

Full Length Research Paper

Effects of agricultural spray adjuvants in surface tension reduction and spray retention on *Eucalyptus* leaves

Evandro Pereira Prado^{1*}, Carlos Gilberto Raetano², Mario Henrique Ferreira do Amaral Dal Pogetto³, Rodolfo Glauber Chechetto⁴, Pedro José Ferreira Filho⁵, Anderson Chagas Magalhães¹ and Celso Tadao Miasaki¹

¹Department of Agronomy Engineering, São Paulo State University - College of Technology and Agricultural Sciences (FCAT), Dracena, SP - Brazil.

²Department of Plant Protection, São Paulo State University - College of Agronomy Science (FCA) - Botucatu, SP - Brazil.

³Dow AgroSciences Industrial Ltda., Mogi Mirim, SP - Brazil.

⁴Department of Rural Engineering, São Paulo State University/ College of Agronomy Science (FCA) - Botucatu, SP - Brazil.

⁵Department of Environmental Science, Federal University of São Carlos/ Sorocaba, SP – Brazil.

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Agricultural spray adjuvants (ASA) are widely used in pesticide applications to enhance the effective control of pest, weed and disease. The aim of this research was to investigate the effects of ASA used in Brazil agriculture on surface tension reducing capacity and foliar spray retention on different *Eucalyptus* species. Static surface tension of adjuvants at concentrations of 0.001, 0.0025, 0.005, 0.01, 0.025, 0.05, 0.1, 0.25, 0.5, 1.0 and 2.0% v v⁻¹ were determined by the drop weight method. Spray retention on *Eucalyptus* leaves using ASA were performed at concentrations of 0; 0.005; 0.01; 0.05; 0.1; 0.5; 1.0 and 2.0% v v⁻¹. The ASA assessed were: vegetal oil, mineral oil, spreader-sticker and a drift reducing based on soybean lecithin more propionic acid. All ASA adjuvants reduced surface tension of aqueous solutions. Heptomethyltrisiloxane (HT) provided the best performance on decrease of the surface tension reaching values below to 20 mN m⁻¹ at concentration of 0.05% v v⁻¹. Spray retention was influenced by *Eucalyptus* species, adjuvants types as well as adjuvants concentrations. The increase of ASA concentration contributed to reduce spray retention. Different characteristic of the adjuvants on spray retention was observed in different *Eucalyptus* leaves species. *Eucalyptus grandis* and *Eucalyptus torelliana* species showed respectively the lower and higher spray retention values. The mineral oil and vegetal oil provide the higher and HT the lower level of spray retention. Application at high spray volume must be carefully performed to avoid losses by run-off when added some ASA.

Key words: Foliar retention, surfactant, application techniques, chemical control.

INTRODUCTION

Despite the negative perception of the society, chemical control using pesticides are still going to be used for many decades to ensure the food supply for the ever growing world population. One of the possible reasons for this is that alternative methods for plant protection are either inefficient or too costly for farmers (Wang and Liu, 2007).

Correct selection of application equipment and spray adjuvants are powerful tools to maximize pesticide efficacy, reduce detrimental environmental effects and improve the economic viability of the farmer (Dorr et al., 2014). Adjuvants are used to modify the physical, biological and chemical properties of spray mixtures to improve chemical performance (Kudsk and Mathiassen, 2007) impacting on viscosity, surface tension, contact angle, droplet retention, and deposits on the target (Gitsopoulos et al., 2014; Lin et al., 2016; Stock and Brings, 2000; Wang and Liu, 2007). The adjuvants may also influence spray atomization and formation, which is important because each type of application requires a certain optimum droplet size for its biological activity (Gimenes et al., 2013).

The spray efficacy depends on the amount of pesticide solution retained on leaves surface. In most cases, the wax on a leaf surface acts as a substantial barrier to wetting for having hydrophobic characteristic, which can make spray applications ineffective and increasing the environmental pollution (Tang et al., 2008). According to Lin et al. (2016) surfactant may not only effectively reduce the surface tension of solution but also dissolve the epicuticular waxes on the leaf surfaces and consequently, the addition of surfactant could strongly enhance the spreading ability of droplets. In order to improve targeting of the spray, it is important to know how formulation/liquid properties interact with the characteristics of the target plant to affect spray deposition (Butler-Ellis et al., 2004).

During disease and pest control applications, surfactant additives are commonly used to improve the efficiency of pesticides (Lin et al., 2016). Several studies showed that surfactants can greatly reduce surface tension and maximize the spread, penetration, and absorption efficacy of pesticides on leaf surfaces (Gimenes et al., 2013; Gitsopoulos et al., 2014; Lin, et al., 2016). Besides foliar uptake and biological efficacy of the active ingredients are improved with the surfactant by overlapping some leaf barriers such as cuticular membrane, trichomes and others features that decrease

droplet deposition, spread and uptake of pesticide solutions.

The global planted area of *Eucalyptus* crop has increased significantly in the last decades. The trend in areas where Eucalypt are being grown in plantation is that pest and pathogen problems are increasing (Wingfield et al., 2003). Despite the contamination problems presented by the use of pesticide, this practice is required in various situations to control satisfactory pest and disease. The addition of corrected adjuvants on tank-mixture could enhance the performance of pesticides on pest, weed and disease control. The comprehension of adjuvants behavior is fundamental to prescribe the correct product/concentration maximizing pesticide control, avoiding losses and environmental contamination.

The aims of this research were to identify (1) the most effective and economic adjuvants/concentrations in reduce surface tension of water and (2) the spray retention capacity of adjuvants/concentrations on leaves of different *Eucalyptus* species used as a target surface.

MATERIALS AND METHODS

Surface tension determination

Static surface tension (SST) assessments were performed at the Laboratory of Pesticide Application Technology Laboratory, at the College of Agricultural Sciences UNESP - Botucatu, SP - Brazil. Details of the adjuvants composition selected for evaluations are summarized in Table 1. The adjuvants were tested at 11 concentrations levels (0.001; 0.0025; 0.005; 0.01; 0.025; 0.05; 0.1; 0.25; 0.5; 1.0 and 2.0% v v⁻¹) plus an additional treatment with no adjuvant (distilled water).

The SST of aqueous solution was determined by gravimetric method quantifying the weight of the droplets formed at the tip of the glass capillary burette (50 mL capacity) placed in a vertical direction. The droplets free detached at the tip of glass fall into a 25 mL Becker containing 10 mL of vegetal oil to avoid solution evaporation losses. Becker was allocated within the analytical precision balance with 0.1 mg accuracy (Marte, model AY 220, São Paulo, SP, BR) and the tip of the glass burette was kept 0.10 m above Becker.

The burette was adjusted to formed droplets at the time between 15 to 20 s and the liquid column was kept at 25 mL of graduation scale. The test was carried out at the room temperature of 25±1°C and air relative humidity of 60±10%. Fifteen droplets were measured per treatment and each droplet considered a repetition. All adjuvants solutions were prepared with distilled water assuming a SST of 72 mN m⁻¹ at 25°C (Vazquez et al., 1995). Since the weight concentrations of surfactant were low, both the liquid density and viscosity of the surfactant solution were considered similar to the distilled water. The average droplets weight data were converted into surface tension according to Equation 1.

*Corresponding author. E-mail: eprado@dracena.unesp.br. Tel: (+55) 18 3821 7485.

Table 1. Agricultural adjuvants descriptions.

Adjuvants	Content	Dosage (v v ⁻¹)	Supplier
Haiten	nonionic spreader sticker surfactant - 200 g L ⁻¹ polyoxyethylene alkyl phenol ether	0.01-0.015%	Arysta LifeScience
Veget'Oil	70 g L ⁻¹ emulsifier and 930 g L ⁻¹ vegetal oil	0.2-1%	Oxiquímica Agrociência
Agral	nonionic spreader sticker surfactant - 200 g L ⁻¹ nonylphenoxy polyethoxy ethanol	0.03-0.05%	Syngenta Proteção de Cultivos Ltda
Silwet L-77 AG	superspreader surfactant - 1000 g L ⁻¹ Polyalkyleneoxide modified heptomethyltrisiloxane	Until 0.1%	Momentive Performace Materiais Industrias de Silicone
Li 700	acidifying and penetrating surfactant - 350 g L ⁻¹ soyal phospholipids and 350 g L ⁻¹ propionic acid	0.5%	De Sangosse Agrochemical Ltda,
Iharol	760 g L ⁻¹ mineral oil	0.5%	Iharabras S.A.

$$\gamma = (w_t \times 72)/w_w \quad (1)$$

Where: γ = Surface Tension (mN m⁻¹); w_t = Droplet weight (g) of treatments and w_w = Droplet weight (g) of distilled water.

Statistical analysis of each adjuvant data was performed using the SAS program (SAS Institute, 1995) and regression analysis was used the Mitscherlich equation model, denoted by $\gamma = a[1-10^{-(x+b)}]$. Mitscherlich model was modified to get better fit to the data (Silva et al., 2006), expressing relationship between surfactant input and surface tension reducing.

$$\text{Modified model used: } \gamma = \gamma_{dw} - a[1-10^{-cx}]$$

Where γ : surface tension in mN m⁻¹; γ_{dw} : distilled water surface tension (72 mN m⁻¹); a : maximum horizontal asymptote attainable in the original model; c : curve concavity representing the efficiency of the surfactant. Higher value of this parameter represents the most effective the surfactant is to attainable the minimum surface tension in a lower concentration; x : surfactant concentration (%v v⁻¹); $\gamma_{dw} - a$: corresponds to the minimum surface tension reached by adding surfactant in aqueous solution. To compare the effects between adjuvants on SST a factorial design 6 adjuvants x 11 concentrations were analyzed.

Spray retention determination

Eucalypti leaf spray retention was evaluated by the same adjuvants used to determine SST. The experimental design was done in a factorial 6 (adjuvants) x 5 (*Eucalyptus* species: *Corymbia citriodora*, *Eucalyptus urophylla*, *E. camaldulensis*, *E. torelliana* and *E. grandis*) x 8 (adjuvants concentrations at 0; 0.005; 0.01; 0.05; 0.1; 0.5; 1.0 and 2.0% v v⁻¹) with seven replicates (each leaf was considered one repetition). *Eucalyptus* mature leaves were classified by Gaskin et al. (2005) as an easy moderate-to-wet. The *Eucalyptus* leaves were collected from a four years old plants grown outdoor located at Plant Protection Department - UNESP – Botucatu, Brazil. *Eucalyptus* branches were removed from the trees and immediately carried to the laboratory. At the time of tests the branches base was kept into a 10 L pot filled with water to avoid leaves humidity losses. *Eucalyptus* mature leaves were removed from the branches and determined the weight by analytical precision balance (Marte, model AY 220, São Paulo, SP, BR). Afterwards the leaves were placed in a vertical position (holding the

petiole leaves by hand) and the solution was sprayed by an adaptation of Potter Spray Tower (Burkard Scientific, Uxbridge, UK) sprayer at 100 kpa pressure, typically producing fine spray droplet diameter. The spraying was performed on both sides of leaves at the distance of 0.3 m until run-off point was reached at temperature of 21 ± 2°C and 50 ± 7% air relative humidity. After spraying the leaves were again weighted and by weight difference the spray solution retained on each leaf was determined. Area of each target was measured using a foliar area meter (Licor Inc., Li 300, Lincoln, NE, US). The weight value was divided by the foliar area of each leaf and the retention results expressed in µg cm⁻².

Statistical analysis

Spray retention and surface tension dates were subject to analysis of variance (ANOVA) using SISVAR Statistical Software (Ferreira, 2011). Fisher's least significant difference (LSD) was calculated to identify significant difference between mean treatments at 5% probability.

RESULTS AND DISCUSSION

Surface tension study

The median values of surface tension (mN m⁻¹) of adjuvants on 11 concentrations levels are showed in Table 2. Significantly difference were verify in the interaction adjuvants x concentrations (F=77.4; p<0.001). All aqueous solutions containing the adjuvants reduced the surface tension of distilled water with the increased concentration. The adjuvant heptomethyltrisiloxane (HT) presented the best performance to reduce surface tension of aqueous solution follow by polyoxyethylene alkyl phenol ether (PAPE), mineral oil (MO), nonylphenoxy polyethoxy ethanol (NPE), vegetal oil (VO) and soyal phospholipids and propionic acid (SPPA) (Table 2). Stevens et al. (1993) verify a more rapid and more extensive reduction of surface tension on aqueous solution when organosilicone surfactant is used.

The surface tension value of HT (18-19 mN m⁻¹) for the

Table 2. Influence of adjuvants and concentrations on aqueous solution surface tension.

Concentrations (% v v ⁻¹)	Surface tension (mN m ⁻¹)					
	PAPE	VO	NPE	HT	SPPA	MO
0			72.0			
0.001	70.6 (0.3) ^{1b}	70.3 (0.2) ^b	70.0 (0.2) ^b	63.6 (0.4) ^a	70.7 (0.2) ^b	69.2 (0.5) ^b
0.0025	70.1 (0.2) ^c	69.3 (0.4) ^{bc}	68.8 (0.2) ^{bc}	60.8 (0.3) ^a	70.4 (0.3) ^c	68.4 (0.4) ^b
0.005	69.1 (0.4) ^c	68.5 (0.9) ^{bc}	67.4 (0.3) ^b	53.3 (1.0) ^a	69.0 (0.3) ^c	68.1 (0.2) ^{bc}
0.01	66.2 (0.2) ^d	67.7 (0.9) ^{de}	64.2 (0.3) ^c	33.9 (0.8) ^a	67.9 (0.3) ^e	61.6 (2.6) ^b
0.025	50.3 (0.5) ^b	61.2 (4.6) ^d	52.2 (0.5) ^c	23.0 (0.4) ^a	64.2 (0.2) ^e	52.3 (0.7) ^c
0.05	38.9 (0.3) ^c	55.6 (1.4) ^e	37.3 (0.3) ^b	19.9 (0.3) ^a	54.6 (1.3) ^e	47.1 (0.4) ^d
0.1	31.8 (0.3) ^b	55.0 (1.9) ^f	33.4 (0.3) ^c	19.2 (0.2) ^a	49.4 (0.2) ^e	38.1 (1.0) ^d
0.25	27.2 (0.7) ^b	44.8 (1.6) ^f	30.1 (0.3) ^c	18.5 (0.2) ^a	42.4 (0.5) ^e	32.1 (0.7) ^d
0.5	27.4 (1.0) ^b	31.7 (0.7) ^d	29.3 (0.2) ^c	18.6 (0.3) ^a	38.7 (0.4) ^e	29.7 (0.4) ^c
1.0	28.2 (0.3) ^b	31.3 (0.4) ^c	29.0 (0.2) ^b	18.5 (0.3) ^a	37.0 (0.8) ^d	27.7 (0.4) ^b
2.0	28.3 (0.2) ^b	30.5 (0.4) ^c	29.0 (0.1) ^{bc}	18.3 (0.2) ^a	34.1 (0.9) ^d	27.4 (0.5) ^b
LSD ²			1.61			

¹Standard deviation/²Least significant difference. Different superscript letters indicate significance difference among adjuvants at the same concentration (LSD, $p < 0.05$), PAPE - polyoxyethylene alkyl phenol ether, VO - vegetal oil, NPE - nonylphenoxy polyethoxy ethanol, HT - heptomethyltrisiloxane, SPPA - soyal phospholipids and propionic acid, MO - mineral oil.

aqueous solution is significantly lower compared to the other adjuvants tested. Lower surface tension of trisiloxanes surfactant (approximately 22 mN m⁻¹) measured by a pendant drop technique is reported by Wang et al. (2015). The surface tension of HT was a little lower than reported by those authors, probably due to the different technique performed in this research. SPPA is the adjuvant with least ability to lowering surface tension of aqueous solutions at all adjuvants studied. Similar to this research Nairn et al. (2014) observe surface tension values approximately of 19 mN m⁻¹ with HT and 31.5 mN m⁻¹ with (alcohol ethoxylate both at concentration of 0.1%. Iost and Raetano (2010) report approximately surface tension values of 19 and 33 mN m⁻¹ for aqueous solutions of the adjuvants HT and SPPA respectively using the same technique adopted in this research.

The SST became steady state at concentrations up to 0.25% to PAPE, 0.5% to VO, 0.25% to NPE, 0.05% to HT and 1% to MO (Table 2). These minimum concentrations points are considered to be the Critical Micelle Concentration (CMC) (Aliverdi et al., 2009). When a surfactant concentration is above the CMC the surfactants produce aggregates called micelles and generally the minimum equilibrium surface tension is achieved (Hazen, 2000). An increase of concentration above the CMC will not modify the surface tension. The CMC of the adjuvant SPPA, apparently, was not reached by the concentrations tested in this research as illustrated in Table 2.

Variance analysis results of surface tension by Mitscherlich modified model are shown in Table 3. High coefficient of determination (R^2) values and low coefficient of variation (CV) indicate that these equations provided

good models profiles and accurate estimate of SST of the aqueous solutions containing adjuvants. According to parameter "c" the adjuvant HT is the most efficient adjuvant to reach the minimum surface tension in a lower concentration (46.3) followed by NPE (11.9); PAPE (11.3); MO (7.8); SPPA (4.6) and VO (4.3) (Table 3).

Linking the minimum surface tension values adjusted by the Mitescherlich model can be verified that HT presents the lower SST to 18.2 mN m⁻¹ followed by PAPE (27.1 mN m⁻¹); NPE and MO with the same value (28.8 mN m⁻¹); VO (30.3 mN m⁻¹) and SPPA (36.5 mN m⁻¹) as shown in Table 3. Stevens et al. (1993) demonstrate that the reduction in surface tension of aqueous solutions is the most important factor controlling droplets adhesion and the organosilicone surfactants must have an advantage because they reduce aqueous surface tension more quickly than conventional hydrocarbon-based surfactants. HT showed lower surface tension values (33.9 mN m⁻¹) at concentration of 0.01% v v⁻¹ (Table 2). Despite the adjuvants NPE and MO reached the same minimum values of surface tension they presented different values of "c" parameter of 11.9 and 7.8 respectively (Table 3).

Spray retention study

A regression analysis among adjuvants concentrations did not fit well in any model with coefficient of determination low than 40% and for this reason a mean comparison was analyzed.

The average retention on leaves provided by all adjuvants and all concentrations on Eucalyptus species

Table 3. Parameters of regression analysis obtained by Mitscherlich modified model equation ($y = \gamma_{dw} - a[1 - 10^{-cx}]$) to the adjuvants.

Adjuvants	a	c	MST ^a (mN m ⁻¹) $\gamma_{dw} - a$	F _{regression}	R ²	CV ^b (%)
Polyoxyethylene alkyl phenol ether	44.9	11.3	27.1	67.166**	0.993	1.77
Vegetal oil	41.7	4.3	30.3	22.236**	0.965	2.29
Nonylphenoxy polyethoxy ethanol	43.2	11.9	28.8	33.022**	0.985	3.79
Heptomethyltrisiloxane	53.8	46.3	18.2	31.898**	0.992	1.25
Soyal phospholipids and propionic acid	35.5	4.6	36.5	41475**	0.977	2.82
Mineral oil	43.2	7.8	28.8	28.178**	0.980	3.33

^aMinimum surface tension/ ^bCoefficient of variation / **p<0.001/ R²: Coefficient of determination.

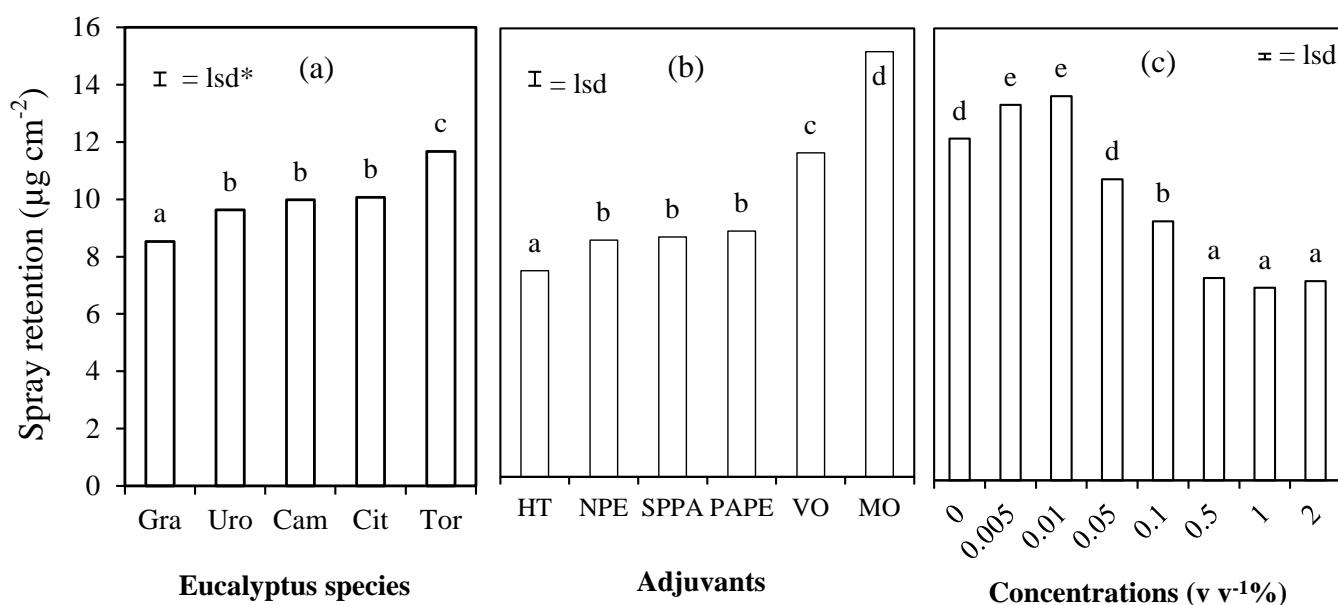


Figure 1. Spray retention on leaves of *Eucalyptus* species using six adjuvants at eight concentrations (a). Spray retention provided by different adjuvants from eight adjuvants concentrations on five *Eucalyptus* leaves (b); Spray retention using eight concentrations of adjuvants from six adjuvants at five *Eucalyptus* leaves. (c). *Eucalyptus* species: *E. grandis* (Gra), *E. urophylla* (Uro), *E. camaldulensis* (Cam), *C. citriodora* and *E. torelliana* (Tor). Adjuvants: heptomethyltrisiloxane (HT), nonylphenoxy polyethoxy ethanol (NPE), soyal phospholipids and propionic acid (SPPA), polyoxyethylene alkyl phenol ether (PAPE), vegetal oil (VO) and mineral oil (MO), *least significant difference.

are given in Figure 1. Significant difference on spray retention values ($F = 47.8$; $p < 0.001$) on leaves of *Eucalyptus* species is detected. Leaves of *E. torelliana* had the highest retention with the mean values differing significantly to the other species. Lowest retention value was attributed to leaf of *E. grandis* (Figure 1a). Spray retention difference verified between the species could be attributed to leaf surface structure as for example surface micro-roughness, trichomes and microcrystalline waxes composition which can vary from *Eucalyptus* species. Lin et al. (2016) verify larger wetted area on *E. tereticornis* leaves comparing to leaves of eucalypt hybrid urograndis

(*E. urophylla* × *E. grandis*) at any surfactant concentrations.

The effect of adjuvants on spray retention was significant ($F = 265.6$; $p < 0.001$). The highest spray retention on leaves was achieved by adjuvant MO, followed by VO. NPE, SPPA and PAPE showed no significant difference of spray retention values. HT was the adjuvant with least ability to increase spray retention (Figure 1b). The greatest spray retention ($15.2 \mu\text{g cm}^{-2}$) provided by MO was approximately 2.1 times bigger than that provided by HT ($7.3 \mu\text{g cm}^{-2}$).

A significant effect of adjuvant concentration in spray

retention on *Eucalyptus* leaves ($F= 189.2$; $p<0.001$) was observed (Figure 1c). Spraying retention reduction was verified as adjuvant concentrations increased becoming steady at concentration of 0.5%. Concentrations over 0.1% reduced the spray retention on leaves. Concentrations of 0.005 and 0.01% increased spray retention differing significantly from treatments applied with only distilled water. The highest spray retention ($13.6 \mu\text{g cm}^{-2}$) appeared on the adjuvant concentrations of 0.005 and 0.01% increased by 2 times over the concentration of 1% ($6.8 \mu\text{g cm}^{-2}$).

Gaskin et al. (2000) report that aqueous solution of the insecticide acephate with addition of spreader-sticker adjuvants reduced spray retention on cucumber plants. The effects were attributed to observable spray droplets coalescence and run-off. As this research had the spraying done until run-off point (maximum volume of leaf saturation) the spray retention decreased as the adjuvants concentrations increased. In the previous papers published by Matuo and Baba (1981); Ocampo-Ruiz and Matuo (1994); Oliveira et al. (1997) and Silva et al. (2008) is verified reduction of spray retention liquid capacity on leaves when spraying was made with aqueous solution containing spreader-sticker adjuvants at high spray volume. Pesticide application using adjuvants which reduce surface tension must ensure more security when is done at high spray volumes in order to avoid run-off and consequently pesticide losses.

It is very important attempt that the values of spray retention obtained in this research were realized using only water plus adjuvants. Different spray retention results can be found when used solution with pesticide formulation due to the different characteristics. Lin et al. (2016) studying the effects of surfactant concentration on the spreading properties of pesticides, observe as nonionic surfactant concentration increased continuously from 0.1% to 0.25, 0.5 and 1%, the wetted area of solution (water + surfactant) droplets present a reduction rather than expansion trend on surface of *Eucalyptus* leaves. When the solutions contained pesticide the trend of wetted area is increase as surfactant concentrations also increase. The same authors conclude that spread properties using only water are distinct than those pesticide droplets containing surfactant and a specific spray solution has an optimal spreading efficacy at a specific surfactant concentration.

Conclusions

The adjuvant heptomethyltrisiloxane show the best performance on reducing surface tension of aqueous solution at the lowest concentrations and exhibited the highest efficiency. Spray retention on *Eucalyptus* leaves varies with the species. *E. grandis* leaves had the higher amount of spray retention and *C. citriodora* the lower.

Mineral oil was the adjuvant which provides the best spray retention on *Eucalyptus* leaves. The adjuvants concentrations of 0.005 and 0.01% increased foliar spray retention while 0.5, 1.0 and 2.0% decreased foliar spray retention. Pesticide spray application using high volumes in the presence of adjuvant which reduces drastically the surface tension should be carefully performed to avoid losses by run-off, increasing the cost of production and environmental pollution.

Conflict of Interests

The authors have not declared any conflict of interests.

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