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The Effectiveness of Adjuvants on Different Formulations of Lambda-Cyhalothrin against Cotton Leaf Worm

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ABSTRACT

The present study undertakes to evaluate the effect of commonly used agricultural adjuvants (Argal and Techno oil) upon addition to different types of locally formulated lambda-cyhalothrin (OD, EC, EW) products. The physico-chemical properties for adjuvants and formulations were both determined separately. The impact of adjuvants and each formulations type, determined the physico-chemical properties with the field application rate in tank mix [full field application rate (M1), ¾ rate (M2), ¾ rate with 0.3 % Argal (M3) and ¾ rate with 0.3 % Techno oil (M4)] were evaluated using these treatments against 4th instar larvae of the cotton leafworm, *Spodoptera littoralis* (Boisd.) under laboratory and semi-field conditions with the same application rate. The results indicated that, the surface tension and pH values of the spray solution decreased whereas the viscosity and conductivity increased in M3 and M4 in the three types of lambda-cyhalothrin formulations. Results showed that M3 gave highest increase in average residual effect than the pesticide alone (M1). The highest effectiveness was that of EW M3 with improvement insecticidal efficiency of lambda-cyhalothrin against 4th instar larvae of cotton leafworm. These results indicated that adjuvants increased the effectiveness of lambda-cyhalothrin formulations. Therefore, adjuvants may be used to reduce the number of applications per season and the application rates of insecticides.

Key words: Adjuvant, Physico-chemical properties, (O.D) Oil dispersion, EC (Emulsifiable concentrate), EW (Emulsion in water), Insecticidal efficiency, Lambda cyhalothrin, cotton leaf worm

Introduction

The pesticide industry has made good progress in terms of development and production of low risk, environmentally friendly pesticide formulation. There is a growing demand for use of environmentally friendly water based formulations (oil-in-water emulsions and aqueous suspension concentrates) instead of conventional pesticide formulations. These formulations are intended not only to replace toxic, non-degradable ingredients from formulations, but also to increase the efficacy of products through a proper choice and balance of all components in the formulation (Slavica *et al.,* 2012).

The primary objectives of formulation technology are to optimize the biological activity of the pesticide and to give a product which is safe for use, formulation additives and adjuvants aim to minimize the residues of pesticides on food crops after spraying. All of these aspects increase the pressure on the development of improved formulation and adjuvant technologies (Alan, Knowles, 2008) and (Hazra *et al.*, 2013).

Adjuvants are quietly helping to revolutionize the pesticide business as the best tools for users to improve application and achieve more cost-effective, better-targeted, and more environmentally acceptable pest control. Adjuvants are essential for the performance of most pesticides and are helping shift use from preventative, high dose applications to low dose, specifically targeted curative applications. Several thousand adjuvants of widely varying quality are currently sold throughout the world (Green, 2000).

The cotton leaf worm, *Spodoptera littoralis* (Boisd.) (Lepidoptera: Noctuidae) is a destructive prolific and highly polyphagous insect in Egypt that causes various ravages not only for cotton plants but also for other field crops and vegetables. It is considered to be a major pest of great economic importance in many countries since it attacks a multitude of host plants. The use of insecticides such as Pyrethroid insecticides plays an important role in controlling the Egyptian cotton leaf worm in Egypt and will likely continue to be

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used until a more biological system with minimum environmental risks based management could be developed (Lobna *et al .,* 2013 and Heidi *et al*., 2015).

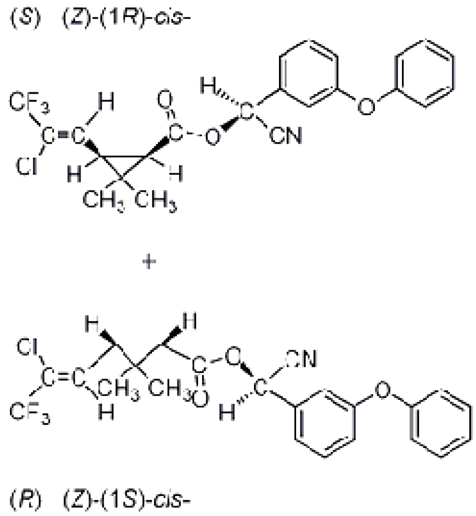
The main objective of this research was to specify the effect of additives in increasing the effectiveness of lambda-cyhalothrin against cotton leaf worm*, Spodoptera littoralis* (Boisd.) under laboratory and semi-field conditions.

Materials and Methods

Pesticides used:

Lambda Cyhalothrin:

Chemical structure:



IUPAC name:
(*R*)-α-cyano-3-phenoxybenzyl(1*S*)-*cis*-3- [(*Z*)-2-chloro-3,3,3-trifluoropropenyl]- 2,2 dimethylcyclopropanecar boxy late and (*S*)-α-cyano-3- phenoxybenzyl (1*R*)-*cis*-3-[(*Z*)-2-chloro-3,3,3- trifluoropropenyl]-2,2- dimethylcyclopropanecarboxylate.

Lambda-Cyhalothrin is a non-systemic pyrethroid insecticide. Its axonic poisons affect or act on the nerve fiber by binding to a protein that regulates the voltage-gated sodium channel. The channels are pathways through which ions are permitted to enter the axon and cause excitation (Heidi *et al.,* 2015).

Adjuvants:

Additives can be used to enhance the biological performance of pesticides, as well to improve the physical characteristics of its pre-diluted formulations (Craig and Holloway, 2003).

ARGAL (Silwet):

It is a non-ionic surfactant based on a trisiloxane ethoxylate that improves the spreading and the speed of penetration of pesticide formulations into the cuticle layer of surface leaf which can result in leaf coverage that is 10-30 times greater than that of conventional surfactants (Alan, Knowles, 2010).

Techno Oil:

It is non-ionic surfactant bio activator, and a water conditioner extracted from botanical source used for agricultural and chemical purposes for foliar application. Techno oil eliminates physical barriers of the insects and fungi such as chitin; waxes (paraffin). Techno oil is a product containing glutamic acid (L-glutamic amino acid).

Insect:

Cotton leaf worm:

The cotton leaf worm, *Spodoptera littoralis* (Boised) (Lepidoptera: Noctuidae) is swarming polyphagous, foliage feeding insect that is distributed throughout the world. This insect is one of the major cotton pests which cause considerable damage to many crops (Hanan and Hosam, 2014).

Experimental work:

Investigation the physico-chemical properties for locally formulated lambda cyhalothrin:-

The physico-chemical properties of the formulation and spray solution were determined prior to and following the accelerated storage test.

Tests carried out for the formulation:

Viscosity:

Viscosity of the prepared formulation was measured with a ″Brookfield DV II+ PRO″ digital Viscometer (Brookfield, USA), UL rotational adaptor (ULA). The temperature was kept at 25°C during the measurement using a water bath (Model: TC-502, USA) and each reading was taken after equilibrating the sample. All prepared formulations were obtained by directly reading the viscosity (CP) from the viscometer (ASTM D2196-15, 2015).

Surface tension:

The surface tension of the formulation was measured using Force tensiomate sigma 700 USA by du Nouy method, a platinum-iridium ring. The instrument was recalibrated before testing by standardized weight 500 mg, the recorded dial reading for this weight was 41.4 dyne/cm. The measured sample should be clean, homogenous and free from any bubbles with a stable surface and the dial reading surface tension (dyne/cm) is recorded from the tensiometer (ASTM D1331-14, 2014).

Flash point:

Flash point is an important property of the formulation, it used to determine the lowest temperature at which a volatile substance can become vaporized into a flammable gas, the vapor may cease to burn when the ignition source is removed. To measure a flash point, with 2 ml of sample which is not open to the outside atmosphere. It is necessary to introduce an ignition source to the substance and wait for a flash, the point at which it is able to be ignited by Kolchler closed cup Flash Tester and glass syringe (ASTM D3828-12a, 2012).

Determination of physico-chemical properties of spray solutions:

Emulsion stability and re-emulsification:

This test is suitable for emulsifiable concentrate (EC) and emulsion oil in water (EW) pesticide formulations according to CIPAC MT 36.3 (2000). Emulsion characteristics are investigated by adding 5 ml of the formulation sample to WHO standard hard water in a 100 ml measuring cylinder to produce 100 ml of aqueous emulsion. Hard water was prepared by dissolving 0.304 g of anhydrous calcium chloride and 0.139 g of magnesium chloride hexahydrate in distilled water and made up to one liter. This provided total hardness equivalent to 342 ppm of calcium carbonate according to CIPAC MT 18 (1995). The cylinder was stoppered and inverted 10 times. Subsequently, the amount of free oil or cream that separated at the top or the bottom of the emulsion was observed after the emulsion was allowed to stand undisturbed for various time intervals (initial time, 0.5h and 24h for re-emulsification).

Dispersion stability:

Checks the effect of dispersions stability of oil dispersion formulations. Two graduated cylinders or emulsion tubes (250 ml) were prepared at room temperature (23 ± 2 °C) to the 240 ml mark with standard water, 10 ml of sample were pipetted (drop wise) and the test tubes were inverted 30 times. The dispersion characteristics (amount of cream, free oil and sediment) were observed after the preparation of the dispersion at zero, 0.5 h and 24 h for complete re-dispersion according to CIPAC MT 180 (1997).

Persistent foam:

Persistent foam is a measure of the amount of foam likely to be present in a spray tank after dilution of the product with water. Specified amount of the material was added to hard water (95ml) in the measuring cylinder and made up to the mark. The cylinder was stoppered and inverted 30 times. The cylinder was left to stand undisturbed on the bench for 5 min. The volume of foam was recorded according to CIPAC MT 47.1 (1995).

pH Measurement:

The pH value of a mixture of a sample with water or of an undiluted aqueous formulation is determined by means of a pH meter and an electrode system, it was measured by using a pH Meter (Model: Jeway 3510) was initially standardized using J.T.Baker buffered solution of pH 4 and 7 (Park, Scientific Limited, Northampton, UK). One gm was weighed from sample into 100 ml distilled water in a beaker and shaken vigorously to mix completely, the electrode was immersed into the sample and left for 5min without stirring during the measurement at a room temperature to allow the pH value to stabilize. The instrument must be calibrated before the measurement. The electrode was thoroughly washed between samples using a stream of distilled water to remove all traces of the previous sample. CIPAC MT 75.3 (1999).

Electrical Conductivity:

The conductivity of spray solutions was measured by Conductivity and Salinity meter "Thermo Orion model 115A$^+$, USA". The measurements were made at 25°C ± 2. Before the measurement, the conductometer was calibrated with 0.01 M KCl solution CIPAC MT 32 (1995).

One gram was weighed from sample into 100 ml distilled water in a beaker and shaken vigorously to mix completely, it was immersed into sample and left for 1-2 min during the measurement at a room temperature to allow the conductivity value to stabilize.

Storage conditions on the specifications of pesticide:

The present study investigated the effect of storage conditions on the stability of the tested pesticides. The study was carried out under the following conditions:

Accelerated hot storage:

Accelerated storage procedure was executed by placing the sample (about 50 ml) in a bottle and placing the capped bottle and its contents in an oven at 54 ± 2°C for 14 days CIPAC MT 46.3 (1999).

Cold storage:

For the stability test at low temperature (0°C), 100 ml of each sample was transferred to a glass tube. For cooling, the tube and its contents were placed in a refrigerator and remained at 0 ± 1°C for 7 days. At the end of day 7, the tubes were removed from the refrigerator, and allowed to remain undisturbed at room temperature for 3 hours. The volume of any separated material at the bottom of the tubes was subsequently recorded CIPAC MT 39.3 (1999).

Determination the physico- chemical properties for selected adjuvants:

Solubility test:-

Solubility test of the prepared surfactant in different solvents as (water, acetone and xylene) was measured by recording the proper volume of the solvent required to complete solubility. Described by (EL - Sisi, 1981) and (Nelson, and Fiero, 1954).

One gram of the sample (surfactant) was exactly weighed in a beaker and the solvent was dropped from a burette, constant stirring at 25°C with glass rod till completely solubilized. The percent of solubility was calculated according to the following equation:-

Solubility % = (w/v) x 100

Where w is weight of surfactant, v is the volume of solvent required for complete solubility.

Surface tension:

The surface tension of surfactant was measured using Force tensiomate sigma 700. USA by du Nouy method, a platinum-iridium ring. (ASTM D1331-14, 2014) as mentioned before.

The critical micelle concentration:

Laurier *et al.,* (2003), Osipow (1964) and Tadros (1995) evaluated the critical micelle concentration (CMC) dyne/cm for surfactant. The stock solution of surfactant was prepared where 2.5 grams of surfactant were added into 250 ml of distilled water and then different concentrations from (0.1to 1%) were prepared and measured by DUNOUY interfacial Tensiometer. The value of the CMC is the point maximum surface activity after which the surfactant tends to coagulate in micelle meaning that at concentrations greater than CMC value, the surface tension of the solution does not decrease further with an increase in surfactant concentration.

Hydrophilic –Lipophilic balance:

The solubility of surfactants in water was considered as an approximate guide to their HLB value and their usefulness measured according to a standard method by Lynch *et al.,* (1974) and Green Waled (1958). 10 grams of surfactant were introduced into 100ml of distilled water and solubility. In the HLB range 10-13 give hazy solutions Alan, Knowles (1998).

The HLB value of such surfactants, according to Griffin (1954), is equivalent to the mass (or weight) percentage of oxy-ethylene content (*E*) divided by 5.

HLB = E/5.

Free acidity or alkalinity:

It was determined according to CIPAC MT 191 (2005), 10 g of surfactant in were dissolved in 100 ml distilled water in a beaker and stirred to homogenize then titrated electrometrically. The normality for NaOH and H_2SO_4 from 0.01 to 0.2 mol/l. by HANNA901 automatic titrator with glass electrode.

Acidity calculated as % $H_2SO4 = (4.904.c_1.t) / w$

Alkalinity calculated as % $NaOH = (4.001.c_2.s) / w$

$C_{1,2}$= normality of sodium hydroxide and sulfuric acid respectively.

t ,s=volume of the end point titrated with sodium hydroxide solution or sulfuric acid respectively .

W=10 g of sample.

Determination of the physico-chemical properties of the spray solution of the locally formulated Lambda Cyhalothrin 5% at the field dilution rate:

The experiments were carried out using Nile water at the field dilution rate where field dilution percentage of pesticides was calculated by using the following equation:

Field dilution % = A/B × 100
Where: A= rate of pesticide/feddan
B= volume of pesticide spray solution/feddan

The recommended field dilution rate for lambda cyhalothrin on cotton is 375Cm^3 /feddan (200 liter Nile water) meaning that the field dilution rate was 0.1875 ml of the formulation/100 ml water.

The experiment was composed of 12 treatments using three types of lambda cyhalothrin formulations (O.D, EC and EW); each formulation was used to make 4 treatments (full dose- 3/4 dose - 3/4 dose +0.3%Argal- 3/4 dose +0.3%Techno oil).

Persistent foam, Emulsion stability, Dispersion stability, pH, Electrical Conductivity, Surface tension and Viscosity were determined by the same methods as mentioned before for spray solutions of the different formulation types at the field dilution rate.

Evaluation of effectiveness of locally formulated insecticide against 4th instars larvae of the cotton leafworm, Spodoptera littoralis (Boisd.):

Field experiments were conducted according to Ministry of Agriculture protocol semi-field, 1993, in cotton plants cultivated in Central Agricultural pesticides laboratory (CAPL) DOKKI, Giza, Egypt. A single nozzle hand sprayer was used for spraying (spray volume was 200 liter/feddan). The effectiveness of the three formulations was evaluated with complete rate, 3/4 recommended rate and their combinations with two adjuvants at 3/4 recommended rate against 4th instars larvae of the cotton leaf worm.

These applications rates were sprayed in separated treatment (each treatment has 3 rows with 4 meter long). Cotton leaves were collected from each treatment immediately (zero time) after spraying and after 1, 3, 6, 9 and 12 days post-treatment. The leaves were transferred to the laboratory for measuring the insecticidal toxicity. The larvae were exposed to the treated leaves, and then mortality percentages were recorded after 24 hr post-treatment.

Susceptible strain of *Spodoptera littoralis* was mass reared in laboratory at 25 ± 2°c and 70% ± 5 R.H.) El-Defrawi *et al.,* (1964).

Statistical analysis:

Mortality data were corrected according to Abbott's formula (1925):

$$\text{Corrected \%} = 1 - \left(\frac{\text{n in T after treatment}}{\text{n in Co after treatment}} \right) * 100$$

Where: n = Insect population, T = treated, Co = control

The improvement insecticidal efficiency for lambda-cyhalothrin treatments were analyzed by using Duncan multiple range test SAS, Duncan (2006), the level of significance was expressed as $p < 0.05$:

As mentioned by Heidi *et al.,* (2015) larvae were fed on castor oil-bean leaves (*Ricinus communis* L.) and kept in 1 liter glass jars covered with muslin which was fixed tightly by a rubber band. The number of larvae per jar differed according to the developing instar. After pupation, they were supplied with a piece of cotton moistened with 10% sugar solution and paper strips to act as sites for egg deposition. The deposited egg masses were daily collected and left till hatching. The newly hatched larvae were transferred to clean jar and supplied with fresh leaves.

Results and Discussion:

The Physico-chemical properties for locally formulated insecticides of lambda cyhalothrin 5 % (OD, EC and EW) formulations:

Proper formulation and efficient delivery systems are the key elements in the performance of different products. One of the most important ways to improve the effectiveness of pesticides and minimize their impact on non-target organisms is through increased penetration of active ingredients into plant foliage Gasic *et al.,* (2011). The physico-chemical parameters for three types of lambda-cyhalothrin formulations according to international specifications of WHO (1979) and JMPS (2002 and 2010) include: emulsion stability, re-emulsification, dispersion stability, persistent foam, pH, conductivity, viscosity, surface tension, flash point, cold storage and accelerated storage.

Results obtained in Table (1) showed that all formulations passed successfully through emulsion stability re-emulsification and no separation of cream or oil was observed or sedimentation after 0.5 hour and 24 hours re-emulsification complete pre and after storage.

Table 1: The physico-chemical properties of lambda cyhalothrin 5% formulations.

Physical properties			Pesticide formulations		
			OD	EC	EW
Pre Storage	Spray solution	Dispersion stability (OD) Emulsion Stability (EC,EW) (ml)	Nil	Nil	Nil
		Persistent foam (ml)	1.5 ml	2 ml	Nil
		pH	6.40	6.45	3.79
		Conductivity µs	649	628	785
	Formulation	Viscosity Cp	68.86	2.17	5.83
		Surface Tension Dyne/cm	32.7	28.9	32.9
		Flash point °C	Over 50	47	Not detected
Cold Storage			Passed	Passed	Passed
Accelerated Storage	Spray solution	Dispersion stability (OD) Emulsion Stability (EC,EW) (ml)	Nil	Nil	Nil
		Persistent foam (ml)	1.5 ml	2 ml	1ml
		pH	6.46	5.26	3.72
		Conductivity µs	680	624	833
	Formulation	Viscosity Cp	69.65	2.26	6.45
		Surface Tension Dyne/cm	34	29	34
		Flash point °C	Over 50	47	Not detected

O.D= Oil dispersion EC =Emulsifiable concentrate EW=Emulsion in water

Acceptable limits for emulsification, re-emulsification, emulsion stability is a maximum of 2 ml cream, or trace of oil after 30 minutes according to CIPAC MT 36.3 (2000). This is in agreement with El-Sisi *et al.,* (2009) that stated that if the physico-chemical properties including emulsion stability test were determined for a product (in their case Chlorpyrifos 48% EC) and the product passed them, then it is considered as a successful formulation according to the international recommendations. Lambda-cyhalothrin OD formulation passed the dispersion stability test both before and after storage, with no separation of cream or oil observed after 0.5 hour and 24 hours re-dispersion was also complete. Acceptable limits for dispersion stability is a maximum of 2 ml cream, trace of oil after 30 min according to APVMA (2015) and consistent with JMPS (2010). Also, in the persistent foam test, the data shown that the formulated lambda-cyhalothrin (OD, EC and EW) pre-storage gave foam of (1.5, 2 and 0 ml) in volume respectively. Post accelerated storage foam volumes were (1.5, 2 and 1 ml) respectively. This matched with Slavica *et al.,* (2012) that mentioned that the variation in pH value ranged from (4.6-6.0), the persistence of foam ranged from 0-6 ml after 5 min before stability test and after two weeks. However these differences are considered to be acceptable by JMPS (2002) and CIPAC MT 47.1 (1995).

The pH value pre-storage (6.40, 6.45 and 3.79) followed by accelerated storage (6.46, 5.26 and 3.72) respectively. These results agreed with El-Sisi *et al.,* (2009) who mentioned that the deposits on treated plant leaves increased by decreasing surface tension and decreasing pH value. A change in pH on storage can give an indication of instability of the active substance or product JMPS (2010).

As shown in Table (1) conductivity pre-storage was (649, 628 and 785 µs) respectively, but after storage there has been a marked increase in data (680, 624 and 833 µs) respectively. The Obtained results from Hussein *et al.,* (2010) indicated that reducing the surface tension and pH value with increasing electrical conductivity led to an enhancement in wetting, spreading and retention of plant oils used as spray solutions on the treated plants, therefore their toxicity was increased.

The viscosities pre-storage for the pesticide formulations (OD, EC and EW) were (68.86, 2.17 and 5.38 Cp), and a slight change occurred in the viscosities post accelerated storage (69.65, 2.26 and 6.45 Cp).

Surface tensions of three types of formulations were (32.7, 28.9 and 32.9 Dyne/cm) and after accelerated storage were (34, 29 and 34 Dyne/cm)

According WHO (1979) and JMPS (2002) where flash point must be more than 22.8 °C , the flash point before and after storage for three types of formulations were (over 50, 47 °C and not detected) .

All formulations passed successfully in the cold test since they didn't show any separation or sedimentation. This complies with APVMA (2015) and JMPS (2002 and 2010).

In some places in Australia, night temperatures regularly approach 0 ˚C or lower. Therefore, the liquid formulations should also be tested at 0 ±2 ˚C or lower for seven days. The effect of low temperatures on stability should be determined and reported according to (CIPAC) method MT 39.3.

Accelerated stability tests at elevated temperatures are designed to increase the rate of chemical degradation or physical change of a product. Testing is performed at elevated temperatures in an attempt to obtain information on the shelf life of a product in a relatively short time, JMPS 2010 recommend testing of the relevant product parameters before and after storage at 54 ˚C for 14 days.

In this respect, Slavica *et al.,* (2012) showed that the storage at 0°C and 54°C has been used to control physical and chemical stability. It has been generally accepted that two weeks at 54°C represent 2 years in normal conditions.

The physico-chemical properties for surface active agents:

The most important requirement in the formulation of a pesticide is the solubility of surfactants in the solvent. Data in table (2) showed that, Argal was insoluble in water, even though it emulsified in water but the solubility in xylene and acetone were different (50 and 83.3 %) respectively. Techno oil made an emulsion in water and was insoluble in acetone. Meanwhile, the results indicated that the surfactants had an HLB of 10 and 11 which gave a hazy emulsion. Alan, Knowles (1998) stated that the HLB range 10-13 gave hazy solutions.

Argal has a weak acidic nature while Techno oil has a highly alkaline nature, therefore it is suitable for use in formulations without expecting any phyto-toxicity effect.

The surface tensions for surfactants were (19.77 and 23.1 Dyne/cm) respectively, and the concentration of surfactants at which no more decrease in surface tension could be obtained is called CMC which could be obtained by increasing surfactant concentration. The reduction of surface tension has been measured for determining the contact angel between the droplet and leaf surface enhance wetting, spreading and deposit of pesticide on the treated plant then improving control target species. Data in the same table showed that the CMC values for Argal and Techno oil were 0.3 and 0.4 % (wt. /v.), respectively.

Table 2: The physico-chemical properties of the tested surfactant.

Surfactant	Solubility % (wt. /v.)			CMC %	Surface tension Dyne/cm	HLB	Acidity as % H_2SO_4	Alkalinity as % NaOH
	Water	Xylene	Acetone					
Argal	Emulsion	50	83.3	0.3	19.77	11	2.94×10^{-3}	
Techno oil	Emulsion	Partially miscible	Non soluble	0.4	23.1	10	-	2.48

The Physico-chemical properties of the spray solution of the locally formulated Lambda Cyhalothrin 5% at the field dilution rate:

The trend nowadays is towards multifunctional spray adjuvants that are designed to make handling and application easier with improved performance, reduced surface tension, increased viscosity, thus improved deposition and reduced droplet size. The ''super spreading'' silicone counterparts aid quick absorption and thus improve rain fastness by reducing surface tension. Surface tension is an important surfactant property to achieve wetting and prevent spray droplets from bouncing off the leaves thus reducing application costs. In some cases, they are environmentally friendly since they can reduce the amount of active ingredient required in a particular application or treatment Green and Beestman (2007) .

Alan, Knowles (2010) discussed super spreading and how trisiloxane surfactants improved the spreading of pesticide formulations. A popular super spreader used as a tank mix additive is Silwet which resulted in leaf coverage that is 10-30 times greater than with conventional surfactants.

Data in table (3) demonstrated the physical properties of three types of 5% lambda-cyhalothrin formulations (OD, EC and EW), each type of formulation consisted of 4 treatments (M1: full rate alone, M2:3/4 rate alone, M3: 3/4 rate +0.3 % Argal and M4: 3/4 rate +0.3 %Techno oil)

Decreasing the pH values of the spray solution would lead to an increased in attraction between spray solution and treated plant thus increased depositing and penetration on the tested surface which will in turn increase the effectiveness (Molin and Hirase, 2004).

Emulsion and dispersion stability test showed no oily separation or creamy layer or sedimentation and the persistence of foam test results conformed to APVMA (2015).

Data in table (3) expressed that the viscosity increased in M3 and M4 than M1 and M2. Richardson (1974) stated that, increasing viscosity of spray solution caused a reduction in drift and an increase in the retention and sticking of spray solution on the surface of plant.

The adjuvant Argal and Techno oil decreased surface tension. Surface tension values for OD, EC and EW for the (full rate , ¾ rate , ¾ rate +0.3% Argal and ¾ rate +0.3% techno oil) were (35.34 , 35.67, 21.38 , 25.5, 31.36 , 33.97 , 22.1 , 28.2 , 28.09 , 41.08 , 21.6 and 28.4 dyne/cm, respectively). It is the parameter most relevant to surfactant-based adjuvants. It is can therefore reduce dynamic surface tension. Surface tension of a liquid that resulted in a zero contact angle indicated complete wetting and spreading on the treated surface then increased pesticide effectiveness (David and Geoff, 2000) and (Vladimir *et al.,* 2007).

Lim *et al.,* (2011) showed that the organo-silicone surfactants reduce Surface tension with dilution concentration is lower than critical micelle concentration (CMC) level, therefore, achieving greater reduction in surface tension. The presence of nano-emulsion system in the nano-formulation effectively reduced the surface tension about 2-3 folds. Reduced surface tension increased spreading and thus improved the pesticide performance.

On the other hand, M3 in three types of formulations gave a noticeable decrease in conductivity. The elevation of the electrical conductivity of spray solution may lead to deionization of pesticide formulation and consequently increasing its deposit and penetration through the surface of tested plant. Consequently, the pesticide effectiveness of these formulations was increased (El-Attal *et al*., 1984).

Table 3: Determination of the physico-chemical properties of the spray solution of the locally formulated Lambda Cyhalothrin 5% at the field dilution rate.

Type of formulation	Lambda Cyhalothrin 5% OD				Lambda Cyhalothrin 5% EC				Lambda Cyhalothrin 5% EW			
Formulation code / Physical properties	M 1	M2	M3	M4	M 1	M2	M3	M4	M 1	M2	M3	M4
pH	7.22	7.72	7.01	7.13	7.48	7.85	7.20	7.33	7.05	7.42	6.86	7.10
Conductivity µs	376	372	456	350	392	365	423	341	369	358	438	348
Viscosity cp	1.95	1.95	2.08	2.39	1.95	1.94	2.23	2.52	1.95	1.96	2.31	2.56
Surface Tension dyne/cm	35.34	35.67	21.38	25.5	31.36	33.97	22.1	28.2	28.09	41.08	21.6	28.4
Dispersion stability (OD) Emulsion Stability (EC,EW) (ml)	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Persistent foam (ml)	0.4	1.1	Nil	1.4	0.2	0.5	Nil	1.0	0.2	0.4	Nil	1.4

M1: full rate alone, M2:3/4 rate alone, M3: 3/4 rate +0.3 % Argal , M4: 3/4 rate +0.3 %Techno oil

Evaluation of the effectiveness of locally formulated insecticides against 4th instars larvae of the cotton leafworm ,Spodoptera littoralis (Boisd.):

The experiment was conducted to determine the initial mortality rate immediately after spraying and the mortality at indicated days (1, 3, 6, 9 and 12 days) after application, then determining the average residual toxic effect for all treatments. Data presented in Table (4) clearly showed that; most additives improved the insecticidal activity of the 3/4 recommended rate against 4th instar larvae of the cotton leafworm, *Spodoptera littoralis* (Boisd.). M1 in O.D with complete recommended rate of application gave 100% initial larval mortality, while the addition of adjuvants to lambda-cyhalothrin using 3/4 of the recommended rate of the application gave different initial mortality (80, 90, 86.66, 86.66, 86.66 and 93.33 % respectively).

According to the mean residual effect and mean general effect, it was noticed that all the adjuvants increased the percentage of larval mortality than that obtained with the full rate application and 3/4 fold application rate. M3 (3/4 rate +0.3% Argal) in EW gave the highest average residual effect when it was combined with all followed by M4 (3/4 rate +0.3%Techno oil) to the same formulation. EC pesticide formulation takes the second place in average percentage residual effect and general effect mortality.

The different significantly from the Duncan multiple range test you can conclude the following:

- The mean of EWM3 is higher than the means for all treatments

The highest effectiveness was that of M3 (3/4 rate +0.3% Argal) in EW with improvement insecticidal efficiency of lambda-cyhalothrin against 4th instar larvae of cotton leafworm.

- The mean of EWM4 and is higher than EWM1, EWM2 and ECM2.

M4 (3/4 rate +0.3%Techno oil) in EW is highly significant and effectiveness than recommended dose ,3/4 rate of application in EW and 3/4 rate of application in EC .

- The mean of ECM3 and ECM1 is higher than EWM1and EWM2.,

The highest effectiveness was that of M3 (3/4 rate +0.3% Argal) and M1 (recommended dose) in EC than M1 (recommended dose), M2 (3/4 rate of application) in EW.

- But the effectiveness of M3 (3/4 rate +0.3% Argal) EC highly than M1 (recommended dose) in EC
- Differences between all other means are not significant.

Ismail *et al.,* (2004) discussed that the water-based insecticide formulations (EWs) offer many advantages to the end-users/operators over the solvent-based insecticide formulations (EC). Cost of production for EW-insecticides formulation can be less expensive compared to the EC insecticides formulation because they replace about 70 to 80% of the oil (solvent) with water. Solvents and surfactants derived from palm based materials have better environmentally friendly characteristics than the petroleum based surfactants and mineral oils. The bioefficacy tests confirmed that the EW-pyrethroid insecticide formulations have comparable effectiveness to the conventional EC-pyrethroid insecticide formulations.

Table 4: Effect of the locally formulated insecticide on the mortality of 4th instar larvae, *Spodoptera littoralis*.

Type of formulations	Treatment	% Initial mortality	% mortality at indicated days after applications					Mean Residual effect (MRE) %	General Effect mortality (GEM) %
			1	3	6	9	12		
OD	M1	100	76.66667	60	10	10	0	31.33333	42.77778
	M2	83.33333	73.33333	63.33333	20	16.66667	0	34.66667	42.77778
	M3	80	73.33333	60	26.66	23.33333	0	36.66533	43.88778
	M4	90	80	60	13.33333	20	0	34.66667	43.88889
EC	M1	93.33333	83.33333	60	30	10	0	36.66667	46.11111
	M2	83.33333	80	60	10	20	0	34	42.22222
	M3	86.66667	86.66667	63.33333	40	0	0	38	46.11111
	M4	86.66667	80	60	10	20	0	34	42.77778
EW	M1	83.33333	63.33333	56.66667	23.33333	20	0	32.66667	41.11111
	M2	83.33333	73.33333	60	10	16.66667	0	32	40.55556
	M3	86.66667	86.66667	63.33333	40	6.666667	0	39.33333	47.22222
	M4	93.33333	80	60	33.33	13.33333	0	37.33267	46.66611

M1 : full rate alone M2 :3/4 rate alone M3: 3/4 rate +0.3 % Argal M4: 3/4 rate +0.3 %Techno oil

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