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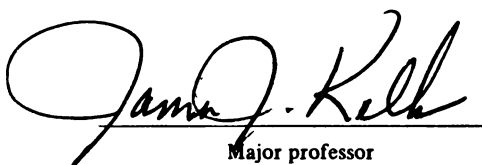
SUBSTITUTES FOR AMMONIUM SULFATE
AS ADDITIVES
WITH GLYPHOSATE AND GLUFOSINATE

presented by

David Vernon Pratt

has been accepted towards fulfillment
of the requirements for

M.S. degree in Crop and Soil Sciences


Major professor

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SUBSTITUTES FOR AMMONIUM SULFATE AS ADDITIVES WITH
GLYPHOSATE AND GLUFOSINATE.

By

David Vernon Pratt

A Thesis

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

Department of Crop and Soil Sciences

2002

Abstract

SUBSTITUTES FOR AMMONIUM SULFATE AS ADDITIVES WITH GLYPHOSATE AND GLUFOSINATE.

By

David Vernon Pratt

Glyphosate and glufosinate are now options for postemergence weed control in herbicide resistant corn and soybeans. Velvetleaf is one of the more difficult to control annual weeds to control with these herbicides at the commonly used rates. Ammonium sulfate is generally used with these herbicides to overcome hard water antagonism and increase herbicide activity. Greenhouse and field trials were conducted with commercial adjuvants that might substitute for ammonium sulfate. The adjuvants were evaluated in de-ionized water, tap water, and de-ionized water containing 500 ppm CaCO_3 . In the absence of ammonium sulfate, hard water reduced velvetleaf control with both herbicides. Regardless of water source, ammonium sulfate increased velvetleaf control with both glyphosate and glufosinate. Several adjuvants increased velvetleaf control with either herbicide, however, none were superior to 2% ammonium sulfate. Other adjuvants decreased velvetleaf control with either herbicide.

ACKNOWLEDGEMENTS

I want to thank my graduate committee for all their support and patients. I certainly was not a normal fit into a graduate program with four boys and a bride of 17 years. My committee consisted of: Major Professor Dr. Jim Kells, Dr Don Penner (Doc) and Dr Chris DiFonzo. I have had the honor to work with both Dr. Kells and Dr. DiFonzo for many years as an extension agent. They both relate very well to their students, colleagues and clientele in the field. Their practical experience and down to earth approach has put them among the very best in their field. My contact with Doc goes clear back to my undergraduate years some 20 years ago when I did a special project testing adjuvants in the greenhouse. He found me a summer job with a chemical company of which I very much appreciated. You all have taught me much more than you will ever know. Thanks for all the great professional advise throughout the degree process and my career.

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Chapter 1

Introduction

The war against unwanted plants, also known as weeds, in commercial production agriculture continues to be a challenge. The advent of herbicide resistant crops has dramatically changed the way weeds are controlled in growing crops. Two of the most popular herbicides using herbicide resistant crop technology are glyphosate and glufosinate.

Glufosinate is labeled for Liberty Link Corn (Bertges et al. 1994). Glufosinate inhibits glutamine synthetase in plants. This enzyme catalyzes the assimilation of ammonia by glutamate to form glutamine. Inhibition of this enzyme results in rapid accumulation of ammonia at toxic levels within the cell (Mersey et al 1990). The accumulation of ammonia depletes plant cells of crucial amino acids (Krieg et al. 1990). Due to the rapid phytotoxicity in plant cells, little translocation of glufosinate out of the treated leaf was reported by Steckel et al. (1977). As a result of the rapid cell breakdown, glufosinate is most effective on annual weeds (Pline et al.1999). Although glufosinate is a broad-spectrum herbicide not all weed species show the same degree of sensitivity (Mersey et al. 1990; Ridley and McNally 1985).

Glyphosate is labeled for use on Roundup Ready corn and soybeans. Glyphosate is a broad-spectrum herbicide that is effective on both annual and perennial plants. It is readily translocated in plants. Glyphosate is an inhibitor of EPSP synthase in the shikimate pathway (Singer and McDaniel 1985). The inhibition of EPSP synthase prevents the production of the amino acids tyrosine, tryptophan, and phenylalanine which results in death of the plant.

Although glyphosate and glufosinate are very effective in controlling most weeds, there are some that are more difficult to control than others. It is apparent through field observation and research that both of these herbicides are inconsistent in controlling certain weed species, in particular velvetleaf (*Abutilon theophrasti*). In Michigan during the 1998 growing season, velvetleaf often survived an application of glyphosate. Ammonium sulfate (AMS) was not included in the spray solution at many field sites.

AMS increases the phytotoxicity of several herbicides, including glyphosate (Blair 1975; Nalewaja and Matysiak 1993; O'Sullivan *et al.* 1981 Suwunnamek and Parker 1975; Turner and Loader 1975). Thelen, et al (1995) reported that the glyphosate molecule reacts with Ca^{++} and other cations to form a less easily absorbed Ca- glyphosate salt. Thelen *et al.* (1995) indicated that the addition of AMS allowed NH_4 to bind with the glyphosate molecule, preventing the formation of the Ca-salt of glyphosate. This resulted in greater absorption of glyphosate into the leaves.

Hall et al. (2000) reported that some plants, including velvetleaf, contain Ca cations within and on the leaf tissue. Thelen et al. (1995) found that the addition of AMS to a Glyphosate treatment using de-ionized water as the carrier resulted in greater absorption of glyphosate due to the prevention of the formation of the Ca-salt of glyphosate.

Thelen et al. (1995) also concluded that the conjugate sulfate ion from the AMS removes free Ca^{++} from solution by forming CaSO_4 thus allowing NH_4 to form the readily absorbed NH_4 -glyphosate salt.

Pline et al. (1999) reported that the addition of 5 % AMS to glufosinate greatly increased the foliar absorption in some weed species. The role of AMS in increasing the activity of glufosinate has not been reported. However, due to similarities in the structure

of the glufosinate and glyphosate molecules, it is widely believed that glufosinate may be antagonized by cations in the herbicide carrier, or on the leaf surface itself.

The objectives of this study were to: 1) determine if water hardness has an effect on the activity of glyphosate and glufosinate, and 2) determine if commercial adjuvants can effectively replace AMS as an adjuvant with glyphosate or glufosinate for velvetleaf control.

Materials and Methods

Greenhouse Study.

Velvetleaf seed was planted into greenhouse potting soil in plastic pots containing drain holes. Fifteen velvetleaf seeds were planted into the potting soil. After the seeds were planted, the pots were placed on benches in the greenhouse and watered with tap water until water ran from the drain holes. The plants were watered frequently to prevent moisture stress. The temperature in the greenhouse was maintained at 25 ± 2 C. Natural sunlight was supplemented with sodium vapor lighting that provided a total mid day light intensity of $1000 \mu\text{mol m}^{-2} \text{s}^{-1}$. The plants were thinned to 2 per pot when they reached 4 cm in height. The plants were thinned based on uniform growth stage while maintaining as much space as possible between the remaining plants. When the plants reached 6 cm the pots were thinned to one plant per pot. When the plants reached 8 cm the soil was treated with 0.1 g of water-soluble fertilizer solution (20%N, 20% P_2O_5 , 20% K_2O) to prevent any nutrient deficiencies.

The timing of treatment was based on the height of the plants. One hundred and twenty plants were selected from the original 250 pots based on a uniform plant height. For the first run of the study the plants were 25-26 cm tall. The plants were 19-20 cm tall for the second run. The adjuvants were added to the carrier solutions prior to herbicide application. This allowed the adjuvant time react with the cations in the spray solution. Herbicide rates were selected to provide incomplete velvetleaf control to aid in observing adjuvant effects. The herbicide rates were .28 kg/ha for glyphosate and .27 kg/ha for glufosinate. The treatments were mixed 30 seconds by hand agitation after each component was added. The sprayer used for application was a continuous link belt

sprayer, which delivered 57 L/ha, using Tee jet 80005 nozzle, at 207 kPa. The experiment was conducted as a two factor completely randomized design. The two factors were carrier solution and adjuvant. The carriers were de-ionized water, tap water (427 ppm Ca CO₃ and 0.5 ppm Fe) and de-ionized water containing 500 ppm of calcium. The stock solution was continuously stirred to keep the un-dissolved CaCO₃ in suspension.

A total of eight adjuvants were examined (Table 1). Separate experiments were conducted using glyphosate and glufosinate. Each experiment was conducted twice with 4 replications each. Plant injury was evaluated 7 and 14 days after treatment. Only the results from the 14-day data will be reported due to similarity between the 7 and 14-day results. The effectiveness of the treatment was evaluated using visual ratings on a scale of 0 - 10 with 0 = no visible injury and 10 = death of the plant.

Field study.

Field experiments were conducted at the Michigan State University Research Farm, East Lansing MI. The site was selected based on a previous history of high velvetleaf density.

The experimental design was a randomized complete block with 4 replications. One study compared adjuvants with glyphosate. A second study compared adjuvants with glufosinate. The plots were 9.1 m long and 3 m wide. Sethoxydim, a grass herbicide, was applied June 18, 2000, one week prior to the experiment treatments, to remove grass weeds that may have interfered with treatment coverage

All herbicide applications were made with de-ionized water plus 500 ppm CaCO₃. The herbicide rates were .38 kg/ha for glyphosate and .36 kg/ha for glufosinate. The

treatments were applied with a tractor mounted compressed air sprayer at a rate of 76 L/ha. Spray pressure was maintained at 207kPa using Tee Jet 8002 nozzle. Applications were made on June 27, 2000 between 9:30 – 10:30 am under favorable conditions for herbicide effectiveness. Herbicide application timing was based on 20 cm velvetleaf.

The plots were evaluated visually at 7 and 14 days after treatment, on a scale of 0-10 with 0 = no damage and 10 = death of all plants in the plot. Due to similarities in data between the two evaluations, only the 14 day evaluation is reported.

Results and Discussion

Herbicide carrier.

The addition of AMS significantly increased velvetleaf control with glyphosate and glufosinate (Figures 1 and 2). The addition of AMS not only increased the level of control when hard water cations were present in the carrier solution, but also in the de-ionized water. This indicates that the AMS has more impact on the control of velvetleaf than just neutralizing hard water cations within the solution. These results support findings of Hall et al. (2000), that Ca cations present on the leaf tissue of velvetleaf form a glyphosate Ca salt that is less easily absorbed. Regardless the amount of hard water cations in the spray solution, the cations on the leaf surface were having a negative effect on velvetleaf control with glyphosate and glufosinate. The addition of AMS is necessary for the control of velvetleaf regardless of the level of hard water cations present in the carrier solution.

Glyphosate.

In the greenhouse study, both the 1% and 2% AMS enhanced velvet leaf control with glyphosate (Figure 3). Some of the adjuvants were equal to the AMS treatments with certain carriers, none were consistently effective in all three carrier solutions (Figure 3). In some cases, the adjuvants resulted in velvetleaf control less than glyphosate with no adjuvants added, indicating an antagonistic interaction with the herbicide (Figure 3). While the addition of some adjuvants enhanced the effectiveness of glyphosate other adjuvants had no positive effect when compared to glyphosate with no additive in controlling velvetleaf (Figure 3).

The field study with glyphosate supports the results obtained in the greenhouse showing that all the adjuvants tested were significantly less effective than the 2% AMS treatment, except Class Act the Next Generation (Figure 3). The 1% AMS treatment was significantly less effective than the 2% AMS in the field study.

These results demonstrate that although some of the adjuvants performed well in one or more of the carrier solutions, only the 2% AMS treatment consistently provided maximum velvetleaf control with glyphosate throughout the entire study (Figure 3). These results also show that glyphosate without an adjuvant consistently performed very poorly and the addition of 2% AMS greatly enhanced the control of velvetleaf.

Glufosinate.

The greenhouse study using three carrier solutions with different levels of hard water cations showed varying results for the different adjuvants (Figure 4). Two of the adjuvants, Class Act Next Generation and CL 9913, performed similar to 2% AMS in all

three-carrier solutions. Some adjuvants enhanced the effectiveness of glufosinate with one or more of the carriers equal to the 2% AMS, but were not consistent, while others were only equal to the glufosinate treatment with no adjuvants added (Figure 4).

In the field study, three of the adjuvants performed at the same level as the 2% AMS (Figure 4). Only Class Act Next Generation was consistent in both the field and greenhouse studies (Figure 4). The CL 9913 was not tested in the field. Glufosinate with 2% AMS provided consistent velvetleaf control in all studies while glufosinate with no adjuvant performed very poorly (Figure 4). These results indicate that 2% AMS and Class Act Next Generation were the only two adjuvants that were consistently effective with glufosinate for velvetleaf control.

The amount of NH_4 concentration was quantified for each of the treatment solutions to determine if there was a relationship between the amount of NH_4 present and velvetleaf control for each treatment (Table 1). These data indicate that the treatments containing the highest levels of NH_4 also performed the best with both glyphosate and glufosinate for velvetleaf control. The treatments that contained the least amount of NH_4 were consistent in their poor performance. Therefore, it appears that the amount of NH_4 present in the adjuvant is critical in the performance of glyphosate and glufosinate. The equivalent of 2% AMS was the most consistent throughout the entire study with both glyphosate and glufosinate.

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Table 1: Adjuvants Used with Glyphosate and Glufosinate .

<u>Adjuvant</u>	<u>Evaluated Rate</u>	<u>Manufacturer Maximum Recommended Rate</u>	<u>Lab Test Results for NH₄ mg N/L</u>	<u>Rate Required to Provide 2% AMS Equivalent ^a</u>
Choice	1% v/v	0.75% v/v	415	9% v/v
Ultra Guard	1% v/v	0.5% v/v	184	20% v/v
Class Act New Generation	2.5% v/v	5% v/v	2449	5% v/v
CL 9913	2.5% v/v	-----	2003	5% v/v
Cayuse Plus	1.2% v/v	.75% v/v	938	5% v/v
Dryve	1% w/v	2% w/v	ND	2% w/v
AMS	1% w/v	-----	2260	-----
<u>AMS</u>	<u>2% w/v</u>	<u>-----</u>	<u>4344</u>	<u>-----</u>

^a Numbers are based on analysis of ammonium nitrogen content of each solution as it was applied

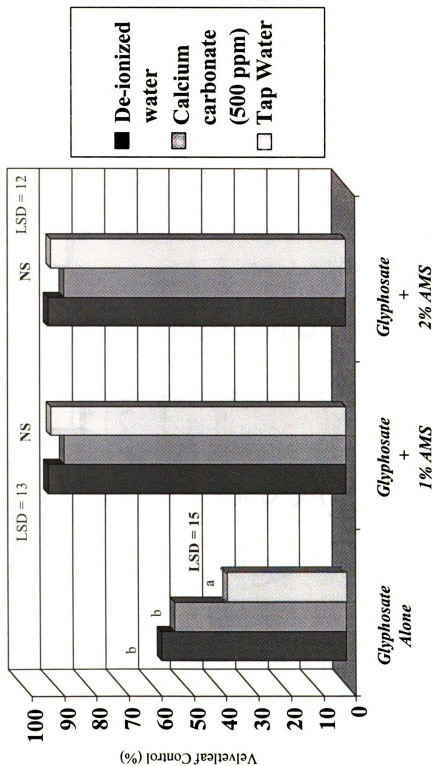


Figure 1. Effect of water hardness and ammonium sulfate on velvetleaf control with glyphosate at 0.28 kg ae/ha in the greenhouse.

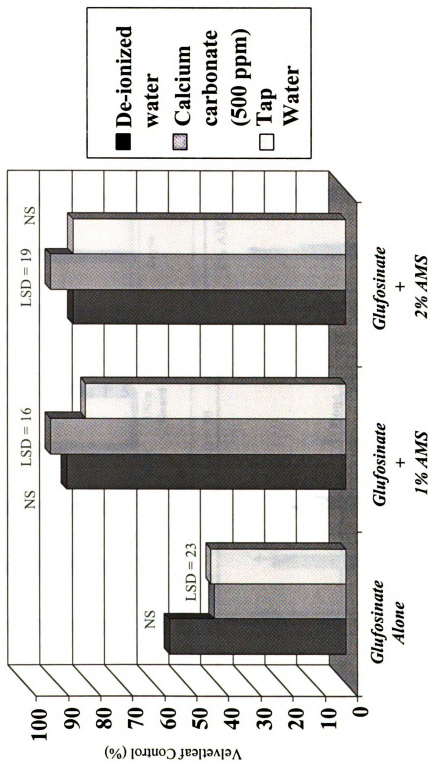


Figure 2. Effect of water hardness and AMS on velvetleaf control with glufosinate at 0.27 kg ai/ha in the greenhouse.

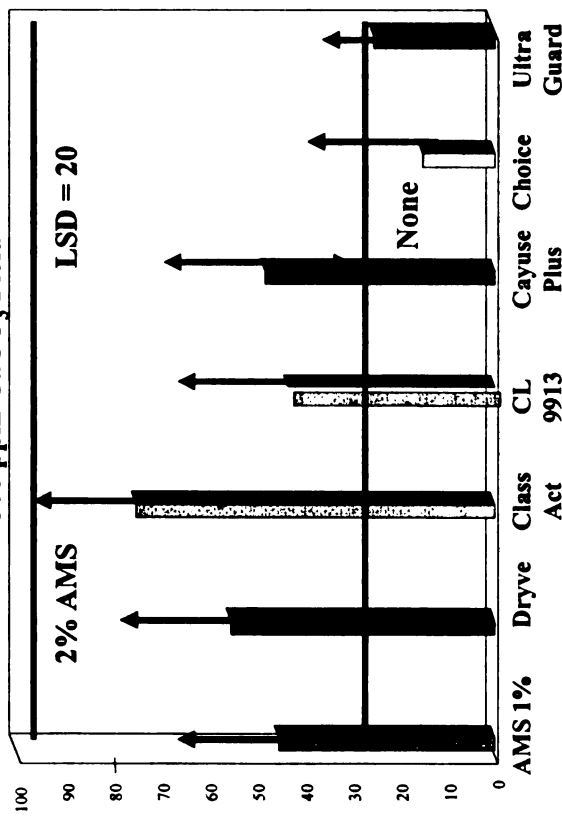
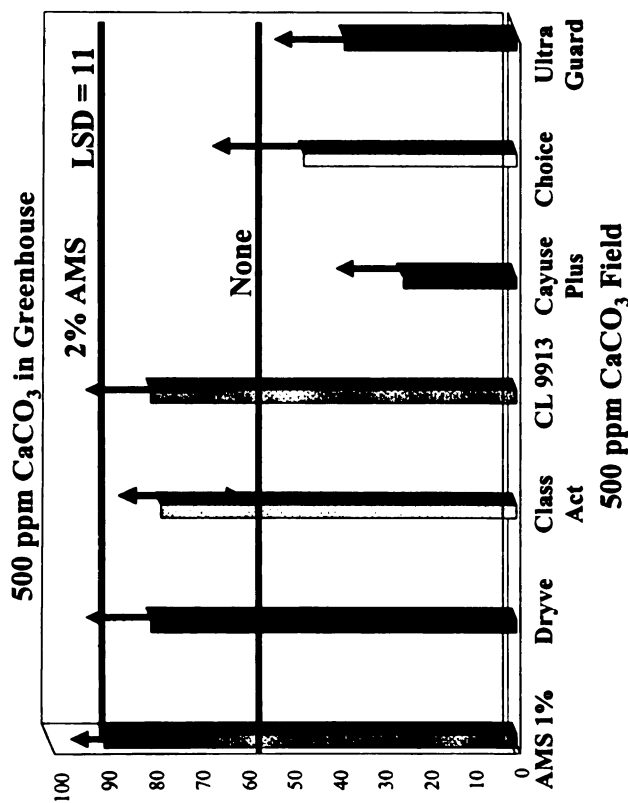
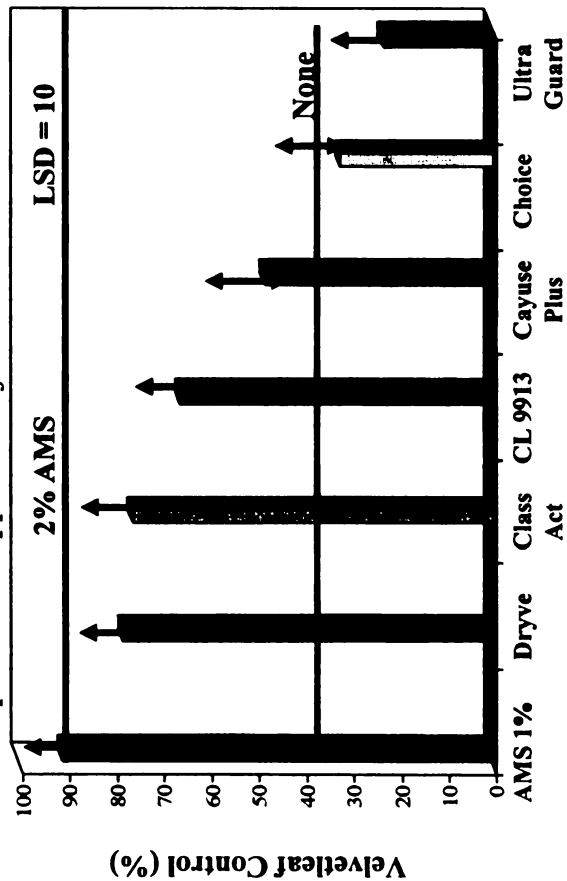
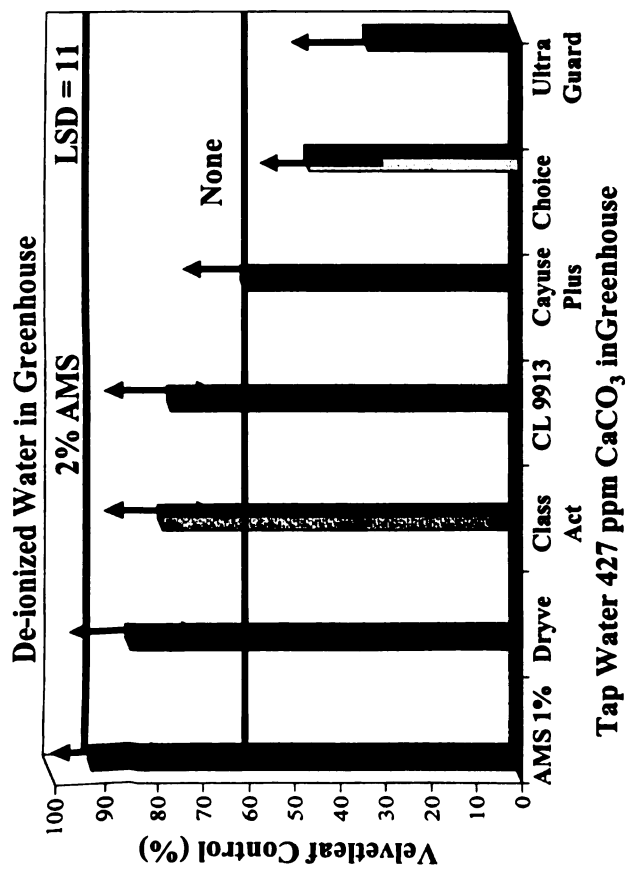


Figure 3. Effect of adjuvants on velvetleaf control with glyphosate at 0.28 kg ae/ha. Water source and study site are identified at the top of each graph

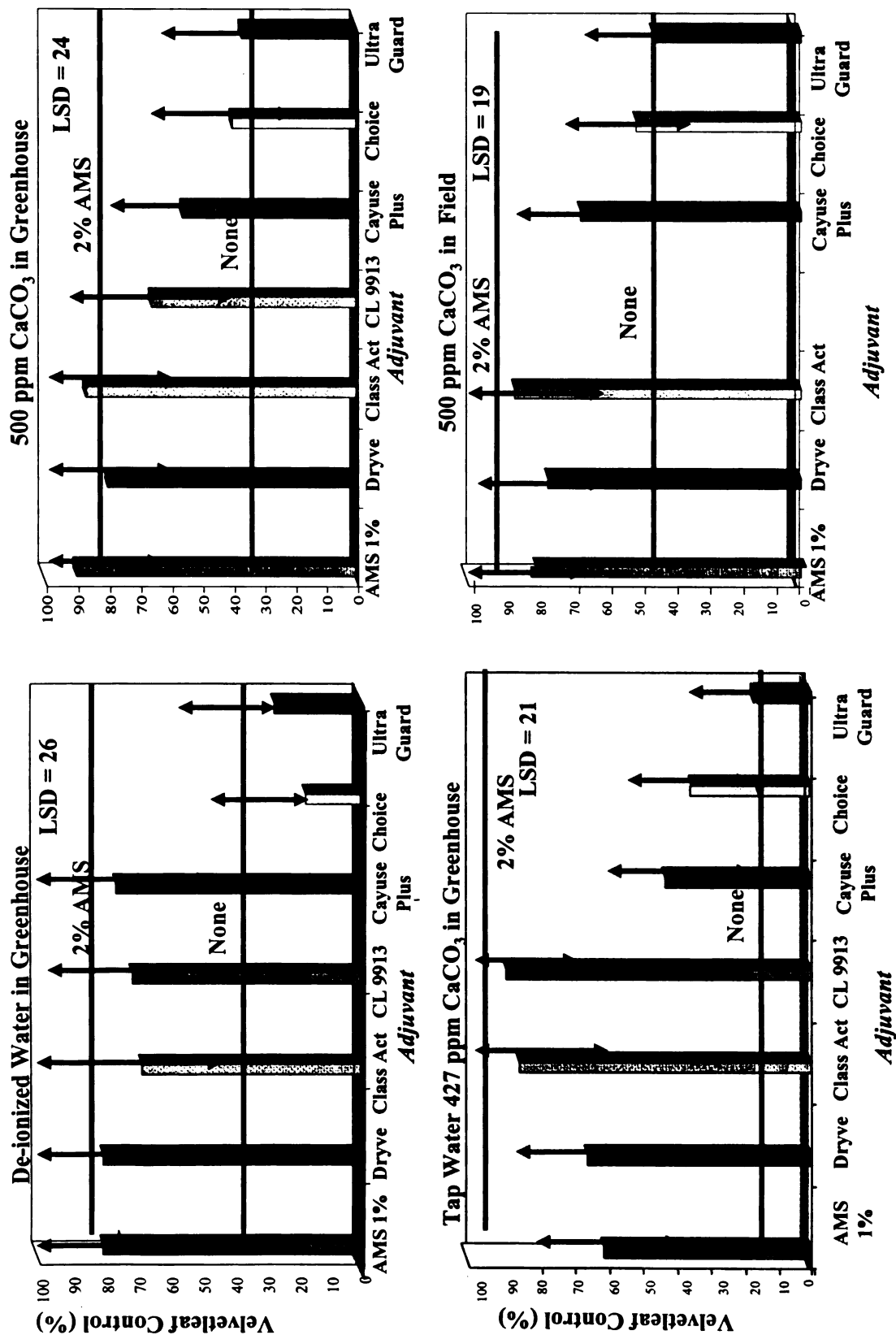


Figure 4. Effect of adjuvants on velvetleaf control with glufosinate at 0.27 kg ai/ha. Water source and study site are identified at the top of each graph

Chapter 2

Adjuvants: What, When, Which, and Why? Questions and Answers

With the advancements in technology both herbicides and adjuvants are increasingly more weed specific. Therefore, when considering a weed control program in which an adjuvant is to be used, it is important to consider the herbicide, the crop, and weeds to be controlled.

What:

Definition of an Adjuvant:

The Weed Science Society of America defines an adjuvant as “any substance in an herbicide formulation or added to the spray tank to modify the herbicide activity or application characteristics.” Adjuvants fall into three categories, either Activator, Spray Modifiers, or Utility Adjuvants.

Activator Adjuvants:

These adjuvants increase herbicide activity by one or more of the following:

- Reduce spray surface tension
 - Ball shaped spray droplets can roll off the leaf and give minimal leaf contact. Adjuvants can change the shape of the droplets to oval or flat reducing run- off and increasing contact area. (Figure 9).
- Make the plant cuticle more soluble
 - A cuticle is made up of a waxy substance that acts as a barrier to herbicides entering into a plant. Depending on the plant species, some adjuvants make the cuticle more soluble.

- Increase drying time
 - Herbicides are more readily absorbed when they are in a liquid solution on the leaf surface. Adjuvants increase the time required for the solution to dry, increasing herbicide uptake.
- Increase spray retention
 - Water based spray solutions are generally repelled by the plant leaf surface. The adjuvant will cause the spray solution to become attracted to the leaf surface rather than repelled.
- Protect the active ingredient from rapid degradation
 - Some herbicides are rapidly converted to different molecules in the environment. These changes can reduce the absorption or activity of the herbicide. Some adjuvants can protect the herbicide molecule from deactivation.
- Increase rainfastness
 - Rainfall too soon after an herbicide application can wash the herbicide from the plant tissue reducing its effectiveness. Adjuvants can reduce the length of time required for a plant to absorb the herbicide, therefore reducing the required rain free period.

Common Activator Adjuvants:

Surfactants (derived from the words “surface-active agent”) These compounds affect how a spray solution reacts on the surface of the plant leaf. The terms adjuvant and surfactant

are often used interchangeably but, in fact, a surfactant is a type of adjuvant. Surfactants are a very large percentage of adjuvants used in agriculture. Surfactants can act as wetting agents to reduce surface tension, which prevents water droplets from beading up on the plant tissue. Surfactants are also used as emulsifiers that keep liquids in solution that normally would separate, such as oil and water.

Nitrogen fertilizers, particularly urea-ammonium nitrate (28% nitrogen) and ammonium sulfate (AMS) are commonly used as activator adjuvants. Cations, such as calcium, iron or magnesium, are commonly found in groundwater (Figure 10). These cations attach themselves to some commonly used herbicide molecules. Glyphosate is an herbicide impacted by these cations. Cations bind to the glyphosate molecule, forming a Ca-glyphosate salt that is poorly absorbed into plant tissue, therefore reducing the activity of the herbicide (Figure 11). Cations are not only present in groundwater, but may be present on the leaf surface of the target weed. The cations in leaf tissue also can attach to the herbicide molecule reducing its effectiveness. The ammonium portion of AMS binds to the glyphosate molecule in the same manner as the cations (Figure 12). The ammonium salt of glyphosate is readily absorbed into plant leaves. Therefore, AMS added to the tank before glyphosate often improves weed control. The sulfur portion of AMS binds to cations so they can no longer interfere with the herbicide (Figure 12). Studies have shown that AMS at the rate of 2% of total volume will give the most consistent performance.

Crop Oil Concentrates (COC) reduce surface tension and make the leaf cuticle more soluble. They also increase spray retention and drying time.

Spray Modifiers:

These adjuvants alter physical characteristics of the spray.

- Stickers cause the spray solution to more strongly adhere to the foliage
- Thickeners increase particle size to reduce drift

Utility Adjuvants:

These adjuvants widen the range of conditions in which an herbicide can be used.

- De-foaming agents reduce or eliminate the amount of foaming in the tank
- Buffering agents are used to adjust pH to desired levels
- Compatibility Agents aid in the mixing of two compounds that, on their own, do not readily mix.

When:

Although some adjuvants have been promoted for use with soil-applied herbicides, university research has concluded that adjuvants improve herbicidal activity only with foliar (postemergence) applications.

Some herbicides require the addition of adjuvants for all applications, while other herbicides only require adjuvants during adverse growing conditions such as drought.

The herbicide label contains detailed information on adjuvant use.

Which:*How to select the proper adjuvant:*

Selecting an adjuvant can be very challenging simply because there is often little information available such as the contents and their percentage. Because there is limited regulation of adjuvants the manufacturer can change the contents at any time. This makes it difficult to make recommendations for specific adjuvants. A product that is effective this year may not be effective next year if the adjuvant contents are changed. Research has shown that the wrong adjuvant can cause unintended damage to the crop, or can actually reduce herbicide effectiveness. No adjuvant does everything. Some herbicide formulations already contain adjuvants and any additional will either waste money or reduce effectiveness. To choose a proper adjuvant if one is required consult the herbicide manufacturer's label. If the herbicide manufacturer's label is not clear on which adjuvant to use, then consulting with a representative from the herbicide manufacturer may be the best option. Other good sources of information are third party consultants or universities with strong agricultural research and education programs.

Considerations when selecting an adjuvant:

- Crop and growth stage
- Weed species and size
- Herbicide/s
- Environmental conditions
- Cost

Recommended Mixing Order:

1. Add 50% of carrier
2. Add recommended adjuvant
3. Add herbicide or herbicides
4. Add remaining amount of carrier

Why:

Why is choosing the correct adjuvant so important?

A study was conducted at Michigan State University to determine the effect of adjuvants on velvetleaf control with Roundup Ultra™ and Liberty™ herbicides. Eight adjuvants were compared in both the greenhouse and field. There were significant differences in the performance of each herbicide depending on which adjuvant was used. The addition of 2% AMS to Roundup Ultra™ and Liberty™ gave the most consistent control throughout the study. Some other adjuvants actually antagonized the herbicides and resulted in less weed control than the herbicide alone. Adjuvants that contained nitrogen fertilizer were more effective than those that did not, and the higher the concentration of nitrogen the better the effectiveness. In this study it was important that an adjuvant containing the proper concentration of nitrogen (equivalent to the concentration of 2% AMS) be used with either Roundup Ultra™ or Liberty™ herbicides for adequate velvetleaf control.

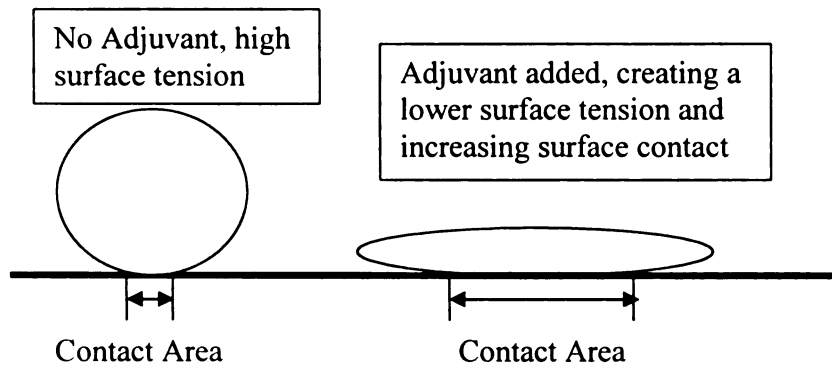


Figure 5. The shape of water droplets before and after an adjuvant is added.

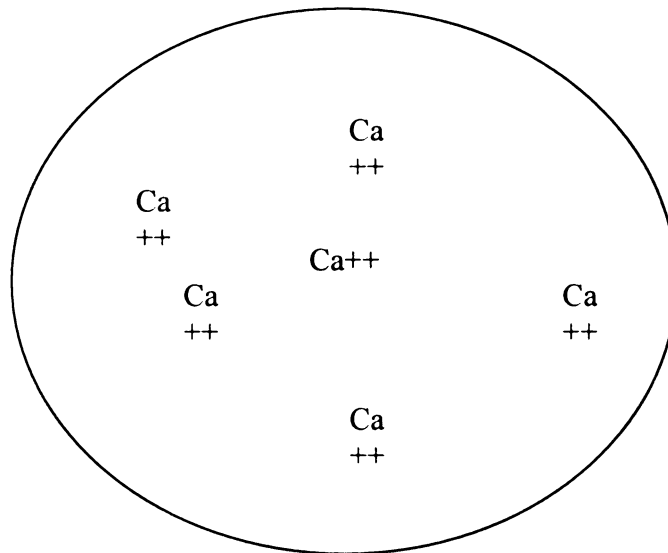


Figure 6. Hard water containing Cations

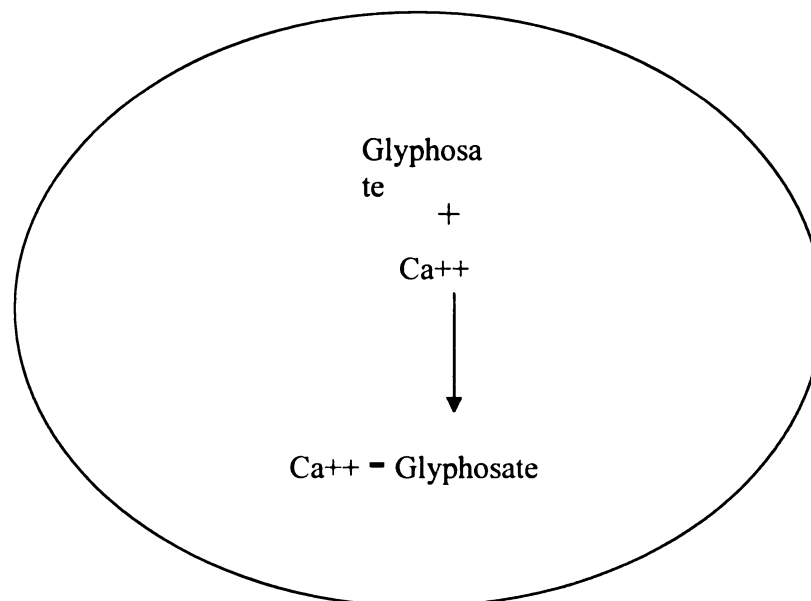


Figure 7. Without the addition of AMS, the calcium cation will bind to the glyphosate molecule, forming a less easily absorbed salt that reduces herbicide activity.

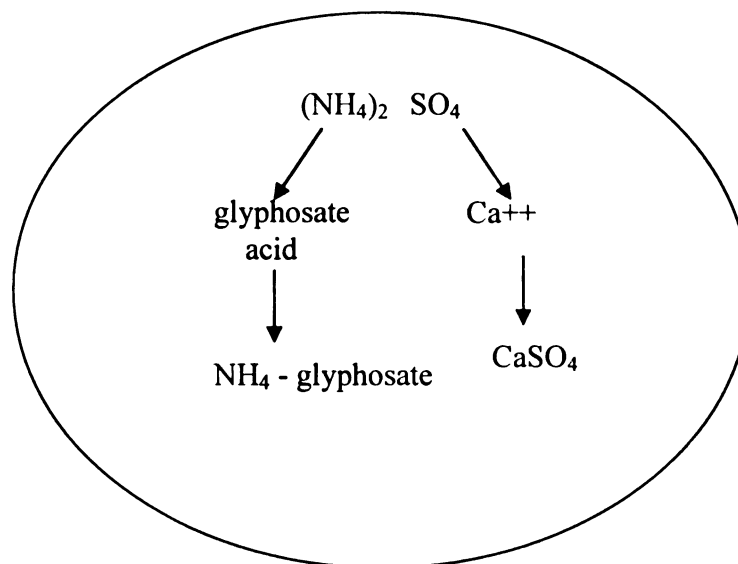


Figure 8. The addition of AMS to the hard water binds the cations so they cannot affect the glyphosate molecule. The ammonium binds to the glyphosate forming a readily absorbed ammonium salt ($\text{NH}_4 - \text{glyphosate}$)

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