

Article

The Effects of Adjuvants on the Wetting and Deposition of Insecticide Solutions on Hydrophobic Wheat Leaves

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Abstract: Pesticide droplet deposition determines the efficacy of pesticide solution and is a critical process in pesticide application. Adding spray adjuvants can improve droplet deposition to a certain extent, but there are currently many types of adjuvants with different properties. The improper selection or unreasonable use of adjuvants may be counterproductive, increasing the loss of pesticides or causing crop damage. In this study, the adjuvants methylated plant oil (Beidatong), alkoxy modified polytrisiloxane (Silwet408), hyperbranched fatty alcohol ether modified polymer (ND500), and polymers adjuvants (G2801) were selected through surface tension, contact angle, the determination of the maximum retention (R_m) and point of run-off (POR), and field deposition to explore the effect of adjuvants on the wetting and deposition performance of pesticides on wheat leaves. Compared with Beidatong and G2801, Silwet408 and ND500 could significantly reduce the surface tension of pesticides and greatly promote the wetting properties on wheat leaves, but R_m and POR value on wheat leaves were reduced. The field test results also showed that the deposition amount of the adjuvant Silwet408 and ND500 on wheat was slightly lower than that of the adjuvant Beidatong and G2801. Studying the effects of adjuvants on the wetting and deposition properties of insecticide solutions can provide practical guidance for the use of adjuvants.

Keywords: spray adjuvant; wetting; deposition; pesticide performance; utilization rate



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1. Introduction

Pesticide is an effective technical means to prevent and control diseases, insect pests, and weeds in the field, which plays an essential role in protecting human health and food security [1]. Pesticide spraying is a traditional and widely used mode of application [2]. However, the insufficient wetting and deposition performance of pesticides on superhydrophobic crop leaves reduces the utilization rate of pesticides and causes harm to the environment [3,4].

At present, adding spray adjuvants to pesticide solution in field spraying is a prevailing tool to improve pesticide solution physical-chemical properties [5,6]. A spray adjuvant is a pesticide assistant before spraying pesticide directly into the spray barrel, also called tank-mixing adjuvants [7]. Researchers have conducted abundant studies on the effects of spray adjuvants on the properties of pesticide solution and the dose transfer behavior of pesticide droplets (including droplet evaporation and drift [8,9] and wetting and depositing [10–13] on target leaves). Spray adjuvants can reduce the surface tension of pesticide solution, enhance the wetting and adhesion of the droplet on the target leaves, or penetrate the waxy layer of the leaf surface to promote the absorption of active components [14,15]. However, the use of spray adjuvants is not unlimited. For example, silicone adjuvants are widely used because of their superior properties, but the key problems caused by the application of the adjuvants are often heard. Improper use of organosilicon adjuvants can lead to “decomposition” of the cuticle of leaf epidermal cells, reduce its water holding capacity,

damage cells, and finally produce pesticide damage [16]. Due to the excellent penetration ability, the main pesticide damage symptoms are loss of green and dry plant leaves [17]. The occurrence of pesticide damage of organosilicon adjuvants is closely related to crop varieties, pesticide types, adjuvants concentration, the dosage of plant leaves, application temperature, and so on. Crops with a thin leaf wax layer and easy to be wetted (such as cucumber, beans, etc.) are more prone to pesticide damage than rice, ginger, green onion, and other crops with a thick leaf wax layer. Gaskin et al. [18] showed that spray adjuvants did not enhance the retention of easily wettable cucumber leaves. The wrong or excessive use of adjuvants causes crop damage and environmental pollution [19].

Overall, we found a relationship between pesticides' wetting and deposition properties during application. Yuan et al. [20,21] used point of run-off (*POR*) and maximum retention (R_m) to describe the law of retention and loss of pesticide solution on leaves. The amount of pesticide solution carried by the leaves of crops has a saturation point, beyond which it will be automatically lost, and this point is called *POR*. The maximum retention of plant leaves is achieved after the pesticide solution is lost. For hydrophobic crops, such as rice, cabbage, and wheat, the surface tension of pesticide solution is generally greater than the critical surface tension of leaves, and the maximum retention of pesticides on their leaves is relatively small [22]. For hydrophilic crops, such as cotton and cucumber, the surface tension of pesticides is generally less than the critical surface tension of the leaves. If the concentration of adjuvant is too high, the phenomenon of "run-off" will occur, resulting in excessive pesticide loss and reducing the deposition of pesticides on the target leaves [23,24].

Four kinds of spray adjuvants and commonly used 22% Thiamethoxam · Lambda-cyhalothrin insecticides were selected here. The trials of adjuvants to investigate the wetting and deposition performance of pesticide solution on wheat leaves were conducted in the laboratory and the field. The purpose of this study is: (1) to establish indoor and field evaluation methods for the deposition and wetting performance of pesticide adjuvants on target crop leaves, to provide theoretical guidance for the rational selection and correct use; (2) to explore the effects of different types of spray adjuvants on the physicochemical properties of pesticide solution; (3) to understand the correlation of the effects of spray adjuvants on wetting and deposition properties.

2. Materials and Methods

2.1. Materials

A 22% thiamethoxam-lambda-cyhalothrin (Thi · LC) suspension concentrate (SC) was purchased from Shanxi Yitianfeng Crop Technology Co., Ltd, Weinan, China. The spray adjuvants used in this study included Beidatong (methylated plant oil, Hebei Mingshun Agricultural Technology Co., Ltd., Shijiazhuang, China), ND500 (hyperbranched fatty alcohol ether modified polymer, Guilin Jiqi Biochemical Co., Ltd., Guilin, China), Silwet 408 (alkoxy modified polytrisiloxane, Momentive Performance Materials, Wilton, CT, USA), G2801 (high molecular polymers, Shantou Shentai Development of new material technology Co., Ltd., Shantou, China).

Wheat (*Triticum aestivum* L., Jimai 22) was cultivated in the Institute of Plant Protection, Chinese Academy of Agricultural Sciences greenhouse. The environmental conditions of the greenhouse culture were 16 h of light and 8 h of darkness. The temperature was 24 °C and 18 °C, respectively, and the relative humidity was 70%. When the wheat grew to the 5–7 leaf stage, the leaves were randomly selected and pasted on the slide with double-sided tape.

2.2. Determination of Surface Tension

The DCAT 21 surface tension meter (Data Physics Instruments GmbH, Stuttgart, Germany) was used to measure the surface tension of the adjuvant solution prepared with distilled water and pesticide solution with adjuvants by the Wilhelmy plate method. The concentrations of adjuvants tested were Beidatong (0.01%, 0.1%, 0.5%, 1.25% and 2%), ND500 (0.0005%, 0.005%, 0.05%, 0.5% and 0.75%), Silwet408 (0.0001%, 0.001%, 0.01%, 0.25%

and 0.5%), G2801 (0.01%, 0.1%, 0.5%, 1% and 1.5%). The critical micelle concentration (CMC) of each adjuvant was obtained by measuring the surface tension of the adjuvant solution with different concentrations. After that, the adjuvant with the critical micelle concentration was added to Thi · LC-SC to test the surface tension of the pesticide solution.

2.3. Wetting and Spreading Performance of Droplets on Wheat Leaves

The glass slide with wheat leaves was placed on the sample table of the contact angle measuring instrument. 2 μ L droplet was dropped on the surface of wheat leaves by the droplet automatic injection system of the OCA 20 contact angle measuring instrument. The instrument's video system recorded the droplet's changing state within 1 min. The wetting and spreading properties of droplets on the wheat leaf surface were evaluated by measuring the changes in dynamic contact angle, diameter, and height of droplets on the wheat leaf surface by analyzing the droplet profile. The environmental conditions were 25 ± 2 °C and $45 \pm 5\%$ humidity. Each measurement was repeated five times, and the average value was taken at the corresponding time point.

In addition, the deposition morphology of droplets evaporated on wheat leaves was viewed using the depth of field and super-resolution imaging mode of the VHX-2000 three-dimensional microscope (KEYENCE Corporation, Osaka, Japan). Meanwhile, the wetting and diffusion of pesticide droplets on wheat leaves were also observed from a top view. About 2 μ L droplets were injected into wheat leaves with a 5 μ L micro syringe. To ensure the accuracy of the experiment, living wheat leaves were used here without cutting leaves from plants.

2.4. Determination of the Point of Run-Off and Maximum Retention of Leaves

Based on the previously reported method [20], we have made improvements and designed the schematic diagram of the device shown in Figure 1 to conduct an indoor evaluation of the deposition and retention of pesticide solution on leaves. Healthy wheat leaves at 5–7 leaf stages were selected, and the leaf area (S) was measured by the YX-1242 leaf area measuring instrument (Beijing Yaxin Technology Co., Ltd., Beijing, China). The wheat leaves were glued on the self-made 0°, 30°, 45°, and 60° stage with double-sided tape. The pesticide solution was put into the spray device. The stage was connected to the bottom tray through a strut. The droplets sprayed out of the sprayer were separated by the isolation hood to prevent droplets from depositing in the platforms, pillars, and electronic balance, ensuring that the electronic balance reading accurately reflects the weight of droplets deposited on the leaves of wheat. A small sprayer was used for spraying. The distance between the nozzle and the blade was about 40 cm to reduce the pressure of blowing air to the platform. During the spray process, when pesticide solution began to flow from the leaf, the maximum reading of the balance W_1 (g) was recorded, and the spray was stopped. The liquid no longer flowed from the leaf, and the balance was read W_2 (g). Then the point of run-off (POR , mg/cm²) and the maximum retention (R_m , mg/cm²) of the leaf according to formulas (1) and (2) were calculated.

$$POR = W_1/S \quad (1)$$

$$R_m = W_2/S \quad (2)$$

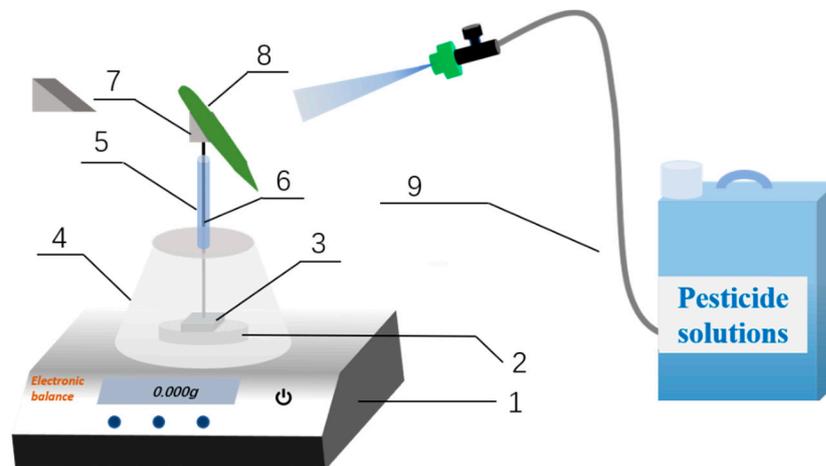


Figure 1. Schematic diagram of the deposition of droplets on leaves for laboratory evaluation. (1) electronic balance, (2) tray of electronic balance, (3) pedestal, (4) droplet isolation cover, (5) transparent plastic pipe, (6) strut (connecting stage and base), (7) 30°, 45°, 60° stage, (8) wheat leaf and (9) spray device.

2.5. Field Experiment

The field experiment on the effects of adjuvants on the canopy distribution of wheat plants was carried out in Shaomen Town, Jining County, Jinan City, Shandong Province, China (117.22 E, 36.98 N). The experiment time was April 26, 2020. The weather was cloudy, the wind speed was 2 m/s, and the temperature was 21 °C. The flying height of a plant protection UAV was 2 m, the nozzle was sector nozzle 015, and the nozzle flow was about 0.6 L/min. Five treatments were set up in the experiment: Thi · LC, Thi · LC + 1.25% Beidatong, Thi · LC + 0.5% ND500, Thi · LC + 0.25% Silwet408, Thi · LC + 1% G2801 and blank control group. The concentration of Thi · LC in the five treatments was 150 g a.i./ha. Temptation red with a mass fraction of 2% was added to each treated pesticide solution to detect the deposition rate of pesticides on wheat leaves.

The determination of pesticide deposition rate was carried out in the following ways. After the spray was dried to the liquid on the wheat plant, 6 points were selected for each treatment (blank control with no spray treatment for 12 spots). About 20 wheat plants with the same growth were selected at each point, cut from the top, middle, and bottom, placed in A4 size bags with written labels, and taken back to the laboratory for cold storage. The standard curve of allure red was plotted according to the method in reference [25], and the amount of allure red on different parts of wheat leaves was calculated according to the standard curve.

2.6. Data Analysis

Data processing and analysis were performed using Origin 2021 and SPSS software (version 25.0). All data were expressed as mean ± standard deviation. The statistical significance of different treatment results was compared by the one-way ANOVA method ($p \leq 0.05$).

3. Results and Discussion

3.1. Determination of CMC Concentration of Adjuvants

Surface tension is an important parameter in characterizing the physicochemical properties of pesticide spray solution [26]. The surface tension of liquid can be understood as the force of liquid surface shrinkage. The smaller the surface tension is, the smaller the shrinkage force is, so it is easier to spread on the surface and the easier the solution is to wet the solid interface [27]. Critical micelle concentration (CMC) is the lowest concentration of surfactant molecules combined in solution to form micelles [28]. When the concentration of surfactant in the solution exceeds this concentration, it is generally believed that the surface tension of the solution will not change. CMC can be used as a reference to determine

the optimal concentration of adjuvants. The critical micelle concentrations of the four adjuvants were determined according to the relationship between the log₁₀ value of adjuvant concentration and surface tension [11]. As shown in Figure 2a, the surface tension value decreases with the increase in concentration. When the concentration of adjuvants reaches a certain value, the surface tension value changes slowly, and an inflection point appears. The concentration at this inflection point is the critical micelle concentration of adjuvants. The CMC of ND500 and Silwet408 are 0.5% and 0.25%, while that of Beidatong and G2801 are 1.25% and 1%. Four adjuvants with CMC concentration were added to the Thi · LC insecticide solution to measure the surface tension (Figure 2b). It was found that the surface tension of adjuvants with ND500 and Silwet408 was lower than that of Thi · LC insecticide solution with Beidatong, G2801, and no adjuvants. The surface tension value of ND500 and Silwet408 added to Thi · LC can be reduced to 20 mN/m. The ability of oil adjuvants Beidatong and polymer adjuvants G2801 to reduce the surface tension of solution is not as good as that of adjuvants ND500 and Silwet408 containing organic silicon.

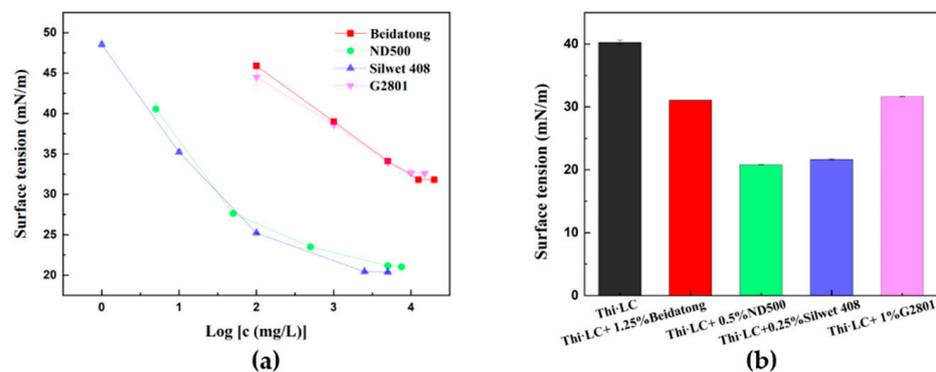


Figure 2. (a) Critical micelle concentration of four adjuvants. (b) The surface tension of Thi · LC without and with four adjuvants.

3.2. Wetting and Spreading of Pesticide Droplets on Wheat Leaves

The wetting and spreading of insecticide droplets at the target interface are closely related to the surface structure of leaves and the physical and chemical properties of insecticides [14]. As shown in Figure 3, under the scanning electron microscope, the surface of wheat leaves presents an orderly stripe structure with different fluctuation degrees. Moreover, the leaf surface is covered with a wax layer, fluff, and stomatal structure, and the size is up to the micro-nano scale.

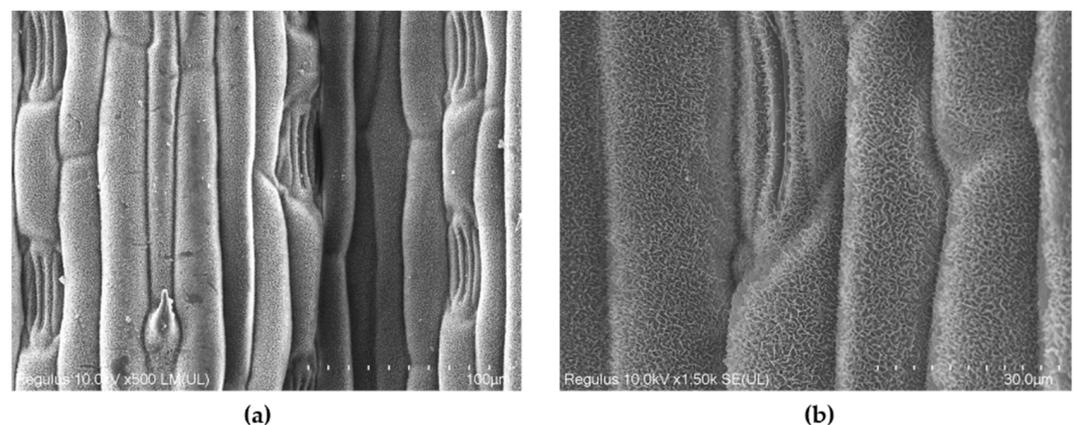


Figure 3. Scanning electron micrograph of the wheat leaf surface. Magnification: Scanning electron micrograph of the wheat leaf surface. Magnification: (a) 500 and (b) 1.5 k.

Due to the existence of micro and nanostructure on the surface of wheat leaves, there is a lot of air at the contact between droplets and wheat leaves, which is difficult to spread on the leaf surface, so it is easy to fall off [29]. The contact angle is usually used to evaluate the wettability of pesticide solution on the target surface. The measured contact angle of water droplets on wheat leaves is about 140° , indicating that wheat leaves are superhydrophobic interfaces. Figure 4 shows the morphological changes of Thi · LC and Thi · LC droplets added with four adjuvants on wheat leaves (including contact angle, droplet height, and droplet diameter). Thi · LC without adjuvants had the worst wettability on wheat leaves. The contact angle of the droplet did not change, and the height and contact diameter of the droplet almost did not change in 60 s (Figure 4). The changes of Thi · LC droplets with ND500 or Silwet408 adjuvants are also very sharp. The solutions with these adjuvants reach the super wetting state in a short time. The changing trend of the solution added with Beidatong and G2801 adjuvants are similar within 1 min. The contact angle and height of the droplet decrease slowly, and the diameter of the droplet increases, indicating that droplet is gradually wetting the leaf surface. For rough surfaces with microstructure/nanostructure surface morphology, the Cassie-Baxter wetting model and the Wenzel model are applicable.

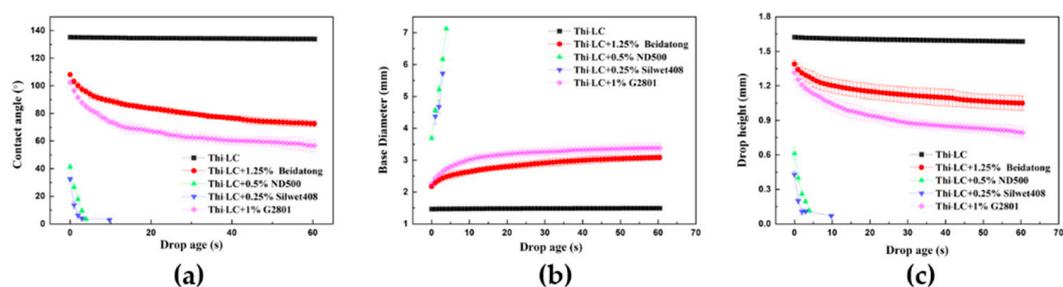


Figure 4. Changes in droplet contact angle (a), droplet height (b), and droplet contact diameter (c) on wheat leaves within the 60 s.

Figure 5 is an image of the droplet spreading state changing with time. Thi · LC droplets were in the Cassie-Baxter state on wheat leaves (Figure 5a). The wetting state of Thi · LC droplets with four adjuvants changed in varying degrees (Figure 5b–e). Thi · LC droplets added with ND500 or Silwet408 adjuvants quickly wetted and diffused on the leaves after contacting the wheat leaves, maintaining the Wenzel state. This corresponds to the result in Figure 4. This is because ND500 is a hyperbranched fatty alcohol ether-modified polymer containing organic silicon, and Silwet408 is a non-ionic surfactant of alkoxy-modified polytrisiloxane. As we all know, the commonly used silicone adjuvants can substantially increase the wettability, expansion, and adhesion of pesticides and reduce the surface tension of pesticide solution to a very low level, up to about 20 mN/m [30]. Both adjuvants contain organic silicon so that they can spread quickly on the wheat leaf surfaces. Beidatong is a plant oil adjuvant, and G2801 is a polymer adjuvant. Thi · LC droplets amended with Beidatong or G2801 wetted relatively slowly on the wheat leaf surface, but the final droplets also reached Wenzel state in about 1 min. Corresponding to the above surface tension results, ND500 and Silwet408 have the most significant ability to reduce the surface tension of tested insecticides. The wettability of Thi · LC added with these two adjuvants on wheat leaves is excellent, followed by Beidatong and G2801. Generally speaking, adding different types of adjuvants has different effects on the properties of pesticides. It is better to choose suitable spray adjuvants in combination with the control objects.

The deposition form of pesticide droplets after evaporation finally at the target interface affects the action effect of active components [31,32]. The interfacial deposition behavior of Thi · LC and Thi · LC droplets added with four adjuvants on wheat leaf surfaces was determined using a super depth field microscope. As can be seen from Figure 6, the spreading area with ND500 or Silwet408 is the largest, followed by Beidatong and G2801. Thi · LC droplets added with four adjuvants spread quickly and evaporate for a short time. The addition of adjuvants significantly improved the wetting distribution of droplets on

wheat leaf surfaces, and the final deposition form was uniform without the coffee ring effect. This increases the contact between pesticides and pests. The Thi · LC droplets without adjuvants had the smallest spreading area and the slowest spreading speed. The spreading of pesticide solutions is more conducive to the effect of effective components of pesticides. To sum up, adding adjuvants can reduce the surface tension of pesticide solution, reduce the contact angle of pesticide droplets on the target interface, make the deposition form of pesticide droplets on the interface more uniform and help to exert the prevention and control effect.

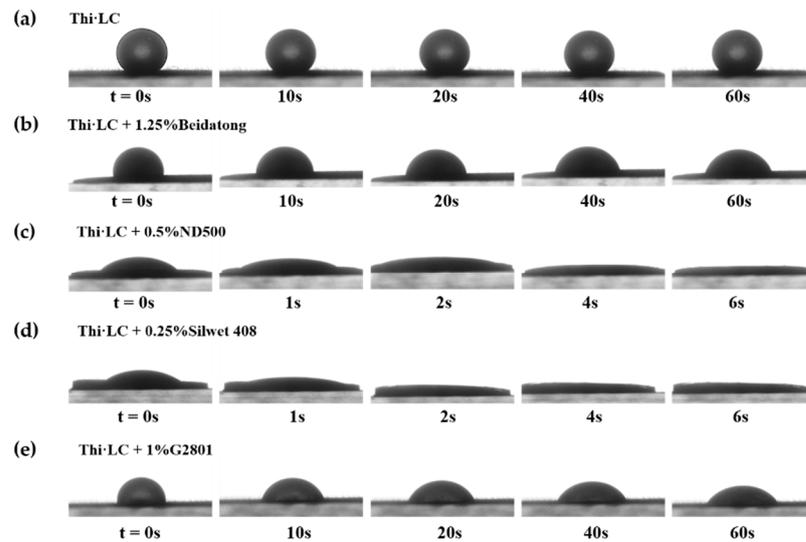


Figure 5. Images of wetting changes of Thi · LC (a) and Thi · LC droplets added with four adjuvants (b–e) on wheat leaves. The corresponding spreading time is listed under each image.

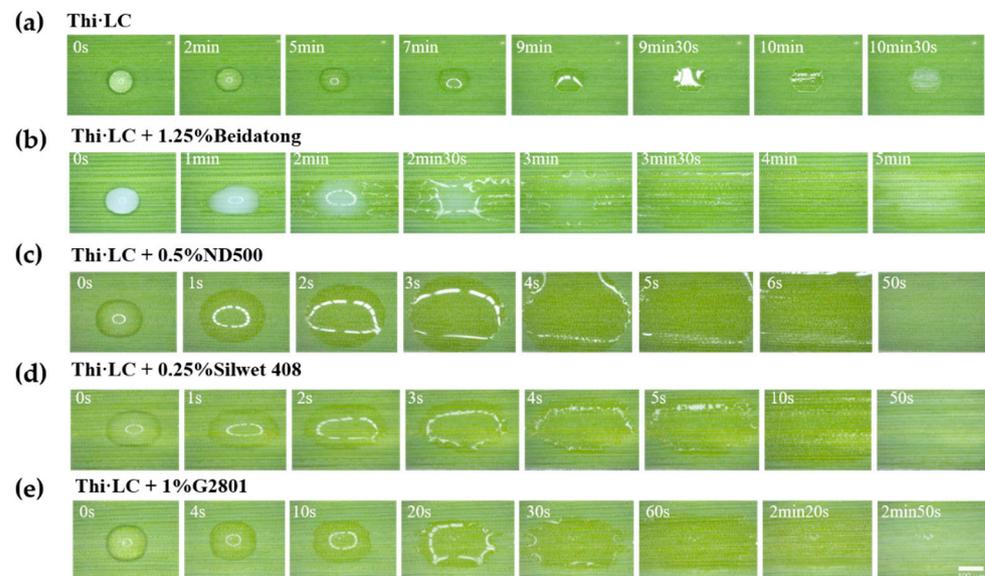


Figure 6. Deposition morphology changes of Thi · LC droplets (a) and Thi · LC droplets added with four adjuvants (b–e).

3.3. Effects of Spray Adjuvants on Pesticide Droplets Deposition

The pesticide droplets materialized by spray equipment can only be deposited accurately at the target interface for accurate effect [33]. However, the surface of hydrophobic plant leaves is difficult to be wetted, and the inclination of leaves brings severe challenges to the deposition of pesticide solution at the target interface [34,35]. Adding spray adjuvants

to pesticides can promote wetting on the target plant surface and increase the deposition of pesticide droplets on the target plant. When the wetting property of the pesticide solution is poor, increasing the spraying fluid will not increase the retention of the pesticide while spraying in the field. Moreover, after adding adjuvants, pesticide solution can reach the super wetting state. If the spray amount is not suitable, it will also cause pesticide loss. Therefore, studying the point of run-off (POR) and maximum retention (R_m) of crop leaves is necessary. In this experiment, we used the self-made device in Figure 1 to determine the POR , and R_m values of Thi · LC droplets added with four spray adjuvants at different tilting angles of wheat leaves. From Figure 7, on the wheat leaves with an inclination of 30° , the R_m and POR values of Thi · LC added with G2801 increased most significantly. The increase of Thi · LC added with Beidatong was not apparent. In contrast, the R_m and POR values of Thi · LC added with ND500 and Silwet408 decreased slightly. On wheat leaves with 45° and 60° inclination angles, the R_m and POR values of Thi · LC added with G2801 and Beidatong increased, while the R_m and POR values of Thi · LC added with ND500 and Silwet408 did not change. Yuan et al. [20] early suggested that the liquid's surface tension and contact angle are small, and the droplets on the wheat leaves are easy to diffuse, accumulate and lose. ND500 and Silwet408 have a strong effect on reducing the surface tension of pesticides because of containing organosilicon. Consequently, combined with the contact angle data of the above on the wheat leaves, Thi · LC droplets added with these two adjuvants are easy to diffuse on the wheat leaves, which may cause the droplets to gather and lose. The leaf inclination (the angle between a leaf and the horizontal plane) also greatly influences pesticide loss. The leaf inclination is negatively correlated with the R_m and POR values. The smaller the inclination of the leaf surface, the larger the R_m value of pesticide solution on wheat leaves, and the droplets can deposit on the leaf surface. The larger the inclination of the leaf, the easier the pesticide droplets are to roll and lose from the leaf surface. When spraying with a large volume, the pesticide droplets are easily lost due to deposition and aggregation. The maximum retention and point of run-off are related to the surface tension and contact angle of pesticides on leaves. When a pesticide is sprayed in the field, the surface tension and contact angle can be reduced by adding adjuvants. Rational use of adjuvants can reduce the bouncing loss of pesticides to a certain extent and increase the deposition effect.

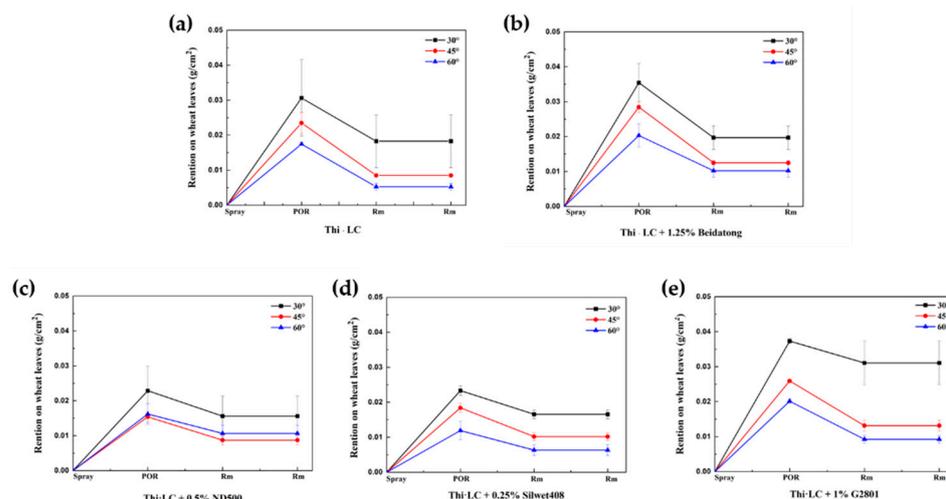


Figure 7. POR and R_m values of Thi · LC droplets (a) and Thi · LC droplets were added with four adjuvants (b–e).

3.4. Field Experiment

Figure 8 shows the deposition amount of Thi · LC with four adjuvants on wheat's upper, middle, and lower leaves. Compared with Thi · LC without adjuvants, the addition of adjuvants increased the deposition and distribution of pesticide solution on wheat plants, among

which G2801 and Beidatong had a pronounced effect, followed by ND500 and Silwet408. Four adjuvants could significantly increase the deposition of pesticides in different parts of wheat compared with Thi · LC. Figure 7 shows that the point of run-off and maximum retention of Thi · LC added with ND500 and Silwet408 adjuvants on wheat leaves are rarely reduced. Corresponding to the results of this part of the experiment, it is further proved that the selection of different types of adjuvants and the added concentration are essential to the efficacy. The correct use of adjuvants can prevent the loss of pesticide solution and increase efficiency. Besides, under the influence of the plant canopy, the deposition of pesticides in the upper leaves of the wheat plant was the largest. In Figure 8, the deposition of pesticide solution with adjuvants in the lower part of the wheat plant increased slightly compared with pesticide without adjuvants, which also showed that adding adjuvants might increase the penetration ability of pesticide solution in the plant canopy.

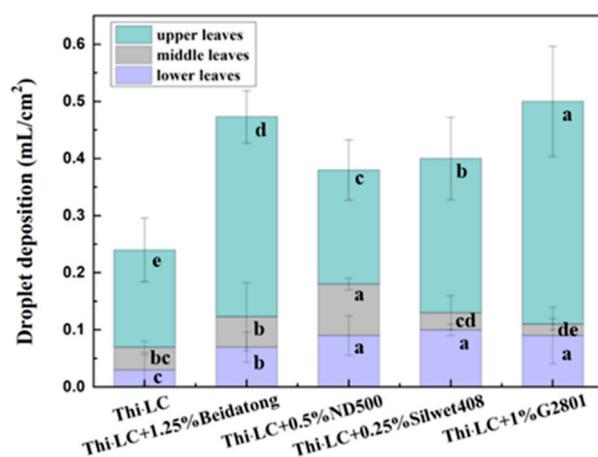


Figure 8. Pesticide droplet deposition and distribution in upper, middle, and lower wheat leaves. Lowercase letters in the histogram indicate significant differences in the data between other treatments at $p < 0.05$ using one-way ANOVA.

4. Conclusions

In summary, our results suggest that even though adjuvants promote the physical properties of the insecticide solution to increase wetting and spreading, excessive wetting of the foliar surface by droplets may result in loss of the solution and reduce the settling effect. We evaluated the maximum retention (R_m) and point of run-off (POR) of tested insecticide solutions on wheat leaves through an indoor self-made experimental device. The results testified that the spray adjuvant ND500 and Silwet408 reduced the R_m and POR values at the set concentration. Still, the wetting and spreading speed on wheat leaves and the ability to reduce the surface tension of Thi · LC are significantly better than the selected plant oil adjuvants Beidatong and high molecular polymer adjuvants G2801. The field test results also showed that the deposition amount of the adjuvants Silwet408 and ND500 was slightly lower than that of the adjuvant Beidatong and G2801. Therefore, when controlling pests, diseases, or weeds in the field, the application of adjuvants needs to combine the properties of adjuvants and pesticide formulations, the characteristics of control targets, and reasonable use concentration to better improve the pesticide control effect.

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