjuvants on diquat for the control of *L. rigidum* was assessed by determining shoot fresh weight of plants cut at the soil surface at 10 DAT. Fresh weight per pot was used for ANOVA. Before the ANOVA, data were log (x + 1) transformed to normalize variance. Since there was no interaction effect between treatments and runs, the data were pooled over runs. Original data means of pot fresh weight are presented (Table 4). Means were separated using Fishers' protected LSD test at 5% level of significance.

Laboratory assessments for surface tension and pH of the spray solutions

The surface tension (ST) of all spray solutions was determined. The ST assessments were performed by using the *Traube* stalagmometer. ST was determined by the number of drops that fall from the stalagmometer, the density of each spray solution, while the ST of distilled water was used as a reference liquid; the drop

Table 4 Effect[a] of diquat applied alone and with various adjuvants on *Lolium rigidum*

Treatment	Fresh weight (g/pot[b])
Diquat	2.37 b[#]
Diquat+ Isodecyl-alcohol-ethoxylate	0.89 d
Diquat + Paraffinic oil	1.62 c
Diquat + Mixture of fatty acid esters and alkoxylated alcohols-phoshate esters	1.47 c
Diquat + Rapeseed oil	1.62 c
Diquat + Organosilicone surfactant	1.62 c
Diquat + Methylated rapeseed oil	0.77 d
Untreated control	4.57 a

[a] Data are means of two runs of the experiment

[b] Log (x + 1) transformation for *L. rigidum* was used for the statistical analysis. Original data are presented

[#] Means followed by same letter do not significantly differ at 5% level of significance weights are proportional to the ST, so that the ST of each spray solution is calculated according to Eq. 1 (Dilmohamud et al. 2005; Momin and Thakre 2016).

$$\sigma_x = \frac{\sigma_w \times N_w \times \rho_x}{N_x \times \rho_w} \qquad (1)$$

where, σ_x is the ST of each spray solution, σ_w is the distilled water ST, N_x is the number of drops of each spray solution, N_w is the number of drops of water, ρ_x is the density of each spray solution and ρ_w is the density of water. The number of drops and the densities were determined in triplicate and the average values were used for calculations of ST of each spray solution. A pH-meter was used for the pH determination. Both ST and pH measurements (Table 5) were performed at room temperature (25 °C). All adjuvant solutions were prepared with distilled water; the ST and the density of the latter was assumed to be 71.97 dynes/cm and 0.9971 g/mL at 25 °C, respectively.

Results

Field experiments

Diquat applied alone or in mixture with each of the adjuvants provided total control of *U. urens* (100% based on visual estimation). The addition of the adjuvants did not alter the control of *U. urens*, indicating no antagonistic effect between the adjuvants and diquat. In *C. album,* diquat caused 42% reduction in fresh weight compared to the untreated control; however, this reduction was significantly pronounced (63 to 74%) when adjuvants were added in the spray solutions (Table 3). In particular, diquat with the non-ionic wetting agent, the rapeseed oil, the paraffinic oil and the methylated rapeseed oil reduced fresh weight of *C. album* by 70 to 74%, whereas the addition of the isodecyl-alcohol-ethoxylate surfactant and the organosilicone caused 68 and 63% fresh weight reduction, respectively (Table 3). In *P. aviculare,* diquat applied alone caused very little (7%) fresh weight reduction compared to the untreated control; however, this reduction was significantly pronounced (39 to 67%) with the addition of adjuvants (Table 3). More specifically, the addition of methylated rapeseed oil caused the highest fresh weight reduction (67%), whereas the isodecyl-alcohol-ethoxylate, the organosilicone, the paraffinic oil, the rapeseed oil and the non-ionic wetting agent caused 53, 50, 46, 42 and 39% fresh weight reduction, respectively (Table 3).

Greenhouse experiments

Diquat applied alone caused 48% *L. rigidum* fresh weight reduction compared to the untreated control; however, this reduction was significantly enhanced to 65 to 83% when adjuvants were added in the spray solution (Table 4). More specifically, the methylated rapeseed oil and the isodecyl-alcohol-ethoxylate surfactant provided consistently the maximum diquat efficacy (83 and 80% fresh weight reduction, respectively), followed by the non-ionic wetting agent (68%) and the other three adjuvants (65%) (Table 4).

Laboratory assessments for ST and pH of the spray solutions

Surface tension was reduced by all adjuvants and in particular to a great extent by organosilicone and the isodecyl-alcohol-ethoxylate surfactants (31.4 and 34

Table 5 Surface tension and pH of diquat spray solutions and distilled water

Treatment	Surface tension (dynes/cm)	pH
Diquat	76.20	6.58
Diquat+ Isodecyl-alcohol-ethoxylate	34.00	6.61
Diquat + Paraffinic oil	42.40	6.57
Diquat + Mixture of fatty acids methyl esters and alkoxylated alcohols-phoshate esters	44.70	6.32
Diquat + Rapeseed oil	62.00	6.56
Diquat + Organosilicone surfactant	31.40	6.58
Diquat + Methylated rapeseed oil	48.50	6.62
Distilled Water	71.97	6.65

dynes/cm, respectively). Rapeseed oil reduced the surface tension less compared to the other adjuvants (62 dynes/cm), whereas the non-ionic wetting agent, the methylated rapeseed oil and the paraffinic oil reduced the surface tension to a range from 42.4 to 48.5 dynes/cm. None of the added adjuvants decreased or increased the pH of spray solution more than 0.5 (Table 5).

Discussion

The results of the present study indicated that diquat efficacy against the broadleaf weeds *U. urens*, *C. album* and *P. aviculare* and the grass weed *L. rigidum* was enhanced by all the adjuvants tested, but differently for each weed species and to a different extent for each adjuvant. *U. urens* was perfectly controlled by diquat alone as much as by all diquat mixtures with the adjuvants, indicating no antagonistic effect. The increased efficacy observed on *C. album* and *P. aviculare* or *L. rigidum* in field or pot experiments, respectively, was not proportional to the ST reduction of the spray solutions caused by the adjuvants. On the contrary, adjuvants that caused low or intermediate ST reduction enhanced diquat efficacy to a similar or greater extent compared to adjuvants that caused high ST decrease. In general, most commercial adjuvants increase the contact area between the spray droplet and the leaf area by reducing the ST of the spray solution and lead to higher adhesion and surface coverage (Basu et al. 2002). However, this response does not always imply higher herbicide efficacy and weed mortality (Singh and Singh 2006); this trend was evident in our study, where although both organosilicone and isodecyl-alcohol-ethxylate surfactants did cause the highest ST reduction among adjuvants, diquat efficacy was not proportionally increased on *C. album*, *P. aviculare* and *L. rigidum*.

Moreover, oil adjuvants increased diquat efficacy on the three aforementioned weed species, despite the fact that due to their hydrophobic character they could be considered more suitable for lipophilic herbicides than hydrophilic and polar compounds such as diquat. According to Lin et al. (2016), a surfactant may dissolve the epicuticular leaf waxes, while vegetable oils and crop oil concentrates could solubilize, dissolve or disrupt the nature of cuticular waxes promoting the penetration of active ingredients (Hazen 2000; Zabkiewicz 2000). This disorder is believed to be another important factor in improving herbicide efficacy by increasing the penetration dose of the active ingredient. Liu (2004) reported that vegetable oil adjuvants seemed to enhance herbicide activity by increasing the spreading of the spray droplets on plants and by enhancing the penetration of the active ingredient into the leaves; the latter is assumed since many oil-based adjuvants act well as penetration enhancers (Stock and Briggs 2000). In a recent study, different etholxylated rapeseed oil adjuvants in diquat spray solutions for the control of *C. album* caused diquat salts to be deposited more centrally in the droplet footprint on the leaves of *C. album* and this enhanced its penetration into the leaves (Basi et al. 2013). These oils might have concentrated on the top of the droplet due to their lighter weight, resulting in ST reduction (Faers and Pontzen 2008). The above mentioned mechanims could have promoted diquat efficacy in our study as well, apart from the reduction in ST reported. For the rapeseed oil adjuvant, it was reported that its addition to diquat-dibromide spray solution superiorly controlled monocot and broadleaf weeds than diquat-dibromide alone (Lanszki 2011), and significantly reduced drift in aerial application. In addition, it improved the desiccation of sunflower as compared to diquat-dibromide applied alone (Palmai and Gyulai 2009). Menendez and Bastida (2004) also revealed enhancement of diquat efficacy against *L. rigidum* with the addition of surfactants, oils, and esters, allowing reduction of herbicide rates. Additionally, Gitsopoulos et al. (2014) reported an increase in diquat efficacy against the grass weeds *Bromus sterilis, Avena sterilis* and *Lolium multiflorum* with the use of some of the adjuvants used in the present study. Finally, diquat showed 99% efficacy for the control of duckweed (*Lemna minor* L.) when applied together with a silicone surfactant (Langeland et al. 2002). The results of these previous studies come in agreement with the present study, supporting the findings of the increased efficacy of diquat with the addition of adjuvants of different classification.

Concerning the weeds tested in the present study, *C. album* is considered a difficult-to-wet species and this makes herbicide diffusion to become limited since *C. album* leaves present a crystalline wax form (Hess and Foy 2000). Indeed, the epicuticular wax in *C. album* impedes the retention and penetration of water and hydrophilic molecules like diquat due to the presence of aldehydes (Taylor et al. 1981), making the leaf surface rough and hydrophobic. Moreover, the epicuticular wax has been shown to be present on both leaf surfaces of this weed species, forming small platelets with greater density and depth on the abaxial surface (Taylor et al. 1981). Thus, leaf surfaces of *C. album* present a high degree of water-repellency. For *P. aviculare*, it has been also reviewed by Pinke et al. (2014) that it exhibits a high phenotypic plasticity and it can significantly reduce its leaf surface making the herbicide foliar adhesion and uptake more difficult (Meerts 1995; Sultan 2003), whereas leaf wax of grasses is generally dominated by the crystalline form (Wang and Liu 2007). More specifically, the epicuticular wax on the lower *Lolium* leaf surface formed amorphous sheets, characterized by extreme glossiness, whereas the upper surface was reported to be glaucous and formed crystalline plate waxes (Carver et al. 1990). The adjuvants used in the present study may have influenced not only the ST of the spray solution, but characteristics of droplet deposit and other partial processes in terms of penetration and bioefficacy (Basi et al. 2012). The adjuvant effect showed to be weed species dependent in association with the adjuvant properties.

Conclusions

The results of the present study revealed that diquat efficacy was increased with the use of all adjuvants tested, albeit not proportionally to ST reduction, and no antagonistic effect of any of the adjuvants on diquat effect was observed. The oil-based adjuvants enhanced the efficacy of the hydrophilic herbicide diquat, as the surfactants did. The increased weed control could be attributed not only on the ST reduction caused by the adjuvants, but on other key points that enhanced diquat absorption, such as more centralized deposit area or more accessible polar pathways. The adjuvant selection for diquat should be based on the target weed species and the present study showed that increased weed control was depended on weed species characteristics and adjuvant properties. However, more experimentation is needed to indicate the exact way that diquat increased its efficacy, in relation to adjuvant rates and weed species targeted. Overall, certain adjuvants used in the present study can increase considerably the efficacy of diquat on the broadleaf and grass weed species tested in orchards and non-crop areas or allow for more efficient banded non-selective diquat applications.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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