

PESTICIDE FORMULATIONS

and Application Systems

**Thirteenth
Volume**

**Berger/Devisetty/Hall
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Pesticide Formulations and Application Systems: 13th Volume

*Paul D. Berger, Bala N. Devisetty, and Franklin R.
Hall, editors*

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Foreword

This publication, *Pesticide Formulations and Application Systems: 13th Volume*, contains papers presented at the symposium of the same name, held in Miami, FL on 18–19 Nov. 1992. The symposium was sponsored by ASTM Committee E-35 on Pesticides and its Subcommittee E-35.22 on Pesticide Formulations and Application Systems. Paul D. Berger of the Witco Corporation in Houston, TX; Bala N. Devisetty of Abbott Laboratories Research Center in Long Grove, IL; and Franklin R. Hall of the Ohio State University in Wooster, OH presided as symposium chairmen and are editors of the resulting publication.

Overview

The 13th Symposium on Pesticide Formulations and Application Systems was composed of 34 papers of which 30 will be reproduced in this Special Technical Publication (STP). The symposium provided a gathering place for those involved in the industry to exchange ideas and improve their understanding of various issues they are currently faced with or will be faced with in the future. We were pleased to have in attendance and on the program several foreign guests who shared their views with the audience. The subjects covered at the Symposium and in this volume involve a wide range of disciplines that will be of interest to formulators, basic pesticide manufacturers, applicators, and suppliers to the agrochemical industry. It is a compilation of the latest developments emerging from industrial and academic research, government regulatory agencies, and ASTM subcommittee task force groups. Many of the papers relate to safer and more efficient uses of pesticides. This is in keeping with the objectives of ASTM Committee E-35 on Pesticides and its Subcommittee E35.22 on Pesticide Formulations and Application Systems who were the sponsors and organizers of the symposium and the 12 others that preceded it.

Several of the papers presented at the 13th Symposium and not included in this volume involved various aspects of government regulations. Dr. John McCarthy, Vice President of Global Scientific and Regulatory Affairs for the National Agricultural Chemicals Association discussed the impact of the European Common Market on technology and regulations in his keynote address to the more than 275 attendees. Dr. McCarthy described the attempt of the various European nations to form uniform standards pertaining to the regulation of pesticides and inerts. Inerts are to be redefined as coformulants or adjuvants. Synergists and safeners will be considered active ingredients and subject to a more stringent regulatory and approval process.

Dr. David L. Schertz, Chief Agronomist for the U.S. Department of Agriculture, Soil Conservation Service, described the impact of conservation compliance and crop residue management. Dr. Schertz explained how new government policies on soil conservation, most notably no-till practices, will effect the way pre-emergent formulations are developed and applied to the soil. He cited several examples of dramatic improvement in soil erosion control, moisture loss control, and soil quality improvement brought about by crop residue management.

Dr. James S. Namnath spoke of fire safety of emulsifiable liquids. He addressed methods of evaluating solvents for potential fire hazards and the interpretation of flash point data by different regulatory agencies. Also, the economic impact of various new packaging, transportation, and warehousing rules were discussed.

This volume will be divided into four main sections: (1) Formulation Technology, (2) Spray Droplet Characteristics and Application Technology, (3) Water Dispersible Granules, and (4) Controlled Release Technology.

Formulation Technology

This section includes papers on the properties and advantages of various ingredients and methods of optimizing and evaluating formulations. Kassebaum describes a means of obtaining low-foaming glyphosate formulations using acetylenic surfactants as defoamers. Leung

and Webster describe a field trial using two adjuvants to determine rain-washing characteristics of glyphosate formulations. Rebmann and Fiquet describe a means of studying and optimizing pyrethroid microemulsions using ternary phase diagrams. Bothast, Schisler, Jackson, VanCauwenberge, and Slininger present a method they developed to improve the storage and efficacy upon spraying biocontrol agents using nutrient amendments and pregelatinized starch. Narayanan, Singh, and Chaudhuri show how copolymers of vinyl pyrrolidone and methyl vinyl ether maleic acid esters can be used in formulations to reduce the leaching of herbicides such as atrazine, dicamba, and metolachlor into soil. Hazen re-introduces guar gum as an ingredient to be considered for the control of formulation rheology. In another study involving rheology, Dexter and Szamosi discuss the relation of the pourability of suspension concentrates to their viscoelasticity. The rinsing properties of several systems are also considered with implications to container disposal. Memula, Jimenez, and Berger discuss their studies on the effect of polyethylene glycol on the adsorption of large block copolymers at the surfaces of suspended solids and the implications of this phenomena on the stability of concentrated suspensions. Skelton shows how fatty acid methyl esters can be used to produce high active microemulsion concentrate formulations and also how these esters can replace some or all of the solvent that would normally be used in these formulations. Sandler and Verbelen discuss the effect of various commonly used agricultural solvents on container material. This work serves as a guide to selecting the proper solvent/container pair for optimum performance, stability, and safety. Becher uses statistical methods to design experiments aimed at optimizing the formulation of a dry herbicide.

Spray Droplet Characteristics and Application Technology

This section includes papers on the selection of the proper equipment for spray application and the fate of droplets in-flight and after impinging on their target. Several studies relating the effect of surfactants on droplet characteristics are also described. Berger and Berger have developed a method of measuring droplet sizes and interfacial tensions for oil/water systems under dynamic conditions. Their paper describes how surfactants and mixing order effects droplet size. Ozkan, Reichard, Zhu, and Akerman evaluate the effect of drift-retardant compounds on droplet size, drift, and spray patterns. Their work is of use in evaluating the importance of these new adjuvants and selecting the one that is most effective. Hall, Thacker, and Downer discuss the effect of in-flight evaporation, equilibrium, and dynamic surface tension for a series of adjuvant solutions on their spreading characteristics. Downer, Hall, Escallon, and Chapple look into the effect of various concentrations of insecticide in oil on their atomization properties using electrostatic atomization. Salyani reports on his study involving the degradation of fluorescent dyes used to monitor spray applications. Sundaram, Leung, and Devisetty report on the rain fastness on various *Bacillus thuringiensis* formulations deposited on conifer foliage. Taylor, Chapple, and Hall have developed a simulation model for dose transfer of insecticides that may help improve the efficiency of application techniques by providing a better understanding of the underlying factors involved. Sanderson, Hewitt, Huddleston, Devisetty, Melchoir, and Ross have applied wind-tunnel studies on *Bacillus thuringiensis* formulations to predict droplet size spectra. Wang, Zhang, Slocombe, and Kuhlman have measured the uniformity of sprays using Fast Fourier Transform (FFT) spectra and have found good agreement with visual observation. Krishnan, Seemans, Gottfried, and Kemble have used process control charts to evaluate the performance and degradation of spray nozzles. Their work is useful in predicting useful nozzle lifetime.

Controlled Release Technology

This section describes recent advances in the development of materials and processes to control the release of agrochemicals to the environment or intended target. Wing, Carr, and Doane describe a continuous method for producing starch-encapsulated herbicides. Latheef, Dailey, and Franz have used sulprofos microencapsulated with various polymers to study the efficacy of each polymer system against tobacco budworm on cotton. Levy, Nichols, and Miller describe the use of superabsorbent polymer/pesticide compositions for use against aquatic and household insects. Meyers, Greene, and Springer have developed unique microcapsules to control the release of pesticide and reducing leaching. These polymers are designed to release their contents through seasonal changes in soil temperature.

Water Dispersible Granules

This section contains studies on means of improving the formulation techniques and measuring the effectiveness and physical properties of water-dispersible granules. Delli Colli describes a unique method of screening WDGs employing coated glass plates to determine relative wettability and dispersibility. Gerety provides a review of the current practices for making WDGs. Munie describes advances in formulating non-traditional pesticides such as low melters and highly volatile technicals into WDGs. Haggard describes a method of determining the dynamic granular dispersibility of WDGs using a laser particle size instrument. Fu, Chaudhuri, and Narayanan have developed graft and copolymers of vinyl pyrrolidone that are designed to be used as binders for water-dispersible granules.

I believe the reader will agree that the papers described above provide adequate support that the 13th Symposium on Pesticide Formulations has met its objectives. I extend my thanks to all the authors, moderators, and reviewers and to Drs. B. Devisetty and F. Hall, my cochairmen, for making the Symposium a success and this STP a reality.

Paul D. Berger

Witco Corporation, Houston, TX; symposium
chairman and editor.

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Formulation Technology

James W. Kassebaum¹

SURFYNOL SURFACTANTS AS DEFOAMERS FOR GLYPHOSATE FORMULATIONS WITH ALKYL GLYCOSIDE SURFACTANTS

REFERENCE: Kassebaum, J. W., "Surfynol Surfactants as Defoamers for Glyphosate Formulations with Alkyl Glycoside Surfactants," Pesticide Formulations and Application Systems: 13th Volume, ASTM STP 1183, Paul D. Berger, Bala N. Devisetty and Franklin R. Hall, Eds., American Society for Testing and Materials, Philadelphia, 1993.

ABSTRACT: A major limitation to the use of alkyl glycosides as surfactants for liquid glyphosate formulations is their unacceptable degree of foaming when diluted in the spray tank. I have found that certain acetylenic diol surfactants can be used as defoamers in glyphosate formulations with an alkyl glycoside. The acetylenic diol surfactant was completely solubilized by the alkyl glycoside in the concentrated formulation. Therefore it remained homogeneously distributed throughout the formulation. This overcomes a severe limitation of using conventional defoamers, such as those based on silicone, with water soluble concentrates, since they are not soluble in the formulation and will separate with time. When the formulation was diluted to spray solution concentrations the acetylenic diol surfactant became apparently insoluble (the solution was hazy) and it functioned as a defoamer. The relative defoaming ability of three acetylenic diol surfactants from Air Products, called Surfynol® surfactants, in a liquid glyphosate formulation containing Henkel APG 325 surfactant, was Surfynol 110D > Surfynol 104 >> Surfynol 420.

KEYWORDS: glyphosate, alkyl glycoside, acetylenic diol, defoamer

INTRODUCTION

Glyphosate is usually formulated as a concentrated aqueous solution of its monoisopropylamine salt, which usually also contains a surfactant. Alkyl glycoside surfactants can be used in these types of concentrated formulations. Alkyl glycosides are known to foam to a greater degree than other nonionic surfactants, and have been noted as excellent surfactants for formulating premium foam markers (Aleksejczyk, 1992). A major limitation to the use of alkyl glycosides as surfactants

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for liquid glyphosate formulations is their unacceptable degree of foaming when diluted in the spray tank. A suitable defoamer for these water soluble concentrates should be completely miscible in the formulation or completely dispersed with no separation over time in any temperature environment.

Foam is stabilized by the adsorption of surfactant molecules at the air-water interface. Antifoams, materials that inhibit the initial formation of foam, function by removing surfactant molecules from the foam film, resulting in destabilization of the film (Rosen, 1988). Compounds used as antifoams in aqueous systems are generally of low HLB and insoluble in water (Kulkarni, et al. 1977). Defoamers, materials which destabilize foam that has already formed, function by modifying the film so it produces a less persistent foam (Schick and Schmolka, 1987). Therefore a material could be either soluble or insoluble in a system and still function as a defoamer.

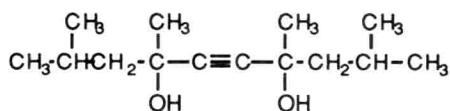
Silicone antifoams, combinations of polydimethylsiloxane and finely divided silica particles, are not water soluble. The proposed antifoaming mechanism hypothesizes surface tension reduction of the aqueous system by the polymer and exposing the silica particles to the interface. The surfactant molecules at the interface then adsorb onto the particles and away from the film, which destabilizes it (Sawicki, 1988). Silica particles in anionic and cationic surfactant solutions become charged (Kulkarni, et al. 1977), evidence of surfactant adsorption onto the particle.

A silicone antifoam would have to be completely dispersed in a water soluble concentrate so that when any portion is used to dilute to a spray solution, the antifoam is present at the right concentration in the spray solution. However, a dispersed antifoam will separate over time and this will be accelerated at high temperature. Therefore, the antifoam will not remain homogeneously distributed throughout the formulation, and if part of the contents of the container is used, antifoam may not be present in the spray solution. Thus, an antifoam that is dispersed in a water soluble concentrate is not acceptable unless the entire container is used to make the spray solution, or the container is small enough so that shaking provides adequate mixing before dilution.

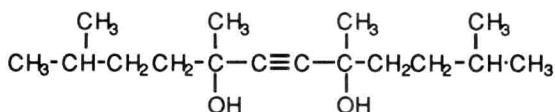
Certain acetylenic diol surfactants provide rainfastness and unit activity enhancement for glyphosate (Dayawon, et al. 1992a), and are solubilized in a formulation by cosurfactants (Brinker, et al. 1992b). They are also known as low foaming surfactants (Leeds, et al. 1965), and some analogs are used as defoamers (Rosen, 1988). I have discovered that certain acetylenic diol surfactants can be used as defoamers in glyphosate formulations with an alkyl glycoside. The acetylenic diol was completely solubilized by the alkyl glycoside in the concentrated formulation. Therefore it remained homogeneously distributed throughout the formulation. However, when diluted to spray solution concentrations the acetylenic diol was apparently insoluble (the solution was hazy) and it functioned as a defoamer. The relative defoaming ability of three acetylenic diol surfactants from Air Products, called Surfynol® surfactants, in a liquid glyphosate formulation containing Henkel APG 325 surfactant, was Surfynol 110D > Surfynol 104 >> Surfynol 420.

MATERIALS

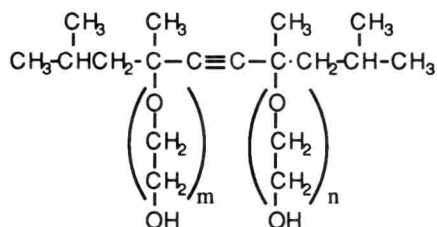
All Surfynol surfactants were from Air Products. Surfynol 104PG50 is a 50% solution of Surfynol 104 in propylene glycol. Surfynol DF110D is a 30% solution of Surfynol 110D in dipropylene glycol. Surfynol 420 is 100% active.



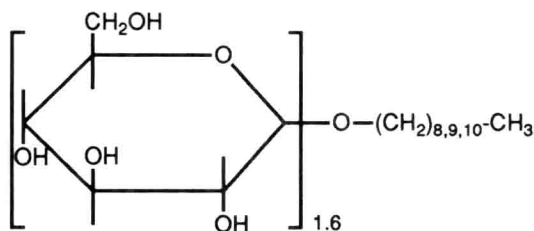
Surfynol 104



Surfynol 110D

Surfynol 420
(m+n = 1.3)

APG 325 was from Henkel and is 65-70% active in water.



APG 325

The source of glyphosate was from Monsanto and was in the form of an approximately 62% aqueous solution of the isopropylamine salt of glyphosate. Propylene glycol was from Fisher. Sag 47 was from Union Carbide and was considered 100% active.

METHODS

Formulations contained the technical glyphosate solution (TGS), alkyl glycoside surfactant, defoamer, and water, and were formulated on a weight % basis. The amount of TGS in a formulation was determined from the glyphosate weight % required. In each formulation the glyphosate acid equivalent (ae) concentration was 30.5%, which was

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equivalent to 360 g/l. The control formulation of 11% active APG 325 without defoamer had the following composition, based on TGS that was 46.3% glyphosate ae.

TGS (46.3% ae)	65.87%
water	18.13%
APG 325	16.00%
	<hr/>
	100.00%

The formulations with defoamers were made by substituting defoamer for water. For example, a formulation that was 3.0% Surfynol 110D would have the following composition:

TGS (46.3% ae)	65.87%
water	8.13%
APG 325	16.00%
Surfynol DF110D	10.00%
	<hr/>
	100.00%

Additional propylene glycol was added to the formulations with Surfynol 104 in some cases, at 4/3 the level of Surfynol 104. This was equivalent to the glycol in formulations of Surfynol DF110D. For example, a formulation that was 4.8% Surfynol 104 would have 6.4% additional propylene glycol. At levels of Surfynol 104 above 5.4%, the additional propylene glycol would cause the formulation composition to exceed 100%, so the maximum amount of propylene glycol possible was added. For example, at 6.6% Surfynol 104, the formulation composition would be as follows:

TGS (46.3% ae)	65.87%
propylene glycol	4.93%
APG 325	16.00%
Surfynol 104PG50	13.20%
	<hr/>
	100.00%

The foaming evaluation involves making a 3% solution of the formulation in a 100 ml graduated cylinder at 50 ml total volume. The solution is then shaken vigorously for 15 seconds, and the foam produced was followed with time. This is similar to the foam stability method of Kulkarni, et al. (1977), the Ross-Miles method - ASTM D1173-53 Standard Test Method for Foaming Properties of Surface Active Agents, and the foam cylinder test described by Aleksejczyk (1992). However, this method is much easier and faster when evaluating the foam of many formulations, and the method is very reproducible and consistent in evaluating the foam stability of these glyphosate formulations with alkyl glycoside surfactants.

The defoaming ability of Sag 47 was evaluated by adding it by weight to a spray solution of the control formulation with no Surfynol in the graduated cylinder, and the foaming evaluation then performed the same way as with the formulations.

RESULTS

Solubility of Surfynols in the Control Formulation

A formulation of 30.5% glyphosate ae and 11% APG 325 (control formulation) was clear and transparent. The control formulation with up to 5% Surfynol 420 was also completely clear at 23°C and 50°C,

indicating that the Surfynol surfactant was solubilized by the alkyl glycoside. This was also true with 2.4%, 3.0%, 3.6%, 4.2%, and 4.8% Surfynol 110D. Formulations with 2.5% and 4.0% Surfynol 104 were soluble at 23°C and 50°C, while formulations with 5.0% to 8.0% Surfynol 104 were insoluble at 23°C but soluble at 50°C. Extra propylene glycol at 4/3 times the amount of Surfynol 104 overcame the insolubility. This was an equivalent amount of glycol that would be in a formulation using Surfynol 110D, since it is only 30% active.

SAG 47 was not soluble in the control formulation.

Foaming of Control Formulation with Defoamers

The control formulation without defoamer had 50 ml of foam after 30 minutes. Surfynol 420 at 1% in the control formulation did not defoam, but at 5% it provided a slow knockdown of foam over 30 to 60 minutes. The spray solution was cloudy, indicating that the Surfynol 420 surfactant was not solubilized in the spray solution. Surfynol 104 and 110D each provided defoaming of the spray solution, and the defoaming increased with increasing levels of Surfynol (Figures 1-2). Surfynol 110D at 2.4% provided relatively good knockdown of foam in 20 minutes, while $\geq 3.6\%$ provided immediate knockdown (Figure 1). Increasing amounts of Surfynol 104 also increased the rate of foam knockdown (Figure 2). However, Surfynol 104 was not as effective as Surfynol 110D (Figure 3), and roughly twice as much 104 as 110D was necessary to achieve the same level of foam knockdown. The spray solutions of formulations with Surfynol 104 and 110D were also hazy, and a light orange oil settled to the bottom of the cylinder after 1 hour.