American Academic Scientific Research Journal for Engineering, Technology, and Sciences
ISSN (Print) 2313-4410, ISSN (Online) 2313-4402
http://asrjetsjournal.org/

Efficacy of Adjuvants Photoprotectors for Herbicides in Sugarcane

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Abstract

Sugarcane is one of the main agricultural crops in the country and is the main raw material used by the sugar and alcohol industry. With the current sugarcane cultivation model, where burning for the harvest is no longer possible, the remaining straw from the mechanical harvest remains in the field. The straw on the ground forms a physical barrier, which reduces the incidence of light and helps to inhibit the growth of some weeds, selecting others adapted to this condition. Among the different weed managements, the application of pre-emergent herbicides is quite usual, but under these conditions the herbicide will be intercepted by straw, which is transported to the soil by leaching in the rainy season or by irrigation. When herbicides are applied during the dry season, they are exposed on the straw surface, subjected to solar radiation and can reach high temperatures. In this environment, herbicides that have photosensitivity characteristics can undergo transformations in their molecules and present photodegradation, not reaching their full effectiveness in controlling weeds. Within this context, this research aimed to study the photosensitivity of the herbicides amicarbazone, metribuzin and sulfentrazone, and evaluating two adjuvants, a concentrated suspension and an emulsifiable concentrate, which allows to protect its molecules from photodegradation. For this an experiment was conducted to simulate the real conditions found in field applications, the samples were subjected to radiation solar for 0, 15, 30, 45 and 60 days. The results were evaluated through the photostability of the herbicides without and with the use of adjuvants. The results found for amicarbazone and sulfentrazone showed that the ideal adjuvant for these molecules was emulsifiable concentrate (EC) formulation and for metribuzin none of the adjuvants were effective, requiring the development of a specific adjuvant for this herbicide.

Keywords: Pesticides; Amicarbazone; Sulfentrazone; Metribuzin; Photodegradation.

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1. Introduction

Sugarcane (*Saccharum spp.*) is one of the most important crops in Brazilian agribusiness. Brazil is the world's largest producer of sugarcane and the estimated production in the 2021/2022 season is 628 million tons [1]. It is a crop that has a semi-perennial cycle, which requires a hot and humid period, with intense solar radiation during the vegetative stage, followed by a dry period in the maturation and harvesting phase. It has high sprouting power, allowing its culm to be harvested for several years in a row without the need for replanting [2]. The mechanized harvesting of sugarcane leaves a thick layer of straw on the soil and its maintenance on the soil surface can hinder the effectiveness of herbicides applied in pre-emergence, since in this system the transport of the herbicide to the soil surface is mainly done by rain or irrigation [3]. When sugarcane is planted in a dry period, the growth of sugarcane is stopped, as is the growth of weeds and, at the return of the rainy season, if the herbicide applied does not have an effective residual action, weeds could germinate again [4].

The use of pre-emergent herbicides is one of the factors that determine great efficiency in weed control during the critical period of competition in sugarcane cultivation [5]. To evaluate the effectiveness of herbicides, it is necessary to know their physicochemical properties, the factors that influence their activities and stability in the soil. It is also necessary to know the period in which the area will remain without rain after application [6], and applications in drier seasons of the year are more likely to have long periods without rain and in these situations the herbicide is exposed on the straw surface.

Photodegradation or photochemical degradation occurs due to the action of light by herbicides, especially at the most destructive wavelength of ultraviolet light [7]. Therefore, when herbicide molecules remain exposed to solar radiation, they will be subject to degradation by light, to a greater or lesser extent, depending on factors such as characteristics and formulation of the molecule, application technology, time and intensity of exposure, type of surface and climatic conditions [8].

In sugarcane crop, the straw surface is directly exposed to solar radiation and this straw can reach high temperatures easily. As a result, herbicides that have photosensitivity characteristics can undergo changes in their composition and present photodegradation [8], reducing their effectiveness and increasing the cost of treatment, as higher doses of the product will be necessary [9]. For these cases, it is necessary to use adjuvants with photoprotective functions incorporated in the spray tank. These adjuvants act as adhesives, retaining the pesticide more quickly on the target, reducing the speed of volatilization and inhibiting degradation by ultraviolet rays (UV) [16]. According to [17], this type of adjuvant can also act through some physical or chemical processes, increasing the rate of herbicide retention by the cuticle or by absorption of ultraviolet rays.

Solar radiation is composed of non-ionizing electromagnetic radiation, classified by its wavelength: ultraviolet (100-400 nm), visible (400-800 nm) and infrared (above 800 nm) radiation. The wavelength is inversely proportional to the energy and its penetration capacity, that is, the solar radiation energy increases with the wavelength reduction [18].

Sunscreens are classified as organic or inorganic, depending on their action mechanism. Organic protectors are formed by molecules containing aromatic rings conjugated with an electron donor group and an ortho electron acceptor group, capable of absorbing UV radiation (high energy) and transforming it into radiations with lower energy, where the excess energy will be re-emitted in the form of heat, light, or is used in a photochemical reaction such as isomerization [19]. Inorganic protectors are composed of zinc oxide (ZnO) and titanium dioxide (TiO_2). These compounds are semiconductors and the absorption of UV radiation allows the excited electrons from the valence band to migrate to the conduction band, releasing energy as a longer wavelength in the form of thermal energy or radiation with a wavelength in the infrared region. Titanium dioxide acts as a physical barrier, capable of dispersing and absorbing UV radiation [20]. When used in sunscreens, it has a wildy protection range with a spectrum that extends from the UVA II to UVB region [21]. It is deposited on the skin and reflects all visible light, having as a final effect a white look, formed by the film of particles on the skin. The whitest particles are those that scatter light most efficiently [22].

Photoprotection is also influenced by the vehicle and its components, as well as the thickness and uniformity of the film formed on the skin [23]. Oily vehicles and O/W emulsions increase protection against the action of light by forming a coating film with emollient property, creating an obstacle to light penetration [24].

This research aimed to study the photosensitivity of the herbicides amicarbazone, metribuzin and sulfentazone, as well as the evaluation of photoprotectors adjuvants in field applications. As the use of photoprotectors adjuvants in spray tank is a topic relatively new, this research found articles limitation on this topic. The developed adjuvants have a similar mode of action to sunscreens used for skin care products. The SC adjuvant is a titanium dioxide concentrated suspension formulation, where the TiO_2 particles are deposited in the straw with herbicides products forming a particles film that reflects visible radiation. The EC adjuvant is a vegetable oil emulsifiable concentrate, where the oil will act on the herbicide products spreadability and penetration. Thus, the adjuvant effectiveness will be related to the thickness of the film and its spreadability.

2. Material and methods

The research project was carried out at the Experimental Station of UPL Brazil – Ituverava – SP, where two different types of adjuvants, a concentrated suspension (SC) and an emulsifiable concentrate (EC), developed by UPL were evaluated for their photoprotection capacity applied in tank mix with agrochemicals: Dinamic (Amicarbazone 700 g/kg WG) , Boral (Sulfentrazone 500 g/L SC) and Unimark (Metribuzin 700 g/kg WG).

2.1. Preparation of de amicarbazone, sulfentrazone e metribuzin solutions

To simulate the real conditions of herbicides application in the field, three different treatments were adopted for each product, simulating different application conditions: with and without the use of photoprotectors adjuvants, being submitted to solar radiation.

Three 1500 mL mixtures were prepared for each product, with the recommended doses for use in the field of each mixture at the concentrations reported in Table 1. For the study, the preparation of the solution was carried out with a volume proportionally smaller than the dose recommended for the field. This calculation was based on the development of the analytical method in High Performance Liquid Chromatography (HPLC) for each molecule, being this dosage the ideal amount for its quantification.

Table 1: Treatments of the products Dinamic, Boral and Unimark

Treatment	Commercial dose recommended for field	Study dose	Preparation of 1500 mL solution
Amicarbazone	2000 g ha^{-1}	10 g ha^{-1}	0.10 g
Amicarbazone + SC adjuvant	2000 g ha^{-1} + 500 mL ha^{-1}	10 g ha^{-1} + 2.5 mL ha^{-1}	0.10g + 0.025 mL
Amicarbazone + EC adjuvant	2000 g ha^{-1} + 500 mL ha^{-1}	10 g ha^{-1} + 2.5 mL ha^{-1}	0.10g + 0.025 mL
Sulfentrazone	1600 mL ha^{-1}	16 mL ha^{-1}	0.16 mL
Sulfentrazone + SC adjuvant	1600 mL ha^{-1} + 1000 mL ha^{-1}	16 mL ha^{-1} + 5 mL ha^{-1}	0.16 mL + 0.05 mL
Sulfentrazone + EC adjuvant	1600 mL g ha^{-1} + 1000 mL ha^{-1}	16 mL ha^{-1} + 5 mL ha^{-1}	0.16 mL + 0.05 mL
Metribuzin	2700 g ha^{-1}	10.8 g ha^{-1}	0.108 g
Metribuzin + SC adjuvant	2700 g ha^{-1} + 1000 mL ha^{-1}	10.8 g ha^{-1} + 5 mL ha^{-1}	0.108 g + 0.05 mL
Metribuzin + EC adjuvant	2700 g ha^{-1} + 1000 mL ha^{-1}	10.8 g ha^{-1} + 5 mL ha^{-1}	0.108 g + 0.05 mL

The product weighing used during the preparation of the mixture was performed on an analytical balance with a resolution of 0.01 mg due to its greater sensitivity. After weighing, the products were added to 1500 mL of water, previously measured with the help of a graduated cylinder, the mixtures were manually stirred until complete homogenization of the products. The mixtures were separated into 2 mL vials and subjected to solar radiation.

The treatments were performed in triplicate and the data were statistically evaluated through simple linear regression analysis, where it was evaluated how changes in the independent variable (exposure time) affect the dependent variable (photosensitivity of molecules).

2.2. Photostability study in solar radiation

This study was conducted at the Experimental Station of UPL Brazil, located in Ituverava-SP. The samples used in this study were exposed throughout the day, where solar radiation hit the vials directly for 0, 15, 30, 45 and 60 days, as shown in Figure 1. After the exposure time, the active ingredients of the mixtures (amicarbazone, metribuzin and sulfentrazone) were analyzed using an Agilent model 1260 Infinity HPLC liquid chromatograph and compared with ambient samples to obtain the real degradation value of each active ingredient.

Figure 1: Sample exposure to solar radiation

The samples were exposed to solar radiation with temperature and incidence of UV radiation, as shown in Table 2 and 3 according to INMET (National Institute of Meteorology), Ituverava-A753 station.

Table 2: Temperature and UV incidence in amicarbazone sample

Period	Exposure time (days)	Average temperature (°C)	Average radiation (kJ/m²)	Total radiation (kJ/m²)
07/01/2019 to 07/16/2019	15	17.59	748.88	287579.19
07/01/2019 to 07/31/2019	30	18.63	793.09	590070.35
07/01/2019 to 08/15/2019	45	19.18	771.39	833105.73
07/01/2019 to 08/30/2019	60	19.80	763.92	1118389.00

Table 3: Temperature and UV incidence in sulfentrazone e metribuzin samples

Period	Exposure time (days)	Average temperature (°C)	Average radiation (kJ/m²)	Total radiation (kJ/m²)
12/20/2019 to 01/04/2020	15	24.39	987.69	361611.81
12/20/2019 to 01/19/2020	30	24.66	1002.44	728188.20
12/20/2019 to 02/03/2020	45	24.49	997.85	1085072.89
12/20/2019 to 02/18/2020	60	24.34	936.10	1556896.70

3. Results and discussion

Some herbicides can present photodecomposition effect of their molecules when exposed to solar radiation. Next, the photodegradation of the herbicides amicarbazone, sulfentrazone and metribuzin exposed to solar radiation, and the action of photoprotectors adjuvants applied in tank mix will be discussed. Herbicide degradation data were analyzed by applying the F test on the analysis of variance, in order to detect the significance of the interaction. When significant, the levels of the degradation factor (%) of the herbicide over time (days) were analyzed using a three-parameter nonlinear sigmoidal regression, using the scientific curve fitting program SigmaPlot v.12.0:

$$Y = \frac{a}{(1+ exp^{-\left(\frac{x-x0}{b}\right)})} \tag{1}$$

Where **y** = percentage of herbicide degradation; **x** = time in days; **a**, **x0** and **b** = parameters of the curve, so that **a** is the estimate of the maximum accumulation of the variable, **x0** is the inflection point and **b** is the slope of the curve.

3.1. Amicarbazone fotodegradation study

At 0, 15, 30, 45 and 60 days after submitting the samples to solar radiation, the degradation of amicarbazone to exposure and the effectiveness of photoprotective adjuvants were evaluated. Table 4 shows the parameters of regression equations for analysis of amicarbazone degradation.

Table 4: Parameter of regression equations for analysis of amicarbazone degradation

Adjuvant	Regression parameters			R^2	F
	a	*b*	*x0*		
No Adjuvant	88,72	8,07	16,04	0,9364	88,35**
Adjuvant SC	88,66	16,65	33,15	0,8470	33,23**
Adjuvant EC	64,34	7,82	29,28	0,9076	58,90**

**Significant at 1% probability

Analyzing the data in Table 5 and Figure 2, it is observed that at the end of 60 days exposed to solar radiation, amicarbazone without the use of adjuvant showed 95.07% degradation, with the use of SC adjuvant a degradation of 74.87 % and with the use of EC adjuvant a degradation of 64.90%. In other words, according to the data obtained, the SC adjuvant presented 21.24% greater photoprotection of the herbicide and the EC adjuvant presented 31.76% greater photoprotection of the herbicide compared to amicarbazone applied without adjuvant.

Table 5: Amicarbazone degradation according to the exposure time to solar radiation

Treatments	Amicarbazone degradation (%)				
	0 days	15 days	30 days	45 days	60 days
Amicarbazone	0.00	50.53	66.80	84.10	95.07
Amicarbazone + SC adjuvant	0.00	34.93	39.60	58.83	74.87
Amicarbazone + EC adjuvant	0.00	7.60	35.47	53.97	64.87

Studies carried out by [10] showed that, for applications of amicarbazone on straw where the treatment receives a precipitation of 30 mm after application, the herbicide showed an excellent level of control from 14 days after application (DAA), remaining so until 56 DAA. For treatments where precipitation did not occur, the control rates were lower, with a maximum control of 65% at 28 DAA. This result shows the herbicide need to reach the soil, for example due to the occurrence of rain after application.

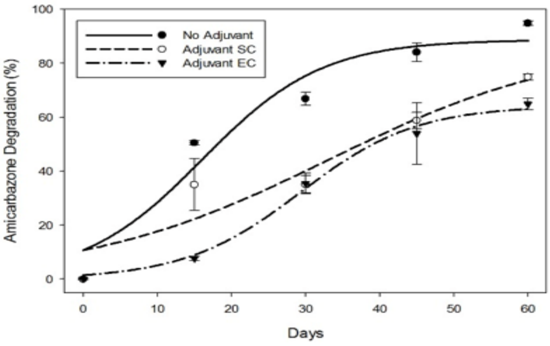

Figure 2: Degradation (%) of amicarbazone under solar radiation without adjuvant and with adjuvants SC and EC. Error bars are ±1 SE

According to [26] applications in periods of greater water restriction, were the ones that promoted the highest concentrations of amicarbazone in different soil depths, which suggests that this herbicide has high mobility in the soil, since that few rains were sufficient to leach the herbicide to deeper layers. At this time of application, low concentrations of amicarbazone were observed in the soil for application on the straw, demonstrating the dependence on rainfall for the herbicide to reach the soil. [11] also observed that the longer the dry periods, the lower the amount of amicarbazone leached from the straw to the soil. For intervals without rain of 1 and 30 days after application, leaching of 81% and 51% was observed, respectively, for a rainfall of 20 mm of rain. These results corroborate those obtained in this research, where the amicarbazone exposed to solar radiation without the use of adjuvant after 30 days showed a degradation of 66.80%, which explains the leaching of only 51% of the herbicide in [11] study.

The samples submitted to solar radiation in this research reproduces the reality of application carried out in the field during dry season and according to [8], the photolysis of amicarbazone can occur in visible and UV radiation, with UVC, UVB and UVA radiation being the main responsible. Comparing the studies, the two adjuvants tested showed photoprotection characteristics improving the herbicide protection, where the EC adjuvant provided a greater protection thant SC adjuvant. It can be due to the fact that the vegetable oil of the formulation acted on herbicide spreadability and penetration capacity. Thus, the effectiveness of the sunscreen was related to the thickness of the film and its spreadability.

Therefore, for dry season applications on the straw, the combination of amicarbazone plus EC adjuvant developed by UPL in tank mix can be an option, where the herbicide can be exposed on straw for long periods and when the first rains occur, it will leach the product in sufficient quantity to control weeds, without the necessity of increase the dose or the number of applications.

3.2. Sulfentrazone fotodegradation study

At 0, 15, 30, 45 and 60 days after submitting the samples to solar radiation, the degradation of sulfentrazone to exposure and the effectiveness of photoprotective adjuvants were evaluated. Table 6 shows the parameters of regression equations for analysis of sulfentrazone degradation.

Table 6: Parameter of regression equations for analysis of sulfentrazone degradation

Adjuvant	Regression parameters			R^2	F
	a	*b*	*x0*		
No Adjuvant	55,10	2,81	12,56	0,9690	187,50**
Adjuvant SC	59,37	6,85	14,50	0,9446	102,35**
Adjuvant EC	51,20	12,28	22,15	0,8990	53,41**

**Significant at 1% probability

Analyzing the data in Table 7 and the graph in Figure 3, it is observed that at the end of 60 days exposed to solar radiation, sulfentrazone without the use of adjuvant showed 62.00% degradation, with the use of SC adjuvant a degradation of 62.7% and with the use of EC adjuvant a degradation of 49.70%. According to the data obtained, the use of SC adjuvant had no protective effect for sulfentrazone. The EC adjuvant presented 19.84% greater photoprotection of sulfentrazone compared to the application without adjuvant.

Table 7: Sulfentrazone degradation according to the exposure time to solar radiation

Treatments	Sulfentrazone degradation according (%)				
	0 days	15 days	30 days	45 days	60 days
Amicarbazone	0.00	38.90	51.50	51.70	62.00
Amicarbazone + SC adjuvant	0.00	35.80	46.00	60.40	62.70
Amicarbazone + EC adjuvant	0.00	26.70	28.40	45.10	49.70

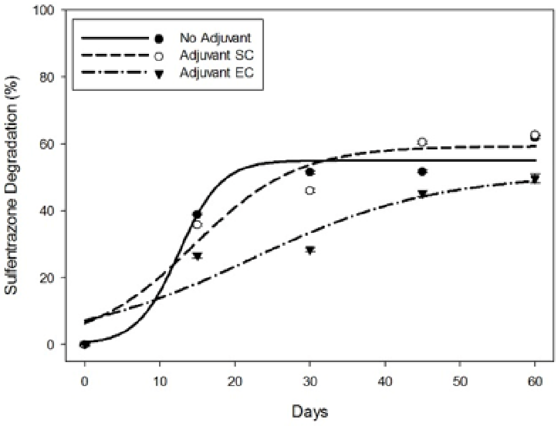

Figure 3: Degradation (%) of sulfentrazone under solar radiation without adjuvant and with adjuvants SC and EC. Error bars are ±1 SE

At the end of 30 days, sulfentrazone exposed to solar radiation without the use of adjuvant showed a degradation of 62%, it means that if there was rain after this period, the herbicide could be leached at concentrations up to 38%. Studies carried out by [12] showed that the transposition of sulfentrazone on sugarcane straw varied as a function of the interval between herbicide application and rainfall simulation. After 30 days of application without rain, 36.61% of the herbicide was recovered, that is, 63.69% of the herbicide was degraded. Comparing the results found by [12] with this research, it could be observed that the use of EC adjuvants showed 19.84% greater photoprotection of sulfentrazone and for this herbicide the use of SC adjuvant had an antagonistic effect.

Reference [13] evaluated the interception of sulfentrazone (800 g ha^{-1}), applied on different amounts of sugarcane straw, different time intervals and intensities of rainfall simulations after herbicide application. They observed that sulfentrazone is more resistant to permanence in the straw, as it was effective even when the rain was simulated 7 days after its application. Similar results were observed by [14], when simulating rain at 55 days after sulfentrazone application, obtained an excellent control of *Cyperus Rotundus*, thus demonstrating a possible permanence of the product in the sugarcane straw. After 60 days exposed to solar radiation, the herbicide without the use of adjuvant showed 62% degradation, comparing with the results found by [13] and [14] it is observed that the dose used of the herbicide is sufficient to guarantee a good efficacy even when it is photodegraded.

The application of herbicides to sugarcane straw during drought periods may apparently promote other losses or degradation processes, including volatilization and photo-degradation. The difference in roughness between the soil surface, the residue surface and the penetration of various wavelengths of sunlight may result in differences in sorption and photodegradation, respectively, of a specific herbicide directly applied to soils or straws [7].

According to [8], sulfentrazone is a low-photosensitive herbicide, with UVB and UVC radiation being the ones that most contribute to his photodegradation. The studies discussed only evaluated the biological efficacy without quantifying the losses; hence, despite the high losses of herbicide when exposed to the atmosphere on the straw surface for long periods of drought, the remaining amount that reaches the soil after the rains may suffice for good control of various weeds. Sulfentrazone dose may need to be adjusted to offset these losses in the dry season. However, the sensitivity of target weeds to sulfentrazone and the aspects related to selectivity should also be considered to avoid any injury on the crop in case early rains occur [25]. Therefore, an option to increase the efficiency of sulfentrazone control in applications on straw without increase the dose, it is to use the herbicide plus EC adjuvant developed by UPL in tank mix.

3.3. Metribuzin fotodegradation study

At 0, 15, 30, 45 and 60 days after submitting the samples to solar radiation, the degradation of sulfentrazone to exposure and the effectiveness of photoprotective adjuvants were evaluated.

Analyzing the data in Table 8, it is observed that at 15 days metribuzin already showed a high degradation, around 85% for all treatments and at 30 days the molecule has already been completely degraded. In this case, for this molecule the adjuvants were not effective, not contributing to the photoprotective effect.

Studies on the dynamics of applied metribuzin on different amounts of sugarcane straw and the effect of periods and intensities of rainfall after its application showed that the first 20 mm are responsible for herbicides leach, presenting high transport capacity [27]. [28] also observed that the greater the amount of straw, the greater the herbicides retention, thus requiring higher precipitation to occur herbicide transportation to the soil.

Table 8: Metribuzin degradation according to the exposure time to solar radiation

Treatments	Sulfentrazone degradation according (%)				
	0 days	15 days	30 days	45 days	60 days
Amicarbazone	0.00	84.70	100	100	100
Amicarbazone + SC adjuvant	0.00	84.00	100	100	100
Amicarbazone + EC adjuvant	0.00	86.60	100	100	100

[6] evaluating the metribuzin effectiveness, applied on straw and subjected to different periods without rain occurrence, found high levels of control of *Ipomoea grandifolia* and *Sida rhombifolia* up to 14 days without occurrence of rain after application. When the rains occurred at 21 and 28 days after application, a low effectiveness of the herbicide in controlling these species was observed. These results corroborate the results found in this research, as the herbicide with 15 days exposed to solar radiation was photodegraded around 85%,

explaining the low effectiveness in controlling these species.

Reference [15] found that the longer the dry periods, the lower the amount of metribuzin leached from the straw to the soil. For intervals without rain of 0, 1, 7, 14 and 28 days after application, leaching of 92, 81, 38, 24 and 16% was observed, respectively, for a rainfall of 20 mm of rain. That is, at 14 days on the straw, 76% of the herbicide had been degraded, a result similar to that found at 15 days in this research. It can be due to the fact that when a herbicide is applied to the straw, is intercepted by the surface of this and becomes vulnerable to volatilization and/or photolysis, until it is leached into the soil.

According to [8], the photolysis of metribuzin can occur in the entire spectrum of solar radiation, with UVB and UVC radiation being the ones that most influence his photolysis. According to the degradation data of metribuzin exposed to solar radiation, Table 6, it was observed that at 15 days the herbicide had degraded 85% and at 30 days it had degraded 100%. What confirms and explains the results found by [15], the herbicide was exposed to solar radiation on the straw for 28 days without rain, when precipitation occurred, only the leaching of the percentage of non-degraded active on the straw occurred and this amount leached is not enough to maintain an effective control, which explains the low efficacy of the herbicide found by [6] at 21 and 28 DAA.

Therefore, for applications of metribuzin under the straw, there is no need to use the adjuvants studied in this research, as they will not be effective. As metribuzin is very photosensitive, a specific adjuvant for this molecule should be developed.

4. Conclusion

The herbicides amicarbazone, sulfentrazone and metribuzin are photosensitive solar radiation. Therefore, the permanence period of them on the straw has a great influence on the efficiency of weed control in the sugarcane crop.

The results obtained in studies comparing EC and SC adjuvants developed by UPL conducted in the laboratory and in the field indicate that for amicarbazone and sulfetrazone the most suitable adjuvant is EC, presenting 31.76% and 19.84%, respectively, greater photostability of the herbicides compared to the application without adjuvant. For metribuzin none of the adjuvants were efficient. Therefore, the development of a specific adjuvant for this molecule is indicated for future studies. It was a limitation found by this research, not be possible develop a photoprothector adjuvant with a broad protection spectrum. Thus, being necessary to develop the adjuvant according to the characteristics of each herbicide. One way to capture this in future studies is to conduct the study by substance groups or similar mode of action groups.

The adjuvants proposed for the molecules of amicarbazone and sulfentrazone, promote an increase in environmental quality and sustainability in their use, since they positively impact their effectiveness, improving the photostability of these herbicides, being able to reduce their degradation and the number of applications and doses necessary to ensure their effectiveness.

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