ea engenharia agrícola

Journal of the Brazilian Association of Agricultural Engineering

ISSN: 1809-4430 (on-line)

DROPLET SPECTRA AND SURFACE TENSION OF SPRAY SOLUTIONS BY BIOLOGICAL INSECTICIDE AND ADJUVANTS

Doi:http://dx.doi.org/10.1590/1809-4430-Eng.Agric.v37n2p292-301/2017

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ABSTRACT: Biological insecticides can be another pest control method used in integrated pest management; hence there is a demand for scientific information about these products to understand the appropriate application technique. The aim of this study was to evaluate the spray spectra data by different nozzle types using biological insecticide isolated and with adjuvants, as well the surface tension of these spray solutions. For droplet spectra data, a particle size analyzer was used and for surface tension of the spray solution, the method of pendant drop was used. Among the nozzles tested, ADI 11002 provided greater droplets median diameter volume, a lower percentage of droplets with diameters less than 100 µm and also the highest coefficient of uniformity. In general, the addition of adjuvants reduced the median droplet diameter and increased the proportion of drops susceptible to spray drift. By adding adjuvants to the spray solution there was a lower surface tension, which were dependent of the adjuvant chemical group. The Silwet L-77® adjuvant showed the lowest surface tension among the spray solutions evaluated.

KEYWORDS: coefficient of uniformity, volumetric median diameter, application technology, tensiometer.

INTRODUCTION

The current farming system uses chemicals for the control of pest/insects. However, this control method is often ineffective, which makes it necessary to find other alternatives that minimize the negative effects of the chemical method (DALVI et al, 2011). Several other techniques are being studied, including the use of biological insecticides (SILVA et al., 2008).

Biological control with entomopathogenic microorganisms, especially the *Bacillus thuringiensis* (Berliner) bacterium, has provided significant results in the control of pests/insect, with emphasis on those of the Lepidoptera order (BRAVO et al., 2011; VAN FRANKENHUYZEN, 2013).

However, these products have not yet been highlighted in insecticide sales, due to problems related to the lack of knowledge of the interaction of biological control agents with other synthetic products used in agricultural crops, such as herbicides, fungicides, adjuvants, etc. (ROSSI-ZALAF et al., 2008). There are other factors that promote variations in responses of biological control agents, such as the application technology used.

Regarding the quality of the spray, the most important aspects found in the literature are related to the droplet diameter and the coverage density of the droplets on the target (YU et al., 2009; BOLLER & RAETANO, 2011). Thus, the correct choice of the spray nozzle that provides droplets of diameters suitable for the different conditions and characteristics of the target can contribute to increase the penetration and deposition of crop protection products (CUNHA et al., 2011).

During the applications of crop protection products, the ideal is that the droplets spectrum is as homogeneous as possible; droplets of approximately the same size may be produced for the desired purpose (VIANA et al., 2010). For this, it is necessary to know the technical characteristics

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Received in: 1-11-2016
Accepted in: 10-13-2016

of the nozzles, aiming at the selection of those that provide the production of more uniform droplets and are less susceptible to drift losses (CUNHA et al., 2010).

Other authors have also highlighted that the addition of adjuvants to the spray solution is a strategy that often improves spray quality (SPANOGHE et al., 2007; CHECHETTO et al., 2013). However, there is still little scientific information about its action and the implications of its use on the control efficiency of the target, especially when using biological products, hindering its correct recommendation (DE SCHAMPHELEIRE et al., 2009).

Most of the adjuvant studies are related to herbicide applications. Hence the need for studies to evaluate the behavior of adjuvants in association with other classes of crop protection products. Due to the possibility of the use of biological insecticides within an integrated management of difficult-to-control pests, such as *Helicoverpa armigera*, in grain and fiber crops, there is a demand for scientific information to assist in the planning of the application technique.

In this context, the aim of this study was to analyze the influence of spray solutions with isolated biological insecticide and the same in association with different types of adjuvants in the droplets spectrum produced by different nozzles models and the influence of the spray solutions on the surface tension.

MATERIAL AND METHODS

The experiment was conducted in the laboratory of the Nucleus of Study and Development in Application Technology - NEDTA, belonging to the Crop Protection Department, São Paulo State University (Unesp), School of Agricultural and Veterinarian Sciences, in which the droplets spectra and the surface tension provided by the different treatments were carried out.

In the evaluations of the sprayed droplet spectrum, the experimental design was completely randomized, in a factorial scheme (6x4), with six replications. The interaction factors were composed of six spray solutions containing the formulation of the biological insecticide based on *Bacillus thuringiensis* (Best HD®, 23.7 g of a.i. L^{-1}, SC, Bio Control Farroupilha) and the combination of the same with five different types of adjuvants, and the second factor by four nozzles models (Table 1).By the association between the levels of the two factors studied (spray x spray nozzle model), twenty-four treatments were obtained (Table 1).

TABLE 1. Description of the proposed treatments (spray solution and nozzles types).

Sprays			
Active ingredient	Commercial brand	Chemical group	Dose (%)*
Bacillus thurigiensis var. kurstaki cepa HD-1 (Bt)**	Best HD®	Biological insecticide	0.75
Bt Lecithin and propionic acid	Li 700®	Lecithin and propionic acid	0.5
Bt + Polyether copolymer and silicone	Silwet L-77®	Organosilicone	0.1
Bt + Nonyl phenoxy poly (ethyleneoxy) ethanol	Agral®	Alkyl phenols ethoxylate	0.03
Bt + Mineral oil	Nimbus®	Aliphatic hydrocarbon	0.5
Bt + Modified vegetable oil	Aureo®	Soybean oil Methyl Ester	0.25
SPRAY NOZZLES			
Nozzle model	Kind of jet	Spray angle	Flow**
AXI 110025	Flat	110°	1.15
AXI Twin 12003	Twin flat	120°	1.39
ADI 11002	Flat with pre-orifice	110°	0.91
ATR 5.0	Hollow cone	80°	1.59

* The doses are recommended as a percentage considering volume by volume. ** The dose of Best HD® insecticide (*Bacillus thuringiensis* var. Kurstaki cepa HD-1) (Bio Control Farroupilha Ltda Laboratory) was set at 750 mL ha^{-1}. *** Flow in L min^{-1} considering the pressure of 60 lbf pol^{-2} and application volume of 100 L ha^{-1}.

We decided to use the Best HD® insecticide because field evaluations will be carried out in a second stage of this study, in which the efficiency of the biological product in the control of the caterpillar complex that occurs in the soybean crop will be evaluated, with and without addition of the adjuvants proposed in this study.

In the region of Morrinhos, GO, where the second stage of the experiment will be carried out, in the spraying of crop protection products, application volumes between 80 and 100 L ha^{-1} are adopted. Thus, in the treatments the product concentrations in the spray solutions and pressure at the spray nozzles (Table 1) of this study were adjusted to an application with a volume of 100 Lha^{-1}.

The spray solutions were prepared and packed in a stainless steel tank with a capacity of 10 liters. The tank was pressurized through compressed air controlled with a precision pressure regulator, manually operated, with an analogical gauge calibrated for the function. The activation of the spray flow was carried out by an electric switch that controls the solenoid valve of the hydraulic circuit.

The variables used for the droplet spectrum evaluation were the volumetric median diameter (Dv0.5), the sprayed droplets coefficient of uniformity (Coef. Unif.) and the percentage of droplet volume with diameters less than 100 µm (% < 100 µm). The coefficient of uniformity was obtained by the equation below, where:

$$Coef.Unif. = \frac{Dv0.9 - Dv0.1}{Dv0.5}$$

where,

Coef. Unif. = Coefficient of Uniformity (dimensionless),

Dv0.1; Dv0.5 and Dv0.9 refer to the droplet diameter (µm), such that 10, 50 and 90%, respectively, of the sprinkled volume is composed of diameter droplets smaller than it (ALMEIDA et al., 2016).

The droplet diameter was determined by laser diffraction during the passage of the spray droplets by the sampling region of a particle size analyzer (Mastersizer, Malvern Instruments Limited), adjusted to evaluate droplets of 0.5 to 900 µm. The data decoding, according to the algorithm elaborated for the characterization of the droplets diameter by laser diffraction, were processed and tabulated directly by the Mastersizer S® program, version 2.19.

The different types of spray nozzles were installed on a radial conveyor positioned at 0.5 m from the laser beam. Two samples of each nozzle model were used for the analyzes and for each one of them three repetitions of the sprinkled jets of each spray were carried out, totaling six repetitions per treatment.

A completely randomized design with six treatments and four replications was used in the evaluation of the surface tension provided by the different spray solutions. The treatments were represented by six spray solutions, one of them containing the formulation of the biological insecticide based on *Bacillus thuringiensis* (Bt) (Best HD®, 23.7 g of a.i. L^{-1}, SC, Bio Control Farroupilha) and the others the combination of the biological insecticide and five different types of adjuvants as described in Table 1.

Four droplets were obtained from each treatment, each representing one replicate. The droplets were formed with the aid of 500 µL graduated microsyringe, dispensing volumes of approximately five µL for each replicate.

The surface tension measurements of each treatment were carried out every second in a total time of 60 seconds by an automated tensiometer, OCA-15 plus model (Dataphysics Germany), in which the surface tension was determined by the droplet method. The image of the droplet is captured by a camera and the equipment analyzes the droplet shape hanging on the end of a needle attached to the emission syringe of the liquid to be analyzed by axisymmetric drop shape analysis (ADSA).

A specific software that uses an ideal position as reference line in the image field is used to identify the key point for the beginning of the recording of the images. The surface tension is determined through digitizing and analyzing the droplet profile, using for adjustment the Young-Laplace equation.

The data obtained after confirming the normality assumptions of the residue (Kolmogorov-Smirnov) and homogeneity of variances (Levene) were submitted to the F test of variance analysis (ANOVA). The averages were compared by the Fisher test (LSD, $P \leq 0.01$) when observed significant effect of the treatments (F, $P \leq 0.01$). All statistical analyzes of the data were processed by the SAS 9.1 program (SAS Institute, Cary, N.C., USA).

RESULTS AND DISCUSSION

There was effect of the spray solutions and spray nozzles on the percentage of droplet volume with diameters less than or equal to 100 μm (% droplets ≤ 100 μm), droplets volumetric medium diameter (Dv 0.5) and also on the coefficient of uniformity (Coef. Unif.). However, the significant interaction of these factors did not occur for the coefficient of uniformity, indicating that for this parameter, adjuvants and spray nozzles are independent factors (Table 2).

TABLE 2. Analysis of variance with F values for droplets smaller or equal than 100 μm (% droplets ≤ 100 μm), volume median diameter (Dv 0.5) and coefficient of uniformity (Coeff. Unif.).

Variation source	GL	% ≤ 100 μm	Dv 0.5 (μm)	Coef. Unif.
Sprays	5	24.25**	6.64**	16.11**
Nozzles	3	374.47**	271.52**	18.70**
Sprays x Nozzles	15	12.88**	6.13**	1.42^{ns}

By the F test, ** significant at 1% probability, ^{ns} not significant.

Table 3 shows the average values of the % of droplets ≤ 100 μm obtained for the different spray solutions and nozzles. Among the nozzles models tested, the ADI 11002 provided a lower percentage of droplets with a diameter equal or less than 100 μm or in all spray solution combinations (Table 3). This result suggests, according to FIETSAM et al. (2004), that this nozzle model offers greater safety in the applications in relation to the drift risk.

OLIVEIRA et al. (2015) report that one of the factors that indicates the risk of nozzle drift is the percentage of droplet volume with a diameter lower than 100 μm. ARVIDSSON et al. (2011), also related drift with drops percentage less than or equal to 100 μm.

The result described above is justified because the ADI 11002 nozzle model is considered to be drift reducing because it has a pre-orifice preceding the final outlet of the sprayed liquid at the spray nozzle. This pre-orifice produces pressure drop in pressurization by reducing the rate of liquid passage through the pre-orifice, resulting in an increase in droplet diameter. Similar results were found by CUNHA et al. (2007), noting that the models of drift reducing nozzles (API and ADI) increased the diameter of the droplets produced.

Still analyzing the results obtained with the ADI 11002 nozzle model, the addition of adjuvants associated with Best HD® increased the percentage of droplets less than or equal to 100 μm (Table 3). Likewise, this result was observed for the AXI 110025 nozzle model (Table 3). Therefore, with this nozzle models, the addition of the adjuvants to the insecticidal spray solution increased the drift risk.

TABLE 3. Average values of the percentage of spray volume composed of droplets with a diameter less than or equal to 100 μm (% ≤ 100 μm) combined with different spray solutions and nozzle types.

Droplets percentage with a diameter of less than 100 μm					
	Nozzles				
Sprays	AXI twin 12003	AXI 110025	ATR 5.0	ADI 11002	Msd
Best HD®	29.67 Dbc*	25.66 Ca	21.35 Ba	11.23 Aa	2.98
Best HD + Agral®	26.95 Cab	33.38 Dc	23.03 Ba	15.24 Ab	2.52
Best HD + Aureo®	23.72 Ba	31.34 Cbc	27.33 Bb	16.69 Ab	3.91
Best HD + Nimbus®	25.75 Bab	30.87 Cb	29.65 BCc	16.52 Ab	4.82
Best HD + Silwet L-77®	33.03 Cc	31.99 Cbc	23.04 Ba	16.30 Ab	2.44
Best HD +Li700®	28.57 Babc	32.93 Cbc	31.89 Cd	19.37 Ac	2.67
Msd	5.27	2.20	2.13	2.11	
C.V %		8.05			

* Averages followed by the same lowercase letter in the column and upper case in the lines do not differ statistically from each other by the Fisher test at 1% (LSD, $P \leq 0.01$). C.V = Coefficient of Variation; Msd: Minimal significant difference.

Differing results were found by FERREIRA et al. (2011) as the addition of adjuvants in the spray decreased the percentage of droplets with diameter smaller than 100 μm using drift reducing nozzle models (AI 110015 and TTI 110015).

With the ATR 5.0 nozzle model, the addition of the Agral® and Silwet L-77® adjuvants in the spray solution did not differ significantly from the spray solution applied only with the biological insecticide in relation to the droplets percentage with a diameter of 100 μm or less. On the other hand, the other groups of adjuvants tested increased the proportion of the droplets more susceptible to drift (Table 3).

When analyzing the AXI twin 12003 nozzle model, we found that the addition of the Aureo adjuvant in the spray solution provided a reduction in the droplet percentage less than or equal to 100 μm to the spray solution applied without adjuvant. Thus, with this spray nozzle model, the use of this adjuvant is justified by decreasing the potential risk of drift (Table 3).

Some patterns were created, and the droplets spectra of a spray were classified into extremely thin (<60 μm), very thin (61-105 μm), thin (106-235 μm), medium (236-340 μm), thick (341-403 μm), very thick (404-502 μm), extremely thick (503-665 μm) and ultra thick (> 665 μm) (ASABE, 2009).

By the values of the volumetric median diameter (Dv 0.5) obtained in the different treatments, we verified that the spectra of the sprayed droplets are classified in the thin class (Table 4).This result was expected because all the models tested, at the pressure at which the study was carried out, should produce thin drops according to the manufacturer's indication.

TABLE 4. Average values of volume median diameter (DV 0.5) (μm) by different nozzle types and spray solution.

Volumetric median diameter (DV 0.5)					
	Nozzles				
Sprays	AXI twin 12003	AXI 110025	ATR 5.0	ADI 11002	Msd
Best HD®	129.46 Db*	143.37 Ca	156.34 Ba	204.07 Aa	9.46
Best HD + Agral®	136.19 Cab	128.10 Cc	154.66 Bab	188.56 Ab	10.91
Best HD + Aureo®	157.99 Ba	135.18 Cb	149.29 BCc	186.12 Ab	21.89
Best HD + Nimbus®	148.96 Bab	135.12 Bb	140.91 Bd	181.81 Ab	20.12
Best HD + Silwet L-77®	126.90 Cb	131.30 Cbc	150.98 Bbc	181.61 Ab	7.27
Best HD + Li700®	140.20 Bab	134.71 Bb	137.54 Bd	178.49 Ab	6.99
Msd	24.26	5.24	4.71	10.28	
C.V %		5.63			

* Averages followed by the same lowercase letter in the column and upper case in the lines do not differ statistically from each other by the Fisher test at 1% (LSD, $P \leq 0.01$). C.V = Coefficient of Variation; Msd: Minimal significant difference.

The volumetric median diameter of the droplets produced by the ADI 11002 nozzle model was higher compared to the other nozzle models tested in all spray solution combinations (Table 4). This means that under the most adverse conditions of temperature and humidity for applications, among the models of nozzles studied, the ADI 11002 is the most suitable. However, the droplets spectrum produced by the different models studied belongs to the same class according to ASABE (2009).

According to GABRIEL & BAIO (2013) for a suitable application of crop protection products is necessary to adjust the diameter of the sprayed droplets to ensure, at the same time, biological efficacy and environmental safety, according to the meteorological conditions. Thus, the volumetric median diameter should be evaluated with caution, because there is a narrow boundary between the droplets diameter that provides good coverage and susceptibility to drift.

For the AXI 110025, ATR 5.0 and ADI 11002 nozzles models, the highest values of median volumetric diameter were obtained with the spray containing only the biological insecticide. Generally, the addition of adjuvants reduced the droplets median diameter and, consequently, increased the proportion of droplets susceptible to drift (Tables 3 and 4).

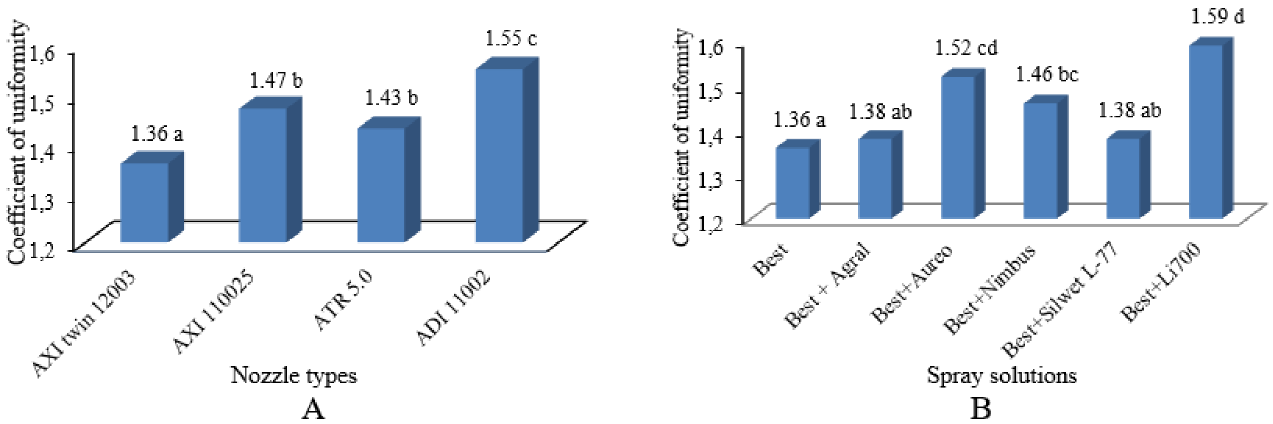
In contrast to the preceding result, the AXI twin 12003 model provided higher volumetric median diameter values when adjuvants were added in the spray solution, except for the Silwet L-77® adjuvant, which reduced the volumetric median diameter and increased the droplets percentage susceptible to drift (Table 3 and 4).

Differing results were found by SPANOGHE et al. (2007), who observed an increase in droplet diameter when the organosilicone Silwet L-77® adjuvant was added to the spray solution. In another study, MOTA & ANTUNIASSI (2013), evaluating the influence of different adjuvants (Nimbus®, Nortox®, Li-700®, Antideriva®, Agral®, In-Tec®, TA 35®, Silwet L-77®) in the droplet spectrum using an air induction nozzle (GA 11003), concluded that all adjuvants tested increased the value of the median volume diameter in spray solution composed only of water.

Possibly, the divergent results found for the Dv 0.5 variable is justified because, according to SPANOGHE et al. (2007), changes in the droplet spectrum are observed with the addition of adjuvants in the spray solution, but they occur in function of the adjuvant class and the nozzle model combined with the applied pressure.

The coefficient of uniformity expresses the uniformity of the sprayed droplets spectrum. The lower coefficient of uniformity value is, the smaller will be the spray size, and the greater will be the uniformity. Thus, the homogeneous droplets spectrum has the value of this coefficient closer to zero.

In this study, the AXI twin model 12003 showed the lowest coefficient of uniformity (1.36) in relation to the other models tested, indicating the production of more uniform drops. The inverse is true for the ADI 11002 model, which presented the highest coefficient of uniformity (1.55) and, therefore, the most heterogeneous droplet spectrum (Figure 1A).



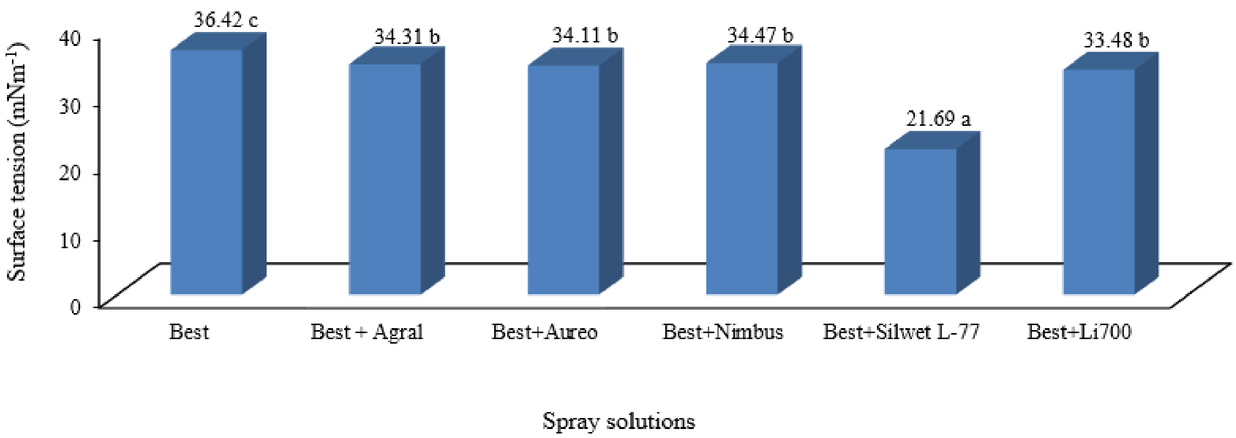
Averages followed by the same letter between treatments did not differ statistically from each other by the Fisher test at 1% (LSD, $P \leq 0.01$).

FIGURE 1. Coefficient of uniformity with different nozzle types and spray solutions.

Despite the greater uniformity of the droplet spectrum produced by the AXI twin 12003 nozzle model, its use must be made with criteria due to the volumetric median diameter produced by this model, avoiding adverse climatic conditions to avoid loss of crop protection product to the environment.

The coefficient of uniformity of Best HD ®, Best HD ® + Agral ® and Best HD ® + Silwet L - 77 ® spray solutions were significantly similar and smaller than the others, which indicates that the droplet size distribution is more homogeneous for these spray (Figure 1 B). However, the addition of the adjuvants did not increase the uniformity of the sprayed droplets spectrum in relation to the spray containing only the biological insecticide.

In the analysis of surface tension, the spray solution composed only with the biological insecticide provided a higher value of surface tension in relation to the other sprays (Figure 2).Considering that the water surface tension, the most used vehicle in agricultural applications is around 72.6 mNm^{-1}, the addition of Best HD® insecticide alone was enough to reduce the water surface tension by half (Figure 2).



Averages followed by the same letter between treatments did not differ statistically from each other by the Fisher test at 1% (LSD, $P \leq 0.01$).

FIGURE 2. Average values of surface tension of the spray solutions analyzed.

By adding adjuvants to the spray solution, the spray surface tension was reduced to values that were dependent on the chemical group of the adjuvant used. The spray with the lowest surface tension value was added to the Silwet L-77® adjuvant (21.69 mNm^{-1}) (Figure 2).

Although the Silwet L-77® organosilicone adjuvant is the most efficient in reducing the surface tension of the sprayed solution, the addition of the same to the spray solution increased the percentage risk of drift in relation to the other treatments as described in Table 3.

According to XU et al. (2011), the surface tension interferes with the coverage provided by the sprayed solution. According to these authors, a greater coverage of the target per droplets can be obtained by reducing the surface tension. This occurs because, according to SPANOGHE et al. (2007), the adjuvants that reduce the spray surface tension consequently provide an increase of the droplets contact surface with the target. In this regard, the addition of the Silwet L-77® adjuvant to the spray will possibly also provide greater coverage of the sprayed solution.

CONCLUSIONS

1. The ADI 11002 nozzle model has a less uniform droplet spectrum and less drift compared to the AXI twin 12003, AXI 110025 and ATR 5.0. models.

2. The spray solutions containing adjuvant change the volumetric median diameter and droplet percentage less than or equal to 100 μm, but it is a dependent factor on the spray nozzle model.

3. The spray solution surface tension characteristics can be altered by the addition of adjuvant, but dependent on the chemical group to which they belong.

ACKNOWLEDGEMENTS

We thank to the NEDTA, belonging to the Crop Protection Department, São Paulo State University (Unesp), School of Agricultural and Veterinarian Sciences, Jaboticabal, to the Farroupilha group and to Jacto Agricultural Machinery for the support granted for the execution of this research.

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