PLANTA DANINHA
SOCIEDADE BRASILEIRA DA CIÊNCIA DAS PLANTAS DANINHAS
<http://www.sbcpd.org>
ISSN 0100-8358 (print)
1806-9681 (online)

Article

Physical Compatibility and Stability of Pesticide Mixtures at Different Spray Volumes

Compatibilidade Física e Estabilidade de Misturas de Agrotóxicos em Diferentes Volumes de Calda

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Received: September 13, 2018
Approved: November 7, 2018

Planta Daninha 2019; v37:e019214004

ABSTRACT - Available information on physical compatibility and stability of pesticide mixtures is scarce and often divergent. This knowledge is of fundamental importance in the success of pest, disease, and weed control programs. Compatibility and stability of two glyphosate formulations (WG and SL) in a mixture with fluazinam + deltamethrin + trifloxystrobin/cyproconazole at four diluted water concentrations and different times were evaluated under laboratory conditions. The evaluations were performed at the following time intervals: immediately after mixing and 1, 5, 10, 30, 120, 900, and 1,440 minutes after the mixing of pesticides. Scores were assigned on a scale ranging from 1 to 5, where 1 meant immediate separation of compounds and 5 the perfect mixture stability up to 30 minutes. The data were submitted to descriptive statistics and presented in tables. Mixture stability differed in glyphosate formulations and their concentrations due to the used spray solution volume. Distinct mixtures between glyphosate (SL) and deltamethrin, as well as between glyphosate (WG) and deltamethrin, were physically compatible when stirred again even after 24 hours at rest. The mixture containing glyphosate (SL), deltamethrin, and fluazinam and trifloxystrobin/ cyproconazole was incompatible at a volume of 30 L ha^{-1}.

Keywords: pesticides, formulation, spraying.

RESUMO - As informações disponíveis sobre compatibilidade física e estabilidade de misturas de agrotóxicos são escassas e muitas vezes divergentes. Esse conhecimento é de fundamental importância no sucesso de programas de controle de insetos-praga, doenças e plantas daninhas. Nesta pesquisa foi avaliada, em condições laboratoriais, a compatibilidade e estabilidade de duas formulações de glyphosate (WG e SL) em mistura com fluazinam + deltametrina + trifloxistrobina/ciproconazol, todas em quatro concentrações diluídas em água e em tempos variáveis. As avaliações foram feitas nos intervalos de tempo assim descritos: imediatamente após a mistura; 1; 5; 10; 30; 120; 900; e 1.440 minutos após a mistura dos agrotóxicos. Atribuíram-se notas em uma escala variando de 1 a 5, em que 1 significava separação imediata dos compostos, e 5, estabilidade perfeita da mistura até os 30 minutos. Os dados foram submetidos à estatística descritiva e apresentados em forma de tabelas. A estabilidade das misturas diferiu quanto às formulações do glyphosate e suas concentrações, em razão do volume de calda a ser utilizado. As misturas distintas entre o glyphosate (SL) e o inseticida deltametrina, bem como entre o glyphosate (WG) e o inseticida deltametrina, apresentaram-se compatíveis fisicamente quando agitadas novamente, mesmo após 24 horas em repouso. A mistura contendo glyphosate (SL), o inseticida deltametrina e os fungicidas fluazinam e trifloxistrobina/ciproconazol foi incompatível no volume de 30 L ha^{-1}.

Palavras-chave: defensivos agrícolas, formulação, pulverização.

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Doi: 10.1590/S0100-83582019370100125

INTRODUCTION

Brazil is one of the world's largest producers and exporters of agricultural products, being agribusiness one of the main pillars of the country's GDP, currently accounting for 21.5% of the total (Cepea/CNA, 2017). This sector generates jobs and income for a large part of the population. However, tropical conditions, monoculture practice, the extension of agricultural areas, and intensive agriculture generate environments conducive to increased frequency and intensity of phytosanitary problems (Petter et al., 2012; Carvalho et al., 2017). Consequently, the frequency of pesticide use is higher than the average found in high latitude countries (Gazziero, 2015).

Although most of the pests found in Brazil are native, many others come from different countries. This situation has worsened in recent times due to some difficult-to-control, dominant pests such as *Helicoverpa armigera* in soybean (Kuss et al., 2016) and *Anthonomus grandis* in cotton (Martin Neto et al., 2016). This fact highlights the importance of integrated management in its entirety, starting with the preventive method.

An essential tool in integrated pest management is chemical control, which has benefits such as quickness, accessibility, and often low cost when appropriately used (Silva and Silva, 2007). On the other hand, the chemical control method can produce irreversible social, economic, and environmental impacts when used without technical knowledge (Castro, 2009).

One of the most common practices adopted by producers is the mixture of molecules in tanks, as a single pesticide does not have an action spectrum capable of controlling the set of pests occurring in the same crop (Guimarães, 2014; Krause, 2014; Gazziero, 2015). These mixtures, when incompatible, result in significant losses for producers. Thus, prior knowledge of the correct use of tank mixtures with products of different active ingredients and formulations becomes even more essential in order to avoid their low efficiency, possible crop damage, and increased environmental contamination (Petter et al., 2013).

Prado et al. (2011) reported that spray solutions are commonly prepared in advance on large farms to improve system operation, aiming to spend less time to spray the entire planted area. Besides, in case of adverse weather conditions after spray solution preparation, it may continue for a long time stored in the sprayer tank itself, allowing a long reaction of products with the diluent (water) and other components.

In addition, the physical quality of water used in spraying is a factor to be observed. Sediments such as clay and organic matter can adsorb herbicides and reduce their activity (Queiroz et al., 2008). Also, they are capable of blocking filters and tips, mainly those with small outlet openings, as well as reducing the operating capacity of sprayers and life of components such as pump and spray tips (Ramos and Araújo, 2006).

Another factor to consider is the chemical quality of water, which can be analyzed in many ways (Queiroz et al., 2008). One of them, which has great interference on the effectiveness of pesticides, is the hardness, which is defined as the concentration of alkaline earth cations (Ca^{2+}, Mg^{2+}, Sr^{2+}, and Ba^{2+}) present in the water. They are expressed as ppm $CaCO_3$, usually represented by Ca^{2+} and Mg^{2+} (Kissmann, 1997).

According to Ramos and Araújo (2006), the mixture of various products within the sprayer tank and the constant reduction in spray solution volume by producers have generated several problems of physical incompatibility of mixtures in the field. Moreover, the complexity of spray solutions becomes even more worrying due to the popularization of spray solution premix systems or ready-to-use systems. These systems intensify the conditions for possible interactions between the spray solution components, as they can be stored for a few hours (Antuniassi, 2015).

Considering the scarcity and divergence of studies regarding the physical incompatibility of phytosanitary products in spray solutions, this study aimed to know the physical compatibility of five different types of pesticide mixture and their stability over time, prepared at different spray solution volumes.

MATERIAL AND METHODS

The experiment was carried out under laboratory conditions using water from the public distribution as a diluent. A water sample was sent to a laboratory to analyze its pH and hardness, allowing its classification as very soft (Table 1).

Table 1 **-** Water hardness classification used in this research

Class	ppm $CaCO_3$
Very soft	<71.2
Soft	71.2–142.4
Semi-hard	142.4–320.4
Hard	320.4–534.0
Very hard	>534.0

Source: adapted from Conceição (2003).

The tested products were chosen because they are widely used in the field, thus presenting practical and economic importance (Table 2). The values of spray solution volumes represented traditional, current, and trend applications, as follows: high (240 L ha^{-1}), medium (120 and 60 L ha^{-1}), and low (30 L ha^{-1}).

Table 2 **-** Products and respective doses of pesticides used in the experiment

Commercial name	Dose ha^{-1} (L or gg of c.p.)*	Technical name (formulation)	Spray solution volume (L ha^{-1})			
			30	60	120	240
			Amount for 1 L of mixture (L or kg c.p.)*			
Roundup® Original	4.0	Glyphosate (SL)	0.1333	0.0667	0.0333	0.0167
Roundup® WG	2.0	Glyphosate (WG)	0.0667	0.0333	0.0167	0.0083
Sphere® Max	0.2	Trifloxystrobin + cyproconazole (SC)	0.0067	0.0033	0.0017	0.0008
Frowncide®	1.0	Fluazinam (SC)	0.0333	0.0167	0.0083	0.0042
Decis® 25 EC	0.3	Deltamethrin (EC)	0.0100	0.0050	0.0025	0.0013

* L or kg of c.p. = liters or kilograms of the commercial product.

Pesticide doses were defined following the manufacturer's recommendation, while the amount of each product was calculated following the proportions for each spray solution volume under analysis (Table 2). Pipettes of 10.0 and 1.0 mL (one for each product) were used to dose products correctly. Granulated products were weighed on a precision analytical balance, with reference in grams and three decimal places.

Mixtures between different phytosanitary products were conducted in clean and transparent beaker containers in the following order of addition: water, glyphosate (WG), fluazinam (SC), trifloxystrobin/cyproconazole (SC), deltamethrin (EC), and glyphosate (SL), as shown in Table 3. Each product was mixed with two formulations of glyphosate and each of them with water. In addition, pesticides were also mixed without glyphosate.

Stability evaluations of mixtures were studied at the following time intervals: immediately after mixing and 1, 5, 10, 30, 120, 900, and 1,440 minutes after mixing. Interactions were observed from the occurrence of supernatant, precipitation, flocculation, or homogeneous mixture. Scores were assigned on a scale ranging from 1 to 5, where 1 meant immediate separation of compounds 5 the perfect mixture stability up to 30 minutes (Table 4).

The data were submitted to descriptive statistics and presented in tables.

Table 3 **-** Order of pesticide placement in the tank mix

Order of addition of products	Formulation
1	Water
2	Wettable powder
3	Water dispersible granules (WG)
4	Dry flowable
5	Suspension concentrate (SC)
6	Water emulsion
7	Emulsified oil
8	Emulsifiable concentrate (EC)
9	Soluble (liquid) concentrate (SL)
10	Concentrated aqueous solution

Source: adapted from Azevedo (2015).

Table 4 **-** Stability scale of pesticide mixtures

Degree	Condition	Recommendation
1	Immediate separation	Do not apply
2	Separation after 1 minute	Do not apply
3	Separation after 5 minutes	Continuous stirring
4	Separation after 10 minutes	Continuous stirring
5	Perfect stability	No restrictions

Source: Brazilian Center for Bio-aeronautics, cited by Azevedo (2015).

RESULTS AND DISCUSSION

Mixtures of glyphosate (SL) + deltamethrin + fluazinam + trifloxystrobin/cyproconazole presented very varied stability of spray solution when prepared at different volumes (Table 5).

Table 5 - Stability of glyphosate (Roundup® Original) mixed with different pesticides at different spray solution volumes, in Viçosa, MG, 2018

Treatment (glyphosate + fluazinam + trifloxystrobin/ cyproconazole + deltamethrin)									
Spray solution volume ($L\ ha^{-1}$)	Spray solution stability over time (minutes)								
	0	1	5	10	30	120	900	1440	1440(1)
30	5	2*	*	*	*	*	*	*	2
60	5	5	3*	*	*	*	*	*	3
120	5	5	3*	*	*	*	*	*	3
240	5	5	5	5	5	*	*	*	4

Note: The values correspond to the degrees of stability described in Table 4; *precipitate formation; **presence of lumps even after agitation; (1) after stirring.

The mixture for the spray solution volume of 240 $L\ ha^{-1}$ showed perfect stability up to 30 minutes, as observed by the degree 5. On the other hand, spray solution volumes of 60 and 120 $L\ ha^{-1}$ had a degree of incompatibility of degree 3, being recommended their continuous stirring. However, it presented a stability degree 2 after one minute of mixing at 30 $L\ ha^{-1}$, being unsuitable for application under these conditions.

Petter et al. (2012) reported that this incompatibility of degree 2 could lead to product precipitation to the bottom of the sprayer tank, resulting in considerable losses since precipitates can generate a much higher concentration of products at certain application times.

In addition, cases of incompatibility of phytosanitary products may cause damages such as increased phytotoxicity, which may decrease yield potential in some crops (Trezzi et al., 2005; Rakes et al., 2018).

On the other hand, the glyphosate (WG) + deltamethrin + fluazinam + trifloxystrobin/ cyproconazole mixture presented a more stable mixture at different volumes (Table 6).

This mixture presented perfect stability up to 30 minutes at volumes of 30 and 60 $L\ ha^{-1}$, indicated by degree 5, showing a different behavior from the results obtained with glyphosate SL, considering the same spray solution volumes. It occurs because WG products currently have highly water-soluble dispersing agents (Matthews et al., 2016).

However, the mixture of glyphosate WG at a volume of 240 $L\ ha^{-1}$ formed a precipitate after five minutes. Interestingly, glyphosate SL remained stable for up to 30 minutes at the same spray solution volume. Although the laboratory analysis showed low values for water hardness (classified as very soft according to Table 1), there may have been a reaction of the WG formulation components with chemical elements in the water, which were not verified in the hardness test.

Table 6 - Stability of glyphosate (Roundup® WG) mixed with different pesticides at different spray solution volumes, in Viçosa, MG, 2018

Treatment (glyphosate + fluazinam + trifloxystrobin/ cyproconazole + deltamethrin)									
Spray solution volume ($L\ ha^{-1}$)	Spray solution stability over time (minutes)								
	0	1	5	10	30	120	900	1440	1440(1)
30	5	5	5	5	5	*	*	*	**
60	5	5	5	5	5	*	*	*	**
120	5	5	5	4*	*	*	*	*	4
240	5	5	3*	*	*	*	*	*	3

Note: The values correspond to the degrees of stability described in Table 4; *precipitate formation; **presence of lumps even after agitation; (1) after stirring.

According to Petter et al. (2013), incompatibilities may result in less efficient pest and disease control. At certain times of application, underdoses flow may occur due to the formation of "dregs" and their retention in nozzle sieves and line filters in the sprayer.

Glyphosate (SL) mixed with deltamethrin, fluazinam, and trifloxystrobin/cyproconazole showed distinct stability, as shown in Table 7. Perfect stability was observed in the mixture between glyphosate (SL) and deltamethrin, regardless of volume. On the other hand, a precipitate formed after five minutes in mixtures with fluazinam or trifloxystrobin/cyproconazole.

These mixtures, but with glyphosate (WG) showed better stability levels, with precipitate formation only after 10 minutes, with fluazinam or trifloxystrobin/cyproconazole. In addition, glyphosate (WG) + trifloxystrobin/cyproconazole at a spray solution volume of 240 L ha^{-1} showed perfect stability up to 30 minutes (Table 8).

Table 7 - Stability of glyphosate (Roundup® Original) mixed with different pesticides at different spray solution volumes, in Viçosa, MG, 2018

Spray solution volume (L ha^{-1})	Mixture	Spray solution stability over time (minutes)								
		0	1	5	10	30	120	900	1440	1440[(1)]
30	Glyphosate + fluazinam	5	5	3*	*	*	*	*	*	3
	Glyphosate + trifloxystrobin/cyproconazole	5	5	3*	*	*	*	*	*	3
	Glyphosate + deltamethrin	5	5	5	5	5	5	5	5	5
60	Glyphosate + fluazinam	5	5	3*	*	*	*	*	*	3
	Glyphosate + trifloxystrobin/cyproconazole	5	5	3*	*	*	*	*	*	3
	Glyphosate + deltamethrin	5	5	5	5	5	5	5	5	5
120	Glyphosate + fluazinam	5	5	3*	*	*	*	*	*	3
	Glyphosate + trifloxystrobin/cyproconazole	5	5	3*	*	*	*	*	*	3
	Glyphosate + deltamethrin	5	5	5	5	5	5	5	5	5
240	Glyphosate + fluazinam	5	5	3*	*	*	*	*	*	3
	Glyphosate + trifloxystrobin/cyproconazole	5	5	3*	*	*	*	*	*	3
	Glyphosate + deltamethrin	5	5	5	5	5	5	5	5	5

Note: The values correspond to the degrees of stability described in Table 4; * precipitate formation; ** presence of lumps even after agitation; [(1)] after stirring.

Table 8 - Stability of glyphosate (Roundup® WG) mixed with different pesticides at different spray solution volumes, in Viçosa, MG, 2018

Spray solution volume (L ha^{-1})	Mixture	Spray solution stability over time (minutes)								
		0	1	5	10	30	120	900	1440	1440[(1)]
30	Glyphosate + fluazinam	5	5	5	4*	*	*	*	*	**
	Glyphosate + trifloxystrobin/cyproconazole	5	5	5	4*	*	*	*	*	4
	Glyphosate + deltamethrin	5	5	5	5	5	5	5	5	5
60	Glyphosate + fluazinam	5	5	5	4*	*	*	*	*	**
	Glyphosate + trifloxystrobin/cyproconazole	5	5	5	4*	*	*	*	*	4
	Glyphosate + deltamethrin	5	5	5	5	5	5	5	5	5
120	Glyphosate + fluazinam	5	5	5	4*	*	*	*	*	**
	Glyphosate + trifloxystrobin/cyproconazole	5	5	5	4*	*	*	*	*	4
	Glyphosate + deltamethrin	5	5	5	5	5	5	5	5	5
240	Glyphosate + fluazinam	5	5	5	4*	*	*	*	*	**
	Glyphosate + trifloxystrobin/cyproconazole	5	5	5	5	5	*	*	*	5
	Glyphosate + deltamethrin	5	5	5	5	5	5	5	5	5

Note: The values correspond to the degrees of stability described in Table 4; *precipitate formation; **presence of lumps even after agitation; [(1)] after stirring.

Overall, the stability of each pesticide in the water had degree 5, resulting in perfect stability up to 30 minutes, regardless of the spray solution volume (Table 9). However, some mixtures for a longer time left to rest showed precipitate formation, as occurred with fluazinam at 120 minutes and trifloxystrobin/cyproconazole at 900 minutes.

Table 9 - Stability of different pesticides in water under different spray solution volumes, in Viçosa, MG, 2018

Spray solution volume (L ha^{-1})	Mixture	Spray solution stability over time (minutes)								
		0	1	5	10	30	120	900	1440	1440[(1)]
30	Water + glyphosate (SL)	5	5	5	5	5	5	5	5	5
	Water + glyphosate (WG)	5	5	5	5	5	5	5	5	5
	Water + fluazinam	5	5	5	5	5	*	*	*	**
	Water + trifloxystrobin/cyproconazole	5	5	5	5	5	5	*	*	5
	Water + deltamethrin	5	5	5	5	5	5	5	5	5
60	Water + glyphosate (SL)	5	5	5	5	5	5	5	5	5
	Water + glyphosate (WG)	5	5	5	5	5	5	5	5	5
	Water + fluazinam	5	5	5	5	5	*	*	*	**
	Water + trifloxystrobin/cyproconazole	5	5	5	5	5	5	*	*	5
	Water + deltamethrin	5	5	5	5	5	5	5	5	5
120	Water + glyphosate (SL)	5	5	5	5	5	5	5	5	5
	Water + glyphosate (WG)	5	5	5	5	5	5	5	5	5
	Water + fluazinam	5	5	5	5	5	*	*	*	**
	Water + trifloxystrobin/cyproconazole	5	5	5	5	5	5	*	*	5
	Water + deltamethrin	5	5	5	5	5	5	5	5	5
240	Water + glyphosate (SL)	5	5	5	5	5	5	5	5	5
	Water + glyphosate (WG)	5	5	5	5	5	5	5	5	5
	Water + fluazinam	5	5	5	5	5	*	*	*	**
	Water + trifloxystrobin/cyproconazole	5	5	5	5	5	5	*	*	5
	Water + deltamethrin	5	5	5	5	5	5	5	5	5

Note: The values correspond to the degrees of stability described in Table 4; * precipitate formation; ** presence of lumps even after agitation; [(1)] after stirring.

These data are relevant because often spraying is interrupted in the field either by work shifts or even by abnormal weather conditions such as rainfall. Thus, spray solutions under these situations may remain in the sprayer tank for several hours until it is stirred again. Nicolai and Christoffoleti (2007) reported physical incompatibility of pesticides characterized by precipitate formation inside sprayer tanks.

In some cases, even after stirring, the presence of lumps may be observed, as evidenced by fluazinam. This situation can lead to reduced application and phytosanitary control quality.

Deltamethrin, fluazinam, and trifloxystrobin/cyproconazole, when analyzed in mixtures between them, showed perfect stability up to 120 minutes, regardless of the spray solution volume (Table 10). According to Azevedo (2015), incompatibility problems when mixing products of the same physical state or formulation are not expected. However, all presented precipitation when observed longer (1,440 minutes or 24 hours). Moreover, even after stirring, all of them exhibited lump formation, which in practice could reduce the control efficiency in the application.

While most physical and chemical incompatibilities are observed in product mixtures with emulsifiable concentrate and water soluble powder formulations, and emulsion of oil in water with suspension concentrated (SC), mixing products with the same SC formulation may also present incompatibility mainly due to high concentrations of actives in these suspensions (Theisen and Ruedell, 2004; Petter et al., 2013).

Besides, it is important to note that pesticide mixtures can be toxic to various non-target species such as aquatic organisms and mammals, thus requiring special attention (Tallarida, 2001; Castro and Chiorato, 2007; Castro, 2009).

However, pesticide tank mixture or similar allows, among other benefits, shorter exposure of the rural worker to phytosanitary products and better management and prevention of pest resistance (Guimarães, 2014).

Different results may be found in the same mixtures if different doses are used than those tested. Krause (2014) pointed out that doses should be adjusted to each situation according to the problem found in the field, and that mixtures represent a way to broaden the control spectrum.

Table 10 **-** Stability of different pesticides under different spray solution volumes, in Viçosa, MG, 2018

Spray solution volume (L ha^{-1})	Mixture	Spray solution stability over time (minutes)								
		0	1	5	10	30	120	900	1440	1440(1)
30	Fluazinam + trifloxystrobin/cyproconazole	5	5	5	5	5	5	5	*	**
	Fluazinam + deltamethrin	5	5	5	5	5	5	5	*	**
	Trifloxystrobin/cyproconazole + deltamethrin	5	5	5	5	5	5	5	*	**
60	Fluazinam + trifloxystrobin/cyproconazole	5	5	5	5	5	5	5	*	**
	Fluazinam + deltamethrin	5	5	5	5	5	5	5	*	**
	Trifloxystrobin/cyproconazole + deltamethrin	5	5	5	5	5	5	5	*	**
120	Fluazinam + trifloxystrobin/cyproconazole	5	5	5	5	5	5	5	*	**
	Fluazinam + deltamethrin	5	5	5	5	5	5	5	*	**
	Trifloxystrobin/cyproconazole + deltamethrin	5	5	5	5	5	5	5	*	**
240	Fluazinam + trifloxystrobin/cyproconazole	5	5	5	5	5	5	*	*	**
	Fluazinam + deltamethrin	5	5	5	5	5	5	*	*	**
	Trifloxystrobin/cyproconazole + deltamethrin	5	5	5	5	5	5	5	*	**

Note: The values correspond to the degrees of stability described in Table 4; * precipitate formation; ** presence of lumps even after agitation; (1) after stirring.

In this context, in addition to the factors studied in this research, several others may also interfere with the stability of mixtures, especially in agriculture as diverse as that found in Brazil. Therefore, studies capable of generating information, combining cost reduction and environmental contamination, and enabling the sustainability of the agricultural system are needed.

Mixture stability differed regarding glyphosate formulations and concentration (due to the spray solution volume to be used). Mixtures between glyphosate (SL) and deltamethrin, as well as between glyphosate (WG) and deltamethrin were physically compatible even after 24 hours left at rest when stirred again. However, the mixture containing glyphosate (SL), deltamethrin, and fluazinam and trifloxystrobin/cyproconazole was incompatible at a spray solution volume of 30 L ha^{-1}.

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