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STUDY OF THE EFFECTS OF ADJUVANTS ASSOCIATED WITH INSECTICIDES ON THE PHYSICOCHEMICAL PROPERTIES OF THE SPRAY SOLUTION AND CHARACTERIZATION OF DEPOSITS ON WHEAT AND MAIZE LEAVES UNDER SIMULATED RAIN

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KEYWORDS

surface tension, contact angle, potential of hydrogen (pH), coverage, and deposit.

ABSTRACT

The association of adjuvants with phytosanitary products has been tested for several years but is commonly recommended without considering technical and scientific criteria. This study aimed to evaluate the influence of the association of insecticides with different adjuvants on surface tension, contact angle, coverage, hydrogen ionic potential (pH), and the formation of deposits exposed to rain. The insecticides thiamethoxam, imidacloprid, and lambda-cyhalothrin were tested at doses of 75 g ha^{-1}, 150 g ha^{-1}, and 150 g ha^{-1}, respectively, with a spray volume of 200 L ha^{-1}. Break-Thru 240®, Break-Thru 233®, Break-Thru Union®, Oleo FC Agraröl®, and Naturo'il® were added to each of these spray solutions. The results showed that the addition of adjuvants reduced the surface tension and contact angle, and consequently, increased droplet coverage. No deposit pattern was found in the thiamethoxam, imidacloprid, and lambda-cyhalothrin insecticides associated with the adjuvants and a higher amount of residue was observed on wheat leaves than on maize leaves. The use of adjuvants can be an alternative to improve the efficiency of insecticides, allowing the product to achieve its full control potential.

INTRODUCTION

The addition of adjuvants to the spray solution interferes with the application of phytosanitary products, modifying certain properties of the spray solution and/or increasing its biological efficiency. The adjuvants can alter the physicochemical properties of the spray solution, thus altering factors such as wettability, adhesion, and spreading of spray droplets and contributing to better retention and penetration of the active ingredient (Azevedo & Castelani, 2013). However, this improvement has not been observed for all adjuvants; it depends on the product used and the biological target (Melo et al., 2014).

Surface tension is a physical phenomenon that occurs on the surface of water droplets. In agricultural applications where water is the main vehicle, this is the result of the attraction of hydrogen molecules. These cohesive forces tend to reduce the surface area occupied by the liquid, thus causing the formation of spherical drops. This shape decreases the contact between the droplet and the leaf surface, resulting in an increased contact angle between the leaf and the product, reducing its penetration rate (Hazen, 2000).

The use of adjuvants can reduce the surface tension of insecticide spray solutions (Costa et al., 2017). This effect improves droplet coverage. However, this response varies according to the characteristics of the adjuvant used (Holloway et al., 2000). The pH of the spray solution is another physicochemical characteristic that can be influenced by the addition of adjuvants (Cunha et al. 2017).

Rain, which is one the environmental factors that cause a reduction in the efficiency of phytosanitary products, has an immediate effect on the products on the leaf surface. The influence of rain on the efficiency of crop protection agents is related to the tenacity of the deposits (Hunsche et al., 2011). Some adjuvants contribute to reducing the effect of rain after spraying (rainfastness), i.e., they reduce the minimum period necessary without rain, so that the action of phytosanitary products is not compromised (Melo et al., 2015).

The aim of the present study was to evaluate the influence of the association of insecticides with different adjuvants on surface tension, contact angle, coverage, potential of hydrogen (pH), and the formation of deposits on wheat and maize plants exposed to rain.

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MATERIAL AND METHODS

The experiments were conducted at the Institute of Crop Science and Resource Conservation at the University of Bonn, Bonn, Germany. The treatments comprised the association of insecticides (Factor A) and adjuvants (Factor D). Tests were performed on the solutions to determine factors including their surface tension, contact angle, and pH. After spraying, additional tests were performed, including tests to assess droplet coverage, and images of the deposits were taken with a scanning electron microscope to evaluate the effect of rain on the treatments.

Wheat (*Triticum aestivum* 'Oakley') and maize (*Zea mays* 'Lorena') plants were sown in pots (TEKU-Container EC 17, volume = 2 L, Pöppelmann GmbH & Co. KG, Lohne, Germany) with a substrate prepared from peat and sand (5 plants for wheat and 1 for maize). Each pot was an experimental unit (EU) and was cultivated indoors. Each treatment consisted of 10 EUs, totaling 180 EUs, which were kept in a controlled environment under a temperature of 20 ± 5 °C and relative air humidity of 50 ± 10%. The plants received fertigation with a standard commercial fertilizer, according to the needs of each crop, during the entire period that they remained in the greenhouse. They were kept until they reached approximately 0.15 m in height, which was considered the ideal size for use in trials.

To determine the adjuvant doses, preliminary tests for surface tension and contact angle of adjuvants were performed with only water at different concentrations. The adjuvants and doses tested were: Break-Thru® S240, Break-Thru® S233, Break-Thru® Union (Evonik Goldschmidt GmbH, Essen, Germany), Oleo FC Agraröl®, and Naturo'il® (Stoller Chemical Co, Houston, USA). Three different concentrations (0.01%, 0.05%, and 0.1% of the spray volume) were tested for Break-Thru® adjuvants, whereas four concentrations (0.1%, 0.5%, 1%, and 2% of the spray volume) were tested for Oleo FC Agraröl® and Naturo'il®.

Surface tension was determined using the pendant drop method (10 drops per treatment solution) and the contact angle was measured using the sessile drop method (3 µL; n = 10). A DSA 30E goniometer (Krüss GmbH, Hamburg, Germany) was used for evaluation of both wheat and maize leaves. The wheat and maize leaves were cut in half and fixed on slides using double-sided tape, and drops were applied to the central area of the leaf, avoiding the central vein.

Based on preliminary studies (Table 2), adjuvant doses were defined for other studies. The same methodology was used to evaluate the surface tension and contact angle of the treatments, and the insecticides evaluated were: thiamethoxam (Actara 25 WG®, Syngenta, Basel, Switzerland) imidacloprid (Confidor®, Bayer, Leverkusen, Germany) and lambda-cyhalothrin (Karate®, Syngenta, Basel, Switzerland), with simulated applications with a spray volume of 200 L ha^{-1}. The treatments are described in Table 1.

TABLE 1. Insecticides, adjuvants, and their doses used in tests on wheat and maize.

Insecticides (Factor A)	Dose (g of i.a. ha)	Adjuvants (Factor D)	Dose (v/v)
		1. Water	-
1. Thiamethoxam	75	2. Break Thru® S240	0.05%
2. Imidacloprid	150	3. Break Thru® S233	0.05%
3. Lambda-cyhalothrin	150	4. Break Thru® Union	0.05%
		5. FC Agraröl®	0.5%
		6. Naturo'il®	0.5%

The pH was measured with a Hanna HI 223 benchtop pH meter, and all spray solutions and the water alone were measured. Four measurements were completed for each solution.

The Assistat® statistical package was used to compare the results of surface tension and contact angle. The tables and graphs were generated in Excel 2013®. The means were compared using Tukey's test (p-value = 0.05).

The quality of the application was evaluated through the droplet coverage of the treatments, using a mechanized sprayer from the University of Bonn (B-PSA-1; Department of Agricultural Engineering, University of Bonn, Germany), equipped with flat-fan nozzles (XR 110.03, Teejet, Illinois, USA) with a travel speed of 6 km h^{-1}, operating with 300 kPa pressure, and calibrated for a spray volume of 200 L/ha. Coverage was evaluated using hydrosensitive cards (Novartis AG, Basel, Switzerland) measuring 5 cm^2, with four repetitions per treatment. The images of the cards were scanned and analyzed using Gotas® software.

To evaluate the effect of rain on the deposits, 2 h after the plants were subjected to treatment, they were exposed to a rain intensity of 5 mm h^{-1} using a rain simulator (B-LRS-2 Department of Agriculture Engineering, University of Bonn, Germany). The plants were taken to the greenhouse for drying for 24 h; plants not exposed to rain were used to compare the images of the deposits. Images were obtained using a scanning electron microscope (ESEM XL 30 FEI; Philips, Eindhoven, Holland), and the most representative images of the deposits were documented after the rain simulation.

RESULTS AND DISCUSSION

Preliminary tests showed that the addition of adjuvants altered the physicochemical properties of the spray volume (Table 2). The most appropriate doses of the Break-Thru® S240, Break-Thru® S233, and Break-Thru® Union organosilicone adjuvants were 0.05% (v/v) and 0.5% (v/v) for the Oleo FC Agraröl® and Naturo'il® oils, respectively. Thus, these doses were used in all trials.

TABLE 2. Surface tension and contact angle for wheat and maize.

Treatments	Dose (v/v)	Surface tension (mN m^{-1})	Contact angle (°)	
			Wheat	Maize
1. Water	-	72.7 a	124.3 a	110.4 a
2. Water + Break-Thru® S233	0.01%	69.2 b	109.2 b	109.5 b
3. Water + Break-Thru® S233	0.05%	34.2 c	94.3 c	93.1 c
4. Water + Break-Thru® S233	0.10%	24.6 d	68.6 d	65.1 d
CV%		2.4	7.0	7.7
Treatments	Dose (v/v)	Surface tension (mN m-1)	Contact angle (°) Wheat	Contact angle (°) Maize
1. Water	-	72.8 a	119.3 a	105.7 a
2. Water + Break-Thru® S240	0.01%	31.1 b	91.4 b	85.2 b
3. Water + Break-Thru® S240	0.05%	22.8 c	54.7 c	52.2 c
4. Water + Break-Thru® S240	0.10%	21.8 c	39.3 d	33.6 d
CV%		2.8	8.4	7.5
Treatments	Dose (v/v)	Surface tension (mN m^{-1})	Contact angle (°) Wheat	Contact angle (°) Maize
1. Water	-	72.7 a	116.6 a	110.9 a
2. Water + Break-Thru® Union	0.01%	41.4 b	97.4 b	93.3 b
3. Water + Break-Thru® Union	0.05%	26.3 c	69.2 c	64.8 c
4. Water + Break-Thru® Union	0.10%	23.9 d	41.8 d	42.9 d
CV%		2.8	8.9	6.3
Treatments	Dose (v/v)	Surface tension (mN m^{-1})	Contact angle (°) Wheat	Contact angle (°) Maize
1. Water	-	72.9 a	118.1 a	107.2 a
2. Water + Naturo'il®	0.10%	68.7 b	99.9 b	105.8 a
3. Water + Naturo'il®	0.50%	38.7 c	96.9 b	85.4 b
4. Water + Naturo'il®	1.00%	34.6 d	84.8 c	77.9 b
5. Water + Naturo'il®	2.00%	32.1 e	72.0 d	68.9 c
CV%		2.2	10.5	7.6
Treatments	Dose (v/v)	Surface tension (mN m^{-1})	Contact angle (°) Wheat	Contact angle (°) Maize
1. Water	-	72.1 a	109.2 b	119.6 a
2. Water + Oil FC Agraröl ®	0.10%	72.2 a	120.1 a	100.5 b
3. Water + Oil FC Agraröl ®	0.50%	71.5 ab	121.6 a	100.3 b
4. Water + Oil FC Agraröl ®	1.00%	69.9 b	103.5 b	94.2 b
5. Water + Oil FC Agraröl ®	2.00%	68.2 c	101.9 b	95.1 b
CV%		1.9	6.5	6.9

*Values with the same letter in a column were not significantly different according to Tukey's test at 5% probability level.

The association of adjuvants with insecticides resulted in reduced surface tension in all treatments (Table 3). However, although all adjuvants have decreased surface tension, the treatment with Oleo FC Agraröl® showed values very close to those for pure insecticide and pure water. The spray solutions containing organosilicone adjuvants (Break-Thru® S240 and Break-Thru® S233) were those with the lowest surface tension values. The use of organosilicone adjuvants to reduce surface tension has been previously reported (Wang & Liu, 2007), although not for solutions associated with these insecticides and adjuvants, which are widely used in Brazilian agriculture. Furthermore, treatment with Naturo'il® had reduced surface tension at levels similar to those of treatments with organosilicone adjuvants.

TABLE 3. Surface tension and contact angle when different insecticides and adjuvants are used on wheat and maize.

Treatments	Surface tension ($mN\ m^{-1}$)	Contact angle (°)	
		Wheat	Maize
1. Thiamethoxam	73.1 a	122.5 a	111.7 a
2. Thiamethoxam + Break-Thru® S240	23.2 f	43.5 d	34.9 e
3. Thiamethoxam + Break-Thru® S233	26.2 e	56.5 c	49.0 d
4. Thiamethoxam + Break-Thru® Union	33.4 d	95.3 b	88.4 c
5. Thiamethoxam + Oil FC Agraröl®	68.6 b	118.8 a	103.0 b
6. Thiamethoxam + Naturo'il®	43.4 c	93.1 b	88.0 c
7. Water	73.8 a	117.8 a	111.3 a
CV%	2.4	4.7	6.7
Treatments	Surface tension ($mN\ m^{-1}$)	Contact angle (°)	
		Wheat	Maize
1. Imidacloprid	71.7 ab	111.8 b	110.9 a
2. Imidacloprid + Break-Thru® S240	22.2 e	46.3 f	38.6 d
3. Imidacloprid + Break-Thru® S233	24.2 d	56.1 e	53.5 c
4. Imidacloprid + Break-Thru® Union	35.7 c	95.1 c	89.6 b
5. Imidacloprid + Oleo FC Agraröl®	70.5 b	113.4 ab	106.6 a
6. Imidacloprid + Naturo'il®	37.2 c	86.0 d	82.5 b
7. Water	73.1 a	120.2 a	108.0 a
CV%	2.7	5.9	8.1
Treatments	Surface tension ($mN\ m^{-1}$)	Contact angle (°)	
		Wheat	Maize
1. Lambda-cyhalothrin	70.8 b	116.4 a	96.4 bc
2. Lambda-cyhalothrin + Break-Thru® S240	22.4 f	51.3 d	43.4 f
3. Lambda-cyhalothrin + Break-Thru® S233	25.2 e	60.9 d	53.6 e
4. Lambda-cyhalothrin + Break-Thru® Union	33.5 d	101.2 b	94.3 c
5. Lambda-cyhalothrin + Oleo FC Agraröl®	63.4 c	104.5 b	102.2 ab
6. Lambda-cyhalothrin + Naturo'il®	32.7 d	84.2 c	77.6 d
7. Water	72.9 a	116.0 a	107.7 a
CV%	3.3	8.1	6.6

* Values followed by the same letter in a column were not significantly different according to Tukey's test at 5% probability level.

The results of contact angle in the treatment using only water showed that the wheat (≅ 117°) and maize (≅ 108°) leaves are hydrophobic, i.e., have contact angles greater than 90° (Yuan & Lee, 2013). Thus, the leaves of these two plants have poor wettability, confirming the importance of using adjuvants to improve this important attribute of spray droplets. The use of adjuvants proved to be an alternative for reducing the contact angle between the drop and the surface of the wheat and maize leaves. The treatment with Oleo FC Agraröl® was the only in which these values did not decrease, with similar results to those for insecticide spraying and water spraying in most cases. The association of Break-Thru® S240 and Break-Thru® S233 adjuvants with the three tested insecticides resulted in the lowest contact angle values, which allows for better wettability and spreading (Hess & Foy, 2000). Thus, it is possible to state that the reduction of surface tension and consequently of the contact angle, after the addition of adjuvants, substantially improves the wettability, and consequently, the spray quality.

Despite the different modes of contamination by the target insects, a higher wettability is a desirable factor to improve the efficiency of the tested insecticides, thus enabling better control of pest insects. Although the Break-Thru® Union adjuvant reduced surface tension to levels similar to the Break-Thru® S240 and Break-Thru® S233 adjuvants, it did not exhibit similar behavior to these products when the contact angle was assessed. This may be caused by its chemical composition; it should therefore not be recommended as a spreader. Although the Naturo'il® adjuvant is a vegetable oil in its basic composition, it already showed signals of reduction in surface tension at a dose of 0.50%. These results differ from the findings of Castro et al. (2018) who reported that Naturo'il® required doses higher than 1% to reduce surface tension. This ability to reduce surface tension results in a lower contact angle, which shows that the product can improve wettability in wheat and maize leaves.

There was no significant change in the pH of the spray solution in the treatments tested (Table 4). This fact indicates that they can be used in the field without affecting the efficiency of insecticides.

TABLE 4. Droplet coverage and pH with different insecticides and adjuvants.

Treatments	Coverage (%)	pH
1. Thiamethoxam	37.6 b	7.7
2. Thiamethoxam + Break-Thru® S240	49.0 a	7.6
3. Thiamethoxam + Break-Thru® S233	42.8 ab	7.6
4. Thiamethoxam + Break-Thru® Union	39.9 ab	7.7
5. Thiamethoxam + Oil FC Agraröl®	42.2 ab	7.5
6. Thiamethoxam + Naturo'il®	45.7 ab	7.2
7. Water	-	7.0
CV%	5.8	-
Treatments	**Coverage (%)**	**pH**
1. Imidacloprid	42.0 b	6.7
2. Imidacloprid + Break-Thru® S240	56.7 a	7.2
3. Imidacloprid + Break-Thru® S233	48.0 ab	6.9
4. Imidacloprid + Break-Thru® Union	42.2 b	7.1
5. Imidacloprid + Oleo FC Agraröl®	43.4 b	7.0
6. Imidacloprid + Naturo'il®	46.9 ab	6.9
7. Water	-	7.0
CV%	5.3	-
Treatments	**Coverage (%)**	**pH**
1. Lambda-cyhalothrin	37.9 b	7.7
2. Lambda-cyhalothrin + Break-Thru® S240	50.6 a	7.6
3. Lambda-cyhalothrin + Break-Thru® S233	43.7 ab	7.6
4. Lambda-cyhalothrin + Break-Thru® Union	41.9 ab	7.7
5. Lambda-cyhalothrin + Oleo FC Agraröl®	42.7 ab	7.5
6. Lambda-cyhalothrin + Naturo'il®	45.4 ab	7.2
7. Water	-	7.0
CV%	5.7	-

* Values followed by the same letter in a column were not significantly different according Tukey's test at 5% probability level.

Droplet coverage showed an inverse relationship with the contact angle, i.e., the treatments with the lowest contact angle had the highest coverage. The addition of Break-Thru® S240 resulted in a coverage of 49% to 56%, varying with the insecticide tested. Because coverage is related to the reduction of surface tension and contact angle, products that have this capacity can increase the coverage area and improve agricultural applications. The interference of surface tension with droplet coverage was also mentioned by Costa et al. (2017), who stated that reducing surface tension may result in increased droplet coverage.

Thus, the use of adjuvants in the spray solution can interfere with factors from the spray tank (pH and surface tension) to the drop reaching the leaf surface (contact angle and coverage). Its use associated with herbicides and fungicides has always been a common practice in Brazilian agriculture. However, adding insecticides has proven to be a viable alternative to avoid losses, improve product coverage, and consequently increase the biological efficiency of insecticides in the field (Melo et al., 2015).

The influence of rain on spraying has been studied for several years. However, its response varies according to the active ingredient and the formulation, so each case should be evaluated separately (Hunsche et al., 2007). Moreover, the characteristic deposit formation after application is one of the factors that interferes with the efficiency of agricultural products. In this context, the use of scanning electron microscopy allows for a qualitative analysis of the deposit properties and the interaction with the plant surfaces. Scanning electron microscope images showed that there is no deposit pattern for any of the insecticides alone or in association with the adjuvants (Figures 1 and 2).

It was possible to observe the residues in most of the images, which indicates that although rainfall considerably affects the removal of these active ingredients, not all the product on the leaf is removed by the impact of the rain drops. Wheat leaves had a higher amount of residue, which may be related to their higher water repellency (more hydrophobic leaves according to the contact angle data), and their greater vertical position compared to maize leaves. Furthermore, treatments with adjuvants had a higher amount of residue, which illustrates the effect of these products.

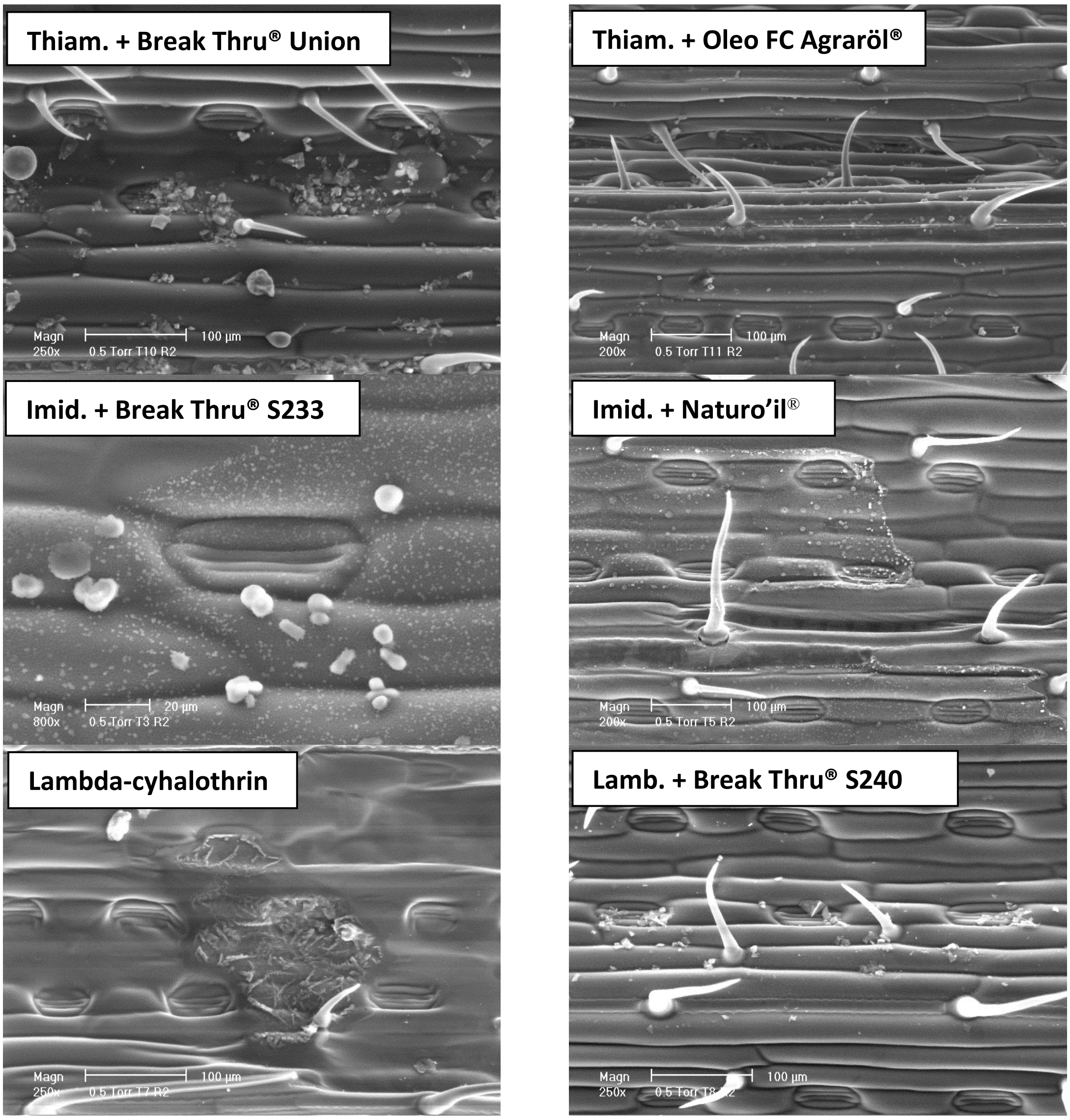
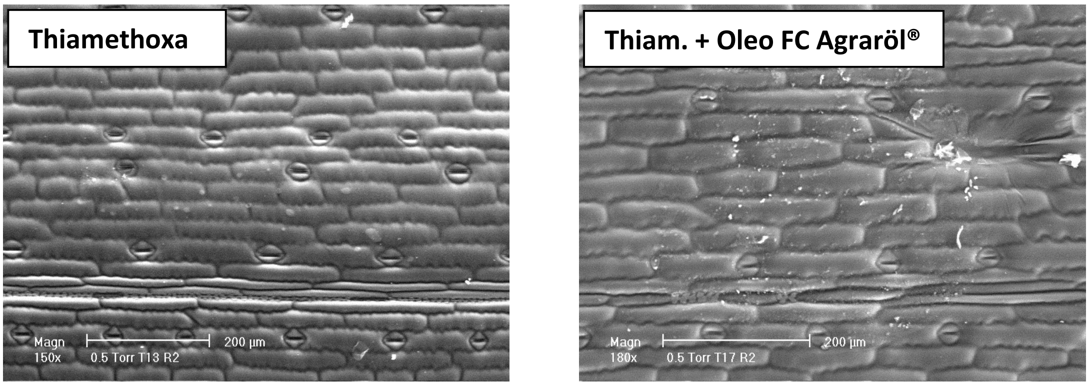


FIGURE 1. Scanning electron microscopy images of insecticide deposits associated with adjuvants after simulated rain (5 mm) on wheat leaves.

The use of adjuvants in association with insecticides can reduce surface tension and the contact angle between the leaf and droplet, increasing the coverage of drops and decreasing the deleterious effect of rain, without altering the pH of the spray solution. Therefore, its use, considering technical criteria when choosing the adjuvant, may lead to an improvement in the biological efficiency of insecticides used in wheat and maize crops.



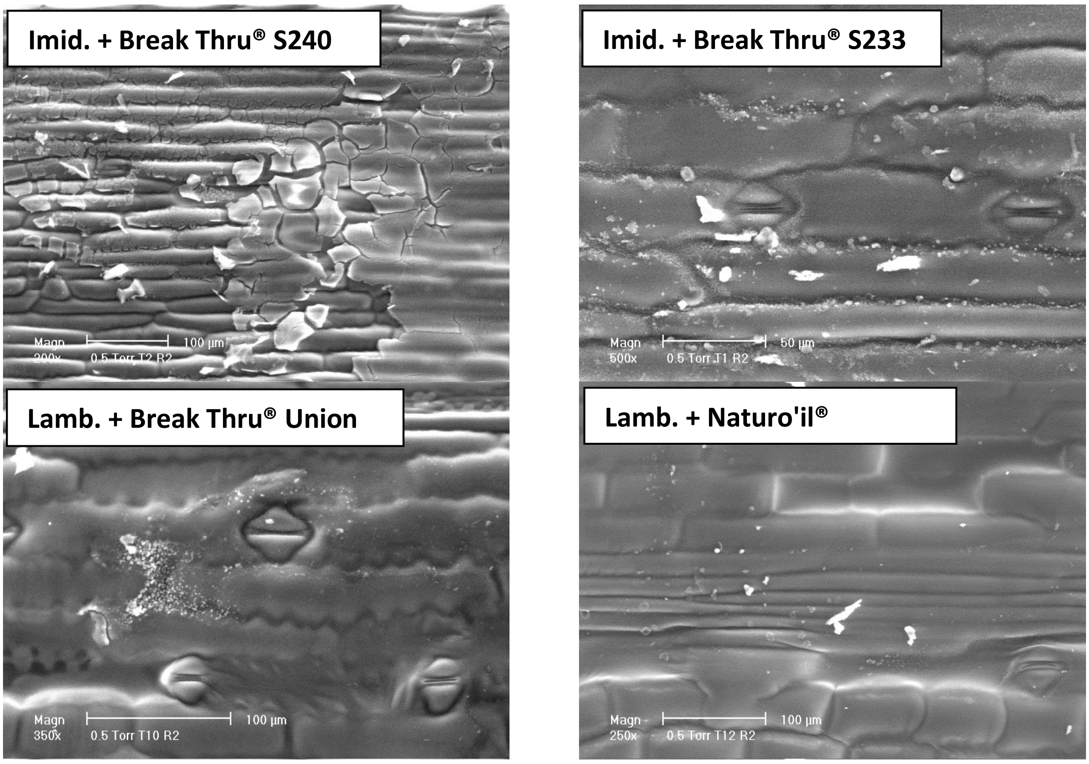


FIGURE 2. Scanning electron microscopy images of insecticide deposits associated with adjuvants after simulated rain (5 mm) on maize leaves.

CONCLUSIONS

The addition of Break-Thru® S240 and Break-Thru® S233 in the thiamethoxam, imidacloprid, and lambda-cyhalothrin insecticides resulted in lower values of surface tension and contact angle in wheat and maize leaves. Moreover, the coverage of these treatments was higher than that of the others, improving the quality of the applications.

The pH of the spray solution did not change with the adjuvants tested. The electron microscopy images showed residues of the active ingredient even after rain. Moreover, maize leaves retain less residue than wheat leaves and the use of adjuvants results in more residue on wheat and maize leaves.

The use of adjuvants associated with insecticides can improve the physicochemical properties of spray solutions. The addition of the adjuvant can be an alternative to improve the efficiency of insecticides, allowing the product to express its full control potential.

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