

Doi: <http://dx.doi.org/10.1590/1809-4430-Eng.Agric.v40n4p555-560/2020>

## TECHNICAL PAPER

# WATER HARDNESS AND pH IN THE EFFECTIVENESS OF GLYPHOSATE FORMULATIONS

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### KEYWORDS

herbicide, water quality, spraying, application technology.

### ABSTRACT

The hardness and pH of the spray water can interfere with the weed control effectiveness with herbicides, but it is not clear the magnitude of this interference, mainly associating different levels of pH and hardness to different glyphosate formulations. This study aimed to evaluate the influence of hardness and pH, in association, of the water used in the application of two glyphosate formulations on the weed control effectiveness. The experiment was conducted in duplicate, in areas with a predominance of *Digitaria horizontalis*, under a randomized block design with a 4×4×2+1 factorial scheme, composed of four water hardness levels (70, 110, 230, and 430 ppm CaCO<sub>3</sub>), four pH levels (3.5, 4.5, 5.5, and 6.5), two glyphosate formulations (ammonium salt and potassium salt), and control without application, with four repetitions. The physicochemical characteristics of the spray solutions and the weed control effectiveness were evaluated at 7, 14, and 21 days after application (DAA). The water pH at the studied range did not interfere with the control effectiveness. The increase in hardness reduced the control at 7 DAA, but this difference was not noticed after 21 DAA. Glyphosate ammonium salt promoted higher control of *D. horizontalis* than that with potassium salt, regardless of water hardness and pH.

### INTRODUCTION

The increase in crop productivity has become an essential factor in ensuring food security (Tilman & Clark, 2015). Thus, the use of phytosanitary products has gained increasing importance in maintaining quality and high yields. However, some of these products have undergone a decrease in the effectiveness of their active ingredients, which is related, among other factors, to the used application technology. One of the points often neglected is related to the quality of the water used in the composition of the spray solution (Farias et al., 2014).

Good quality water is vitally important to increase the efficiency of agricultural spraying, as it influences the effectiveness of products that use it as a diluent. Water quality is associated with different physical and chemical parameters.

Hardness and pH are considered significant factors regarding chemical quality (Devkota & Johnson, 2016).

Hardness is related to the concentrations of carbonates, sulfates, chlorides, and nitrates of various cations, mostly calcium and magnesium, being presented in the form of CaCO<sub>3</sub> (ppm). On the other hand, pH is a measure of the degree of activity of free hydrogen ions (H<sup>+</sup>) in the solution (Kissmann, 1997). Changes in these parameters are pointed out as responsible for reducing the effectiveness of different active ingredients, among which are some herbicides used for weed control (Dan et al., 2009; Devkota et al., 2016).

Hard water (high Ca<sup>2+</sup> and Mg<sup>2+</sup> concentrations) can act on some herbicides replacing the Na<sup>+</sup> and K<sup>+</sup> present in their structures by Ca<sup>2+</sup> and Mg<sup>2+</sup>, changing their properties and reducing their effectiveness. Similarly, high pH values

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can contribute to the early degradation of the active principle, or even interfere with the herbicide absorption by plant tissues (Kissmann, 1997).

Glyphosate [N-(phosphonomethyl)glycine] is the world's most widely used herbicide, with an important role in agriculture (Benbrook, 2016). It is applied in desiccation and post-emergence, being classified as a non-selective herbicide with systemic action. It has a wide spectrum of action, controlling annual or perennial weeds, dicots and monocots (Melo et al., 2019).

Glyphosate is found in several formulations, all of which have the same mechanism of action, but varying regarding the salts that compose it (May et al., 2016). Isopropylamine, ammonium, and potassium stand out among these salts, which have no herbicidal activity, but they are converted into an acidic form with herbicidal activity when absorbed by the plant (D'Amato, 2017).

However, little is known about the relationship between the different glyphosate salts and the representative parameters of water quality, and the results of these interactions on the active ingredient effectiveness in the field. In general, there is an understanding that glyphosate should be applied using water with a low pH (usually 4 to 5) and hardness, as mentioned by Carvalho et al. (2009), but the magnitude of this interference to control weeds in the field is still not clear, especially when associating these two characteristics. Ramos & Araújo (2006), for instance, stated that the pH of application water should be analyzed only as an indicator of possible changes in the chemical characteristics of water. Interference in the effectiveness of the applied product is rarely observed if only the pH is changed. Soltani et al. (2011) found no differences in weed control (*Abutilon theophrasti*, *Amaranthus retroflexus*, *Chenopodium album*, and other grasses) with glyphosate using water with varying degrees of hardness.

Given this divergence relative to the effect of water characteristics on the effectiveness of phytosanitary products, this study aimed to evaluate the influence of hardness and pH, in association, of the water used in the application of different glyphosate formulations on the weed control effectiveness.

## MATERIAL AND METHODS

This study was carried out at the Capim Branco Experimental Farm (18°53'19" S and 48°20'57" W, an altitude of 835 m, and average annual precipitation of 1250 mm), and the laboratory stage was carried out at the Laboratory of Agricultural Mechanization, both belonging to the Federal University of Uberlândia, Uberlândia, MG, Brazil.

The experiment was conducted in duplicate under a randomized block design with a 4×4×2+1 factorial scheme, composed of four water hardness levels (70, 110, 230, and 430 ppm), four pH levels (3.5, 4.5, 5.5, and 6.5), two glyphosate formulations (ammonium salt and potassium salt), and a control treatment without application, with four replications, totaling 132 plots for each experiment. The total area of each plot had 10 m<sup>2</sup> (5 × 2 m), and 1 m from each end was not considered for evaluation purposes.

Field applications were carried out independently in two fallow areas for three months, with an infestation of 90% of *Digitaria horizontalis* and 10% of other species in the first area, and 50% of *D. horizontalis*, 10% of *Eleusine indica*, 10% of *Cenchrus echinatus*, 10% of *Ipomoea*

*indivisa*, 5% of *Alternanthera tenella*, and 15% of other species in the second area. The plants had an average height between 15 and 25 cm at the application time.

The applications of herbicide spray solutions were carried out using a CO<sub>2</sub>-pressurized knapsack sprayer with a 2-m boom and four air-induction flat fan spray tips 11001 (AirMix<sup>®</sup>, Agrotop). The application rate was 100 L ha<sup>-1</sup>, with a pressure of 200 kPa and an application speed was 4.0 km h<sup>-1</sup>. The meteorological conditions during the applications were monitored by a thermo-hygro-anemometer (4000, Kestrel), which registered temperature, relative air humidity, and wind speed values ranging from 25 to 31 °C, 65 to 76%, and 1.0 to 3.2 km h<sup>-1</sup>, respectively.

Herbicide doses were selected according to the product label, using *D. horizontalis* control as a reference, i.e., 1 kg ha<sup>-1</sup> for ammonium salt and 1.4 L ha<sup>-1</sup> for potassium salt. The ammonium salt of N-(phosphonomethyl)glycine (Roundup<sup>®</sup> WG, Monsanto) was used at a concentration of 792.5 g kg<sup>-1</sup> (720 g acid equivalent kg<sup>-1</sup>) in the dispersible granule formulation, while the potassium salt of N-(phosphonomethyl)glycine (Zapp Qi<sup>®</sup> 620, Syngenta) was used at a concentration of 620 g L<sup>-1</sup> (500 g acid equivalent L<sup>-1</sup>) in the soluble concentrate formulation.

The spray solutions were prepared with distilled water by adjusting their hardness with calcium carbonate (CaCO<sub>3</sub>) at the four levels proposed in the test (70 ppm – 70 mg CaCO<sub>3</sub> L<sup>-1</sup>, 110 ppm – 110 mg CaCO<sub>3</sub> L<sup>-1</sup>, 230 ppm – 230 mg CaCO<sub>3</sub> L<sup>-1</sup>, and 430 ppm – 430 mg CaCO<sub>3</sub> L<sup>-1</sup>). Subsequently, the pH was regulated at each hardness level using phosphoric acid (H<sub>3</sub>PO<sub>4</sub> – 85% pure) and sodium hydroxide (NaOH – 95% pure) at a concentration of 0.5 M for both solutions and, finally, the herbicides were added at each pH level.

The pH levels were adjusted using a pH meter (AK59<sup>®</sup>, Akso). The hardness levels were adjusted by the titration method with EDTA (ethylenediaminetetraacetic acid) and eriochrome black-T indicator from the water buffer at pH 10.

Part of the spray solution was used for its physicochemical characterization. The surface tension, viscosity, and density were analyzed according to the methodology used by Assunção et al. (2019). The surface tension was obtained using a tensiometer (K6<sup>®</sup>, KRÜSS) and the Du Nouy ring method. A micro-processed rotational viscometer (Q860M21<sup>®</sup>, Quimis) was used to determine the dynamic viscosity. The density values were obtained by measuring a fixed volume of 100 mL of the spray solution in a volumetric flask and weighing it on an analytical electronic scale.

Evaluations of weed control effectiveness were performed at 7, 14, and 21 days after application (DAA) using a visual scoring scale in the percentage of control, where 0% represented uncontrolled plots and 100% represented the total weed control (ALAM, 1974). Plots for which there was no application were taken as a reference.

The statistical analyses were performed using the software R (R Package<sup>®</sup>, Vienna). An analysis of variance was performed for the physicochemical properties and the percentage of weed control, taking each evaluation day (7, 14, and 21 DAA) as a different variable. The Tukey test at a 5% significance was performed when the analysis of variance was significant both for the herbicide factor and for the hardness and pH factors. The Dunnett test at a 5%

significance level was used for comparison with the control. Although hardness and pH were quantitative factors, the Tukey test for mean comparison was used because no regression model was adjusted the data dispersion.

## RESULTS AND DISCUSSION

### Surface tension, viscosity, and density

The surface tension, viscosity, and density were also studied in an attempt to verify possible interferences of pH and hardness in other physical and chemical properties. The surface tension varied from 40.97 to 42.38 N m<sup>-1</sup>, viscosity from 0.9525 to 1.1000 mPa s, and density from 1.0019 to 1.0025 g cm<sup>-3</sup>. No statistical differences were observed between treatments. The variation ranges were very small for the three variables to the point that no interference was observed during the field applications. It proves the suitability

of the chemicals used to change the pH and hardness characteristics, not interfering with other properties.

### Control effectiveness

#### Area 1

The hardness factor was significant at 7 DAA and the herbicide factor was significant in all evaluations of weed control (Table 1). The interactions between herbicide, hardness, and pH were not significant, indicating independence from each other. Glyphosate ammonium salt promoted better control than potassium salt, with 68.9% control at 7 DAA and over 90% control at 14 and 21 DAA, regardless of water hardness and pH. However, both formulations promoted control higher than 91% in the last evaluation, being considered excellent (ALAM, 1974). The main weed present in the area was *D. horizontalis*, with 90% infestation in the control area.

TABLE 1. Weed control effectiveness (Area 1) at 7, 14, and 21 days after application (DAA), with the use of the herbicide glyphosate ammonium salt and potassium salt as a function of water hardness and pH.

Hardness (ppm CaCO <sub>3</sub> )	pH	Control effectiveness (%)					
		7 DAA		14 DAA		21 DAA	
		Ammonium salt	Potassium salt	Ammonium salt	Potassium salt	Ammonium salt	Potassium salt
70	3.5	60.0 *	55.0 *	91.2 *	92.0 *	97.5 *	97.7 *
	4.5	65.0 *	60.0 *	95.7 *	92.0 *	98.2 *	96.7 *
	5.5	75.0 *	50.0 *	95.2 *	90.0 *	98.2 *	97.0 *
	6.5	60.0 *	50.0 *	92.0 *	93.2 *	97.7 *	96.0 *
110	3.5	67.5 *	57.5 *	94.2 *	93.7 *	98.0 *	97.5 *
	4.5	65.0 *	47.5 *	95.7 *	87.5 *	98.5 *	95.0 *
	5.5	57.5 *	52.5 *	96.0 *	92.5 *	98.7 *	96.5 *
	6.5	57.5 *	52.5 *	95.0 *	89.0 *	98.2 *	96.7 *
230	3.5	67.5 *	55.0 *	97.0 *	92.0 *	99.2 *	98.0 *
	4.5	62.5 *	57.5 *	92.5 *	92.5 *	98.5 *	98.0 *
	5.5	60.0 *	55.0 *	92.5 *	91.2 *	97.7 *	96.7 *
	6.5	70.0 *	57.5 *	96.5 *	93.7 *	99.5 *	96.7 *
430	3.5	60.0 *	52.5 *	93.5 *	88.7 *	97.2 *	95.5 *
	4.5	55.0 *	52.5 *	93.2 *	95.7 *	98.5 *	98.0 *
	5.5	55.0 *	47.5 *	93.7 *	87.5 *	98.7 *	94.7 *
	6.5	57.5 *	50.0 *	92.0 *	88.7 *	98.0 *	94.0 *
Control		0		0		0	
Overall mean		62.2 a	53.3 b	94.1 a	91.3 b	98.3 a	96.6 b
CV (%)		5.36		2.22		2.80	
F <sub>Hardness</sub>		4.1956**		1.0451 <sup>ns</sup>		2.0631 <sup>ns</sup>	
F <sub>pH</sub>		0.7639 <sup>ns</sup>		0.2022 <sup>ns</sup>		0.5538 <sup>ns</sup>	
F <sub>Herbicide</sub>		36.6763**		14.3828**		24.9944**	
F <sub>Hardness x pH</sub>		1.2054 <sup>ns</sup>		1.2063 <sup>ns</sup>		0.9523 <sup>ns</sup>	
F <sub>Hardness x Herbicide</sub>		0.4929 <sup>ns</sup>		0.6537 <sup>ns</sup>		0.9054 <sup>ns</sup>	
F <sub>pH x Herbicide</sub>		0.1919 <sup>ns</sup>		0.2821 <sup>ns</sup>		1.1380 <sup>ns</sup>	
F <sub>Hardness x pH x Herbicide</sub>		1.3759 <sup>ns</sup>		1.3874 <sup>ns</sup>		0.7215 <sup>ns</sup>	
F <sub>Additional x Factorial</sub>		186.8145**		1812.6051**		9560.6230**	

Means followed by different lowercase letters in the row for each evaluation differ from each other by the Tukey test at 0.05 significance. \*Treatments that differ from the control by the Dunnett test at 0.05 significance. CV (%): coefficient of variation; F<sub>Hardness</sub>: F value for hardness; F<sub>pH</sub>: F value for pH; F<sub>Herbicide</sub>: F value for herbicide; F<sub>Hardness x pH</sub>: F value for the interaction hardness x pH; F<sub>Hardness x Herbicide</sub>: F value for the interaction hardness x herbicide; F<sub>pH x Herbicide</sub>: F value for the interaction pH x herbicide; F<sub>Hardness x pH x Herbicide</sub>: F value for the interaction hardness x pH x herbicide; F<sub>Additional x Factorial</sub>: F value for the interaction of the additional x factorial. \*\*Significant at 0.05; <sup>ns</sup>not significant.

The water pH did not interfere with weed control. Ruiz & Ortiz (2005) evaluated the pH effect in the control of *Brachiaria extensa* with glyphosate and observed a reduction in its control with the increase in water alkalinity. However, the authors evaluated the pH ranging from 4 to 9, which may explain the difference compared to the present study. A similar effect was found by Dan et al. (2009), who obtained a lower control of *Brachiaria brizantha* with glyphosate spray solution prepared with water of pH between 1.3 and 9.0. According to the authors, the best control levels were obtained with pH ranging from 3.5 to 5.5.

Farias et al. (2014) analyzed the quality of water used for spraying in Rio Grande do Sul and found that most of the collected samples had a pH between 6 and 7. Although there is no similar work at a national level, most of the waters in the Brazilian territory is believed to have a pH close to neutrality or slightly acidic, which favors the application as seen in the present study. However, the use of the herbicide glyphosate in a mixture with other products can change the pH out of the range tested in this study (3.5 to 6.5), which can lead to a change in product effectiveness.

Table 2 shows the effect of hardness on the control effectiveness at 7 DAA. The increase in hardness promoted a reduction in control. The hardness of 430 ppm CaCO<sub>3</sub> caused the least control effectiveness, although similar to 110 ppm. This effect was not observed at 14 and 21 DAA. Thus, although the hardness did not compromise the treatment at the end of the evaluated period, the difference found at 7 DAA suggests interference in glyphosate activity, which could be verified in plants that are difficult to control, which did not occur in the evaluated area, or with water of higher hardness (>430 ppm CaCO<sub>3</sub>).

TABLE 2. Weed control effectiveness at 7 days after application (DAA) as a function of water hardness (Area 1).

Hardness (ppm CaCO <sub>3</sub> )	Control effectiveness (%)
70	59.4 A
110	57.2 AB
230	60.6 A
430	53.7 B

Means followed by different letters differ from each other by the Tukey test at 0.05 significance.

The presence of ions such as Ca<sup>2+</sup>, Fe<sup>3+</sup>, Mn<sup>2+</sup>, Zn<sup>2+</sup>, and Cu<sup>2+</sup> can interfere with the effectiveness of glyphosate, as this herbicide can complex these ions, making it more difficult to act on the plant (Dan et al., 2009). Complexes formed between glyphosate and cations form salts that are not so easily absorbed.

Mueller et al. (2006) evaluated the effectiveness of glyphosate in three formulations (isopropylamine salt, diammonium salt, and potassium salt) at different hardness levels and observed a reduction in effectiveness from 500 ppm of calcium. The authors evaluated *Urochloa platyphylla*, *Ipomoea lacunosa*, *Amaranthus palmeri*, and *Cyperus esculentus*. On the other hand, Soltani et al. (2011) observe no differences in the control of various monocot and dicot weeds using water with different degrees of hardness (0, 353, and 1,799 ppm). This divergence of results could also be observed in the study of Manuchehri et al. (2018). The authors found that the water source, with hardness ranging from 91 to 1,046 ppm, affected weed control (*Triticum aestivum* and *A. palmeri*) with glyphosate in seven out of ten performed evaluations.

Mirzaei et al. (2019) studied the effect of hard water on different weed species and suggested that this effect is species-dependent. Apparently, the interference is more pronounced in dicot plants than in monocot plants. Thus, the predominance of *D. horizontalis* should be taken into account in the present study, which may have contributed to the more restricted effect of water hardness.

**Area 2**

The results of control effectiveness in Area 2 are shown in Table 3. There is a behavior similar to Area 1. Weed control showed no difference only as a function of hardness. The ammonium salt promoted higher control than the potassium salt, with no interference of the pH and hardness, confirming the previous results. However, the third evaluation showed that the formulations promoted excellent control, according to the ALAM scale (ALAM, 1974). The surfactants presented differences in the two formulations, which may have generated a slower effect of potassium salt.

Area 2 also presented *D. horizontalis* as the main weed, but with the presence of *E. indica*, *C. echinatus*, and *I. indivisa*, which showed similar behavior to *D. horizontalis* in the three evaluations.

TABLE 3. Weed control effectiveness (Area 2) at 7, 14, and 21 days after application (DAA), with the use of the herbicide glyphosate ammonium salt and potassium salt as a function of water hardness and pH.

Hardness (ppm CaCO <sub>3</sub> )	pH	Control effectiveness (%)					
		7 DAA		14 DAA		21 DAA	
		Ammonium salt	Potassium salt	Ammonium salt	Potassium salt	Ammonium salt	Potassium salt
70	3.5	67.5 *	67.5 *	90.0 *	88.7 *	94.5 *	94.5 *
	4.5	75.0 *	60.0 *	91.2 *	88.7 *	93.2 *	93.7 *
	5.5	62.5 *	62.5 *	88.7 *	85.0 *	95.7 *	91.2 *
	6.5	67.5 *	57.5 *	90.0 *	88.7 *	95.7 *	93.7 *
110	3.5	62.5 *	57.5 *	92.0 *	85.0 *	95.2 *	91.2 *
	4.5	72.5 *	62.5 *	90.0 *	86.2 *	96.5 *	93.7 *
	5.5	70.0 *	60.0 *	91.2 *	86.2 *	94.5 *	92.5 *
	6.5	70.0 *	57.5 *	92.5 *	86.2 *	97.2 *	92.5 *
230	3.5	72.5 *	62.5 *	92.5 *	85.0 *	95.7 *	92.5 *
	4.5	72.5 *	67.5 *	91.2 *	90.0 *	96.5 *	93.2 *
	5.5	65.0 *	55.0 *	91.2 *	82.5 *	94.5 *	87.5 *
	6.5	67.5 *	57.5 *	88.7 *	87.5 *	95.7 *	94.5 *
430	3.5	60.0 *	52.5 *	88.7 *	83.7 *	95.0 *	90.0 *
	4.5	70.0 *	62.5 *	91.2 *	86.2 *	96.5 *	90.0 *
	5.5	72.5 *	57.5 *	91.2 *	86.2 *	93.7 *	92.5 *
	6.5	75.0 *	62.5 *	90.7 *	88.7 *	94.5 *	95.0 *
Control		0		0		0	
Overall mean		68.9 a	60.2 b	90.7 a	86.6 b	95.3 a	92.4 b
CV (%)		2.26		3.08		1.55	
F <sub>Hardness</sub>		0.0763 <sup>ns</sup>		0.0790 <sup>ns</sup>		0.3380 <sup>ns</sup>	
F <sub>pH</sub>		1.3646 <sup>ns</sup>		0.9062 <sup>ns</sup>		2.2253 <sup>ns</sup>	
F <sub>Herbicide</sub>		19.9348**		28.1382**		23.7706**	
F <sub>Hardness x pH</sub>		1.0312 <sup>ns</sup>		0.6301 <sup>ns</sup>		0.6333 <sup>ns</sup>	
F <sub>Hardness x Herbicide</sub>		0.2204 <sup>ns</sup>		0.8107 <sup>ns</sup>		0.6642 <sup>ns</sup>	
F <sub>pH x Herbicide</sub>		0.3560 <sup>ns</sup>		0.8744 <sup>ns</sup>		0.4003 <sup>ns</sup>	
F <sub>Hardness x pH x Herbicide</sub>		0.3503 <sup>ns</sup>		0.4463 <sup>ns</sup>		1.2132 <sup>ns</sup>	
F <sub>Additional x Factorial</sub>		131.4260**		1551.3334**		3005.2093**	

Means followed by different lowercase letters in the row for each evaluation differ from each other by the Tukey test at 0.05 significance. \*Treatments that differ from the control by the Dunnett test at 0.05 significance. CV (%): coefficient of variation; F<sub>Hardness</sub>: F value for hardness; F<sub>pH</sub>: F value for pH; F<sub>Herbicide</sub>: F value for herbicide; F<sub>Hardness x pH</sub>: F value for the interaction hardness x pH; F<sub>Hardness x Herbicide</sub>: F value for the interaction hardness x herbicide; F<sub>pH x Herbicide</sub>: F value for the interaction pH x herbicide; F<sub>Hardness x pH x Herbicide</sub>: F value for the interaction hardness x pH x herbicide; F<sub>Additional x Factorial</sub>: F value for the interaction of the additional x factorial. \*\*Significant at 0.05; <sup>ns</sup>not significant.

Inoue et al. (2007) observed that pH values between 3.8 and 6.8 did not change the control of *Euphorbia heterophylla* using the recommended glyphosate dose (diammonium salt), reinforcing the data found in the present study. However, the authors observed that a reduction in pH improved control when using half a dose, suggesting that the spray solution acidification may assist the herbicide treatment under more extreme situations. Sobiech et al. (2020) also stated that the use of pH reducers and small water hardness can be of great importance when using reduced herbicide doses.

## CONCLUSIONS

Taking into account the predominant presence of *D. horizontalis* in the two studied areas, we can conclude that:

The water pH in the studied range from 3.5 to 6.5 showed no interference in the effectiveness of glyphosate ammonium salt and potassium salt.

The increase in water hardness reduced glyphosate effectiveness at 7 DAA, but with no difference in weed control after 21 DAA.

Glyphosate ammonium salt promoted higher control of *D. horizontalis* than the potassium salt, regardless of the water pH and hardness.

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