



Synthesis and Physico-chemical Properties of some New Surface Active Agents and their Improvement Effects on Emamectin benzoate against *Spodoptera littoralis*

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Abstract

Esters are among compounds produced in industries all over the world and regarded as significant products due to their application in the production of herbicides, pesticides, paints, solvents, and plastics. The most commonly used method of synthesizing esters is the Fisher esterification reaction. Fatty acids (caprylic, lauric, and palmitic) were reacted with polyethylene glycol (PEG 400 and PEG 600) to synthesize non-ionic surfactants, then they were elucidated by IR spectrum and mass spectrometry, and then their physico-chemical properties were estimated such as (HLB, CMC, free acidity or alkalinity and solubility). Physico-chemical characteristics of pesticide spray solution were estimated separately before and after mixing with esters. After that, the 4th instar larvae of the cotton leaf-worm were treated with serial concentrations of the synthesized esters alone, recommended pesticide and also treated by LC₅₀ of the pesticide with esters. Results showed that, the synthesized non-ionic surfactants were enhanced the physico-chemical characteristics of spray solutions. As a result of mixing pesticide with surfactants, pesticides' viscosity and conductivity increased while their surface tension and pH value decreased. The findings of the bioassay test also showed that all surfactants decreased the LC₅₀ value of the insecticide Emamectine benzoate 1.9% EC, however adding PEG 600 MC to the pesticide had the best effects, lowering its LC₅₀ value to 0.021 ppm. Thus, after the necessary investigation has been done on these esters, it is recommended to combine them with commercially recommended insecticides in order to decrease the amount of pesticide utilized and subsequently contamination of the environment.

Keyword: Emamectin benzoate, Esterification, Fatty acid esters, Nonionic surfactants, PEG

Introduction

Non-ionic surfactants with amphiphilic structures are used in the daily chemical industry, pesticides, metal processing, textiles, food, and other industries as biodegradable green non-ionic surface-active agents. One of the non-ionic surfactants with various applications and perfect wettability, emulsification, and solubilization is polyethylene glycol fatty acid esters (Gamayurova *et al.*, 2015; Wu *et al.*, 2021).

Pesticides formulations have many trade names. Simply put, a formulation is a particular product's shape that is typically created based on the needs of the consumer, physiological and chemical characteristics of the active ingredients (Purkait and Hazra, 2020). The majority of technical pesticides are made ahead of time by mixing active ingredients with inert, diluents, preservatives, adjuvants, etc. to create an effective product, simple to handle, apply, has a good shelf life, doesn't have any unfavorable side effects, and used in a practical way to obtain a

cost-effective, convenient, and safe approach to pest management after being stored, transported, and treated using practical methods, to convert it into a product that can be used efficiently, safely, and economically suitable (Hazra and Purkait, 2019).

Persistence and efficacy of insecticides improve using adjuvants. This could reduce the effective pesticide concentration by up to ten times. As a result, adjuvants can be utilized to reduce the number of treatments made over the season as well as the rates at which insecticides are applied. Adjuvants can commonly increase the quality of pesticide treatments by increasing their efficiency and biological activity against insect pests (Dewer *et al.*, 2017; Abdelgaleil *et al.*, 2018; El-Bassouiny *et al.*, 2023). Also, these can reduce surface tension or improve the surface activity of pesticide dilution during field application, and hence the residual efficacy increase. This impact may improve the treated plant surface (Dewer *et al.*, 2017).

Esterification reaction is one of the greatest processes in the synthesis of organic compounds, by

heating a mixture of carboxylic acids and an abundance of alcohols. The reaction proceeds to equilibrium after a defined period, which is governed by process kinetics and thermodynamics, to alter the balance correctly. The product yield is ultimately reduced when the reaction does not complete (Khan *et al.*, 2021). Esters are organic materials present everywhere, whether naturally or chemically, biofuels like biodiesel (Ambat *et al.*, 2018), coatings, plastics (Tang and Chen, 2019), medicines (de Oliveira *et al.*, 2020), and some are used as insecticides and herbicides, which are the two principal applications for esterification products (Mohsin *et al.*, 2017; Wang *et al.*, 2020).

The cotton leaf-worm (CLW), *Spodoptera littoralis* (Boisd.) (Lepidoptera: Noctuidae) severely damages commercially important crop species (Moustafa *et al.*, 2023). Additionally, it is believed to be a polyphagous insect that affects more than 100 hosts across Africa, Mediterranean Europe, and the Middle East, including cotton, eggplant, tomatoes, alfalfa, and many ornamental plants. Due to the activity of the larvae in devouring foliage, resulting in the loss of leaves, buds, flowers, bolls and eventually a 50% drop in production, it is the most detrimental pest to numerous agricultural commodities (Benelli *et al.*, 2017; Magholifard *et al.*, 2020; Hamouda *et al.*, 2022; Hussein *et al.*, 2023; Lana *et al.*, 2023). The economic cost associated with crop damage caused by CLW makes pest control a critical problem. Farmers aggressively utilized synthetic chemical insecticides to combat the risk of this pest, which resulted in the appearance of resistance pest strains and contamination of the periphery, which affects all living organisms (Ayoub *et al.*, 2021; Moawad, 2021).

The main objective of this study was to produce nonionic surfactants, evaluation of their physico-chemical properties and their impact on improving the efficiency of emamectin benzoate by decreasing the dosage rate of pesticides against cotton leaf-worm under laboratory conditions.

Materials and Methods

Chemicals used:

- 1- Fatty acids:** Caprylic acid (molar mass 144.2 g mol⁻¹), Lauric acid (molar mass 200.32 g.mol⁻¹), were purchased from (LOBA Chemie., India, 99% purity) and Palmitic acid (molar mass 256.4 g.mol⁻¹), was produced by (VEB-LABORCHEMIE APOLDA, Germany, purity 98%).
- 2- Polyethylene-glycol (P.E.G):** Polyethylene glycol 400 (PEG 400), molar mass 390:410 g mol⁻¹ and Polyethylene glycol 600 (PEG 600), molar mass 590:610 g mol⁻¹ were obtained from LANXESS, Co. Germany.
- 3- Solvents:** Acetone) C₃H₆O(, Xylene (C₈H₁₀) and Dimethylformamide (DMF C₃H₇NO) were

provided by El-Nasr Co. for Pharmaceutical Chemicals. Egypt.

- 4- Insecticide:** Beno zid 1.9% EC (Emamectin benzoate 1.9% EC) was purchased from The National Agri., Chemi., Com., Egypt.

Synthesis of mono-fatty acid esters:

Nonionic surfactants mono-fatty acid esters were prepared by heating fatty acids caprylic, lauric and palmitic acids (0.1 mole) for 30 minutes before adding dropwise polyethylene glycol 400 and 600 (0.1 mole). Then, the temperature was raised to 350 °C while stirring continuously for 5-6 hrs. The product (surfactant) was allowed to cool to obtain mono-fatty acid esters (3a-f) according to Osipow, (1964) and Wasfy *et al.*, (2011).

Structural elucidation of the newly prepared esters:

Spectral analysis was utilized to identify the chemical structure of the expected substances using two techniques: IR spectrum were obtained (KBr) pells on a Pye-Unicam SP-1000 Spectrophotometer. This analysis was performed in the micro-analytical center at Cairo University, Faculty of Science, Giza, Egypt. Mass spectrum were obtained using a GCMS-QP/1000 EX mass spectrometer set to 70 eV. This analysis was obtained from the Food Development and Safety Centre (FDSC), Qaha, Al Qalyubia Governorate, Egypt.

The physico-chemical characteristics of the newly synthesized esters as surfactants:

- 1- Solubility:** One gram of a substance was measured for total solubility or miscibility at 20 °C using volume of distilled water, DMF, acetone and xylene (Nelson and Fiero, 1954). The % solubility was calculated using the equation below.

$$\% \text{ Solubility} = W/V * 100$$

[Where; W = surfactants weight and V = volume of solvent required for complete solubility]

- 2- Free acidity or alkalinity:** It was evaluated according to CIPAC MT 191, (2005).
- 3- Hydrophilic-Lipophilic Balance (H.L.B):** The solubility of a surfactant in water has been used to indicate its hydrophilic-lipophilic balance (Lynch and Griffin, 1974).
- 4- Surface Tension:** It was determined using a Stalagmometer unit (Monika and Hina, 2017).
- 5- Critical Micelle Concentration (C.M.C):** The critical micelle concentration is the emulsifier concentration at which additional decreases in surface tension cannot be achieved by increasing the emulsifier concentration. The method depends on measuring surface tension, which

was estimated as the method stated by Osipow (1964).

Physico-chemical parameters of spray solution at field dilution rate:

The physico-chemical properties of the pesticide solution alone or in combination with a surfactant were evaluated as follows:

- 1- Emulsion stability: It was performed according to CIPAC MT 36.3, (2003).
- 2- PH value: It was calculated using a pH meter. "Hanna Instruments pH 211 Microprocessor and Hanna pH electrode (Dobrat and Martijn, 1995).
- 3- Electrical Conductivity: The electrical conductivity was measured using a consort C830 PH/Conductivity meter, where μhos is the unit of measurement for electrical conductivity according to Dobrat and Martijn, (1995).
- 4- Surface tension: It was determined as previously stated.
- 5- Viscosity: The Ostwald viscometer was used to determine the viscosity using the method outlined by Monika and Hina, (2017).

Bioassay tests:

1- Rearing of the cotton leaf-worm:

Syngenta Group Co., Qaha, Al Qalyubia Governorate (Egypt), supplied a laboratory strain of the cotton leaf worm *Spodoptera littoralis*. This strain was used to make a stock culture using the method described by El-Defrawi *et al.*, (1964), and the culture was kept. The culture was raised in a lab. Condition at a temperature of 25 ± 2 °C and a relative humidity of $65 \pm 5\%$, away from any chemical exposure and contamination.

2- Larval bioassays:

2.1- Toxicity of the synthesized surfactants on the cotton leaf-worm:

Serial concentrations from the synthesized compounds were tested (5000, 10000, 20000, and 40000 mg/L) to assess the latent influence. Ten larvae employed into three replicates of each concentration, and another three replicates were used as control (untreated with freshwater). Larvae of the 4th instar were examined. Caster-bean leaves were dipped in each concentrate for 30 seconds, then let to dry and the larvae were allowed to consume the treated leaves for 1 day while the control larvae were allowed to eat the untreated leaves (El-Defrawi *et al.*, 1964; Fahmy *et al.*, 1988; Fadaa *et al.*, 2020). Mortality was recorded after 1, 2, 3, 4, and 5 days after treatment to measure the latent effect.

2.2- Sub-lethal concentrations of emamectin benzoate against *S. littoralis*:

A leaf dipping technique was used as mentioned previously. Pieces of homogeneous castor bean leaves were soaked for 10 seconds in each of the six pesticide concentrations evaluated (made in water) before drying at room temperature with distilled water as the control, after being fasted for two hours, ten 4th instar larvae were given treated castor bean leaf pieces. The plastic containers were stored at a temperature of 25 ± 2 °C. Each concentration was replicated three times. After the first 24 hrs. each replicate was filled with fresh, untreated castor bean leaf pieces. Mortality was observed at 24, 48, and 72 hours after treatment.

2.3- Effect of emamectin benzoate after mixing with synthesized surfactants:

The insecticide's lethal concentration (LC_{50}) was mixed with a 0.5% surfactant solution. Then, serial concentration from emamectin benzoate concentrations were made (0.1, 0.08, 0.06, 0.04, 0.02 mg/L). The 4th instar larvae of the cotton leaf-worm were then treated with the mixture using the same way as previously used to test the pesticide's toxicity. After 24, 48, and 72 hours, mortality was recorded. Thereafter, the slope and LC_{50} and LC_{90} values are then computed using probit analysis.

Statistical analysis:

The mortality percentages were corrected using Abbott's formula (1925) as follows:

Mortality % = $(\% \text{ Observed mortality} - \text{Control mortality}) / 100 - \% \text{ Control mortality}$.

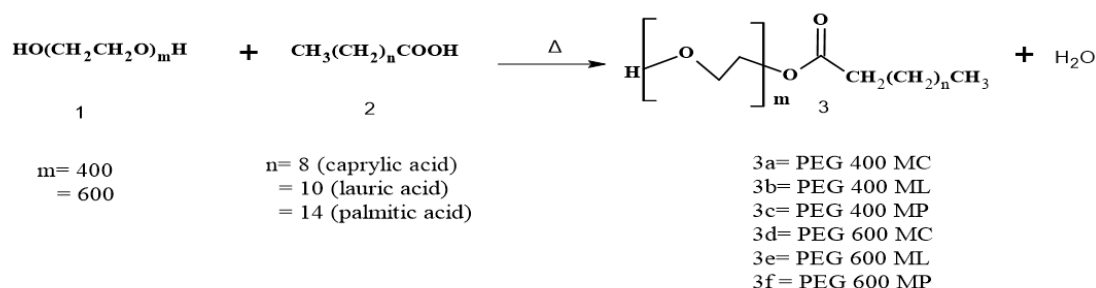
Also, the slope, LC_{50} , and LC_{90} values were computed using the Finney approach (1952).

Results and Discussion

3.1 Chemistry part:

Esters are organic compounds that are widespread and can be obtained naturally or chemically. Due to their low toxicity, lack of immunogenicity and antigenicity, and solubility in water and organic solvents. Fatty acid esters of polyethylene glycols (PEG) are particularly attractive. They are used in industrial, medicinal, and cosmetic applications as detergents, lubricants, wetting agents, solubilizers, and emulsifiers.

The reaction pathways of six mono-fatty acid esters (**3a-f**) were depicted in (Scheme 1) as follow :



Scheme 1: Reaction mechanism

Heating a mixture of carboxylic acids and an excess of the matching alcohols is a step in the Fisher esterification process. The reaction finally reaches equilibrium, which is governed by process kinetics and thermodynamics, after a predefined period of time.

Polyethylene glycol 400 (PEG 400) and Polyethylene glycol 600 (PEG 600) **1** were reacted with fatty acids (caprylic, lauric and palmitic) **2** to synthesize polyethylene glycol 400 mono esters (**3a-c**) and polyethylene glycol 600 mono esters (**3d-f**) (Scheme 1). The structure of compound **3a** was confirmed by IR spectrum and showed the following absorption bands for (ν_{OH}) at 3400 cm^{-1} , ($\nu_{\text{CH-aliphatic}}$) at 2929 cm^{-1} and ($\nu_{\text{C=O}}$) at 1713 cm^{-1} . Moreover, the mass spectrum accurately showed the molecular ion peak at $m/z = 527$.

3.2 Determination of the physico-chemical properties of the prepared esters:

Data in Table (1) showed the physico-chemical properties of the newly prepared surface active agents (esters), PEG 400 mono esters (3a-c) and PEG

600 mono esters (3d-f). All of them provided emulsions with water and a good degree of solubility in acetone and DMF, while all esters gave good solubility with xylene, except PEG 400 MC (3a) and PEG 600 MP (3f). All prepared surfactants showed a lower surface tension than that of water 72 dyne/cm. Additionally, PEG 400 ML (3b), PEG 400 MP (3c), PEG 600 ML (3e), and PEG 600 MP (3f) all have HLB values of 6-8 and cause milky dispersion in water, whereas PEG 600 MC (3d) has an HLB value of 10-13 and causes persistent milky dispersion and PEG 400 MC (3a) has the highest HLB value of 12-18 and causes complete solubility in water. Higher HLB values occur due to shorter alkyl chains (Sisi *et al.*, 2006). In addition PEG 400 MC (3a), PEG 400 ML (3b) and PEG 400 MP (3c) have the same CMC was 0.4% but PEG 600 MC (3d) and PEG 600 MP (3f) have the same CMC was 0.5% however, PEG 600 ML (3e) showed a higher CMC value. On the other hand, all esters demonstrated acidic characteristics from a free acidity or alkalinity perception.

Table 1. Physico-chemical characteristic of newly synthesized surfactants.

Surfactants	Solubility % (W/V)				Surface tension dyne/cm	CMC%	HLB	Free acidity as % H ₂ SO ₄
	water	DMF	Acetone	Xylene				
PEG 400 MC (3a)	G.E*	100	100	N.S	34.96	0.4	12-18	25.7
PEG 400 ML (3b)	G.E*	100	100	66	49.09	0.4	6-8	22.1
PEG 400 MP (3c)	G.E*	100	50	20	50.10	0.4	6-8	18.7
PEG 600 MC (3d)	G.E*	100	100	20	35.21	0.5	10-13	16.1
PEG 600 ML (3e)	G.E*	100	100	20	42.63	0.7	6-8	11.6
PEG 600 MP (3f)	G.E*	66	40	N.S	54.00	0.5	6-8	15.7

G.E*: Give Emulsion

N.S: Insoluble

Betana *et al.*, (2004) stated that PEG 600 DL give emulsion in water and El-Sisi *et al.*, (2006) reported that several non-ionic surfactants (PEG 400

ML, PEG 400 DL, PEG 600 DL, PEG 400 MP, PEG 400 DP, PEG 600 MP and PEG 600 DP) generated an emulsion in water while dissolved to varied

degrees in acetone and xylene, and PEG 400 ML was the most soluble ester in acetone and xylene and to avoid micelle formation, the CMC value was evaluated to ensure that the applied concentration was significantly less than CMC. Non-ionic surfactants (fatty acid esters synthesized from PEG) reduce surface tension and have an effect on the physico-chemical properties of water, indicating that these surfactants could be used as wetting and spreading agents and for insecticide dilution when added at 0.3: 0.5% (Betana *et al.*, 2004; El-Sisi *et al.*, 2006; El-Sisi *et al.*, 2011; Saad *et al.*, 2013; El-bassoiny *et al.*, 2023).

3.3 The physico-chemical properties of spray solution at field dilution rate:

Data in Table (2) displayed the physico-chemical parameters of emamectin benzoate 1.9%

EC spray solution at concentration of 100 and 75% of the field dilution rate in two types of water, tap and Nile water. The viscosity of the spray solution was 9.75 cm/poise, and also passed the emulsion stability test at the above dilution rates and in different types of water used. In addition, pH value was 7.22 in both types of water and at different dilution rates, while the electrical conductivity at 100% of field dilution rate was 403 μ mhos in tap water and 400 μ mhos in Nile water, while the values were 361 and 397 μ mhos in tap and Nile water, respectively when 75% of the field dilution rate was used. When using tap water, the surface tension was 64.8 dyne/cm for spray solutions at different dilution rate, while when using Nile water, the values were 61.59 and 64.8 dyne/cm at the recommended rate and 75% of the recommended rate, respectively.

Table 2. Physico-chemical properties of the spray solution alone at concentration of 100% and 75% of the field dilution rate.

Tests	Conc.	Emulsion stability		PH		Conductivity μ mhos		Surface tension dyne/cm		Viscosity cm/poise	
		Tap water	Nile water	Tap water	Nile water	Tap water	Nile water	Tap water	Nile water	Tap water	Nile water
Insecticide Emamectin benzoate 1.9 %EC 250 cm ³ / Fed	100%	pass	pass	7.22	7.22	403	400	64.8	61.59	9.75	9.75
	75%	pass	pass	7.22	7.22	361	397	64.8	64.8	9.75	9.75

Data in Table (3) showed that the spray solution after mixing with the prepared surfactants passed the emulsion stability test in (tap and Nile water) at 100 and 75% of the field dilution rate, as well as the effect of these esters on the physical and chemical properties of the spraying solution. The table clearly showed that the highest ester that affected the pH value was PEG 600 MC (3d), where it was 4.84 and 4.94 at the field dilution rate in tap and Nile water, respectively, and 4.83 and 4.89 at 75% of the field dilution rate in tap and Nile water, respectively. The highest electrical conductivity value recorded by adding PEG 400 MC (3a). It was added, and it was 633 and 349 μ mhos at the field dilution rate in tap and Nile water, respectively, while at 75% of the field dilution rate it was 641 and 345 μ mhos in tap and Nile water, respectively. Also, PEG 400 MC (3d) adding exhibited the best value of surface tension, which reached 34.03 dyne/cm at the field dilution rate in tap water. While, it reached 34.4 dyne/cm at 75% of the field dilution rate in tap water and the Nile water, as well as the field dilution rate in Nile water. However, after adding the synthesized esters, the spray solution's viscosity increased. The

spray solution's viscosity in Nile water at two different dilution rates reached 10.73 cm/poise when adding the PEG 400 MC (3a), PEG 400 ML (3b), PEG 600 MC (3d), and PEG 600 ML (3e), which were the best values.

The physico-chemical characteristics of the spray solution, which included high viscosity, high electrical conductivity, an acidic PH value, and low surface tension, these characteristics demonstrate the spray solutions expected to be effective. Reducing the pH of the spray solution may improve the attraction between the sprayed solution and the treated plants enhancing the insecticidal efficacy (Abdalla *et al.*, 1989; EL-Metwally *et al.*, 1989; Radwan *et al.*, 1994; Hussein 2002; Molin and Hirase, 2004; Green and Hale 2005; Hamouda *et al.*, 2022). Increased electrical conductivity in an insecticide spray solution results in the insecticide's de-ionization, which increases the insecticide's deposit and penetration on the treated plant surface and boosts insecticidal effectiveness (EL-Attal *et al.*, 1984; Tawfik and EL-Sisi 1987; Attia *et al.*, 2023). A decrease in the surface tension of a pesticide spray solution would lead to an

increase in spreading across the treated surface, increasing insecticidal effectiveness (Furmidge, 1962; Gaskin *et al.*, 2005; Ryckaert *et al.*, 2007; Pereira *et al.*, 2016). As indicated by Richardson (1974); Bode *et al.*, (1976); Bode *et al.*, (1976); McMullan, (2000); Spanoghe *et al.*, (2007); Saad

et al., (2013) pesticide spray solutions require to have an increase in viscosity. Increasing the spray solution's viscosity decreases drift, improves spray solution retention on the surface of plant leaves, and boosts pesticidal effectiveness.

Table 3. Physico-chemical characteristics of emamectin benzoate spray solution at 100% and 75% of field dilution rate after mixing with mono-esters (0.5%).

Tests	Conc.	Emulsion stability		PH		Conductivity μ mhos		Surface tension dyne/cm		Viscosity cm/poise			
		Tap water	Nile water	Tap water	Nile water	Tap water	Nile water	Tap water	Nile water	Tap water	Nile water		
Insecticide and mono-esters	3a	100%	pass	pass	5.17	4.94	633	349	34.03	34.4	10	10.73	
		75%	pass	pass	5.14	4.84	641	345	34.4	34.4	10	10.73	
	3b	100%	pass	pass	6.15	6.06	538	240	54	43.7	9.75	10.73	
		75%	pass	pass	6.25	6.02	465	242	54	43.7	9.75	10.73	
	Emamectin benzoate 1.9 %EC 250 Cm3/ Fed	3c	100%	pass	pass	6.5	6.1	454	265	52	52	9.75	9.75
			75%	pass	pass	6.46	6.1	603	265	49	52	9.75	9.75
3d		100%	pass	pass	4.84	4.94	355	338	34.4	35.21	10	10.73	
		75%	pass	pass	4.83	4.89	344	336	34.4	34.4	10.48	10.73	
3e		100%	pass	pass	6.36	6.39	241	232	43.4	46.28	10.73	10.73	
		75%	pass	pass	6.27	6.27	246	240	41.53	45	10.73	10.73	
3f	100%	pass	pass	6.61	6.6	306	311	54	54	10.48	10		
	75%	pass	pass	6.87	6.34	318	291	54	54	10.24	10		

3.4 Biological part

3.4.1 Toxicity of the synthesized surfactants on the 4th instar larvae of *Spodoptera littoralis*:

The results obtained from the experiments showed that all synthesized non-ionic surfactants (3a-f) with serial concentrations 5000, 10000, 20000, and 40000 mg/L don't have a lethal effect when applied to the 4th instar larvae of a laboratory strain of the cotton leaf-worm.

These findings agree with (Betana *et al.*, 2004), who established that none of the adjuvants tested (Castor bean oil, local mineral oil, PEG 600 DL, phosphoric acid, acetic acid, glue, and Arabic gum) had any harmful effects on *S. littoralis* larvae in their 4th instar when used at 0.3%.

3.4.2 Toxicity of emamectin benzoate alone against *S. littoralis*:

The insecticidal efficacy of emamectin benzoate against the 4th instar larvae of *S. littoralis* were displayed in **Table 4**. The findings demonstrated that, at all concentrations assessed, the mortality percentage increased with increasing concentration and exposure period. The mortality percentage recorded after 1, 2, and 3 days from treatment 76.66, 63.33, 50, 46.66, 40, and 30% for concentrations of 0.6, 0.4, 0.2, 0.02, 0.018, and 0.016 mg/L after 3 days, respectively.

Table 4. Mortality percentage of emamectin benzoate 1.9 %EC against 4th instar larvae of *S. littoralis*.

Tested formulation	Concentration mg/L	Mortality % after days		
		1	2	3
Emamectin benzoate 1.9 % EC	0.6	30	66.66	76.66
	0.4	26.66	50	63.33
	0.2	16.66	30	50
	0.02	13.33	20	46.66
	0.018	13.33	20	40
	0.016	10	16.66	30
Control		0	0	0

Emamectin benzoate was shown to be the most efficient insecticide of all tested insecticides against the cotton leaf-worm (Korrat *et al.*, 2012; El-Morshedy *et al.*, 2016). At 2, 3, and 4 days after treatment (DAT), the LC₅₀ values were, respectively, 8.8 x10⁻², 1.1 x10⁻², and 4.8 x10⁻³, ppm a.i. Emamectin benzoate generated the greatest lethal activity at 4 DAT, and the least lethal activity at 2 DAT. Emamectin benzoate's toxicity with the *S.*

littoralis 4th instar larvae is observed to increase with longer exposure times (Shawer *et al.*, 2022).

The lethal concentrations of emamectin benzoate to larvae of *S. littoralis* were shown in Table 5. Results showed that, the computed lethal concentration for emamectin benzoate LC₅₀ and LC₉₀ values were 0.103 and 26.500 ppm after 3 days of exposure, respectively. Also, the slope values of the toxicity line was 0.539 ± 0.307.

Table 5. Susceptibility of 4th instar larvae of cotton leaf-worm *Spodoptera littoralis* to emamectin benzoate without additives.

Tested formulation	LC ₅₀ (Lower-upper)	LC ₉₀ (Lower-upper)	Slope ± SE	R ²
Emamectin benzoate 1.9 % EC	0.103 (0.026±0.41)	26.500 (6.62±106.06)	0.539±0.307	0.803

SE = Standard error

3.4.3 Efficacy of emamectin benzoate after mixing with synthesized esters against *S. littoralis*:

The lethal concentration (LC₅₀) of the pesticide was combined with a 0.5% surfactant solution (3a-f) against cotton leaf worm larvae in their 4th instar. Data were recorded for 1, 2, and 3 days, and the results were summarized in Table 6. Results showed that, the mortality percentage increased with increasing concentration and exposure period with all

tested concentrations. After 3 days from treatment with the highest concentration of 0.1 mg/L for all treatments, it is evident that 3d, which has a death rate of 86.6%, is the highest ester (surfactant) that causes significant mortality rates when combined with LC₅₀ of pesticides. 3a, which has a mortality rate of 76.6%, followed by 3f, which has a mortality rate of 70%, before 3c and 3e, which have mortality rates of 66.6% and 63.3%, respectively.

Table 6. Mortality percentage of insecticides after mixed with synthesis mono-esters against 4th instar larvae of *S. littoralis*.

Tested formulation	Concentration mg/L	Mortality % after days			
		1	2	3	
Emamectin benzoate 1.9 % EC	+ 3a	0.1	40	70	76.6
		0.08	33.3	53.3	66.6
		0.06	33.3	46.6	60
		0.04	30	46.6	56.6
		0.02	16.6	20	33.3
	+3b	0.1	36.6	56.6	63.3
		0.08	36.6	56.6	60
		0.06	33.3	46.6	56.6
		0.04	26.6	40	46.6
		0.02	13.3	26.6	26.6
	+3c	0.1	23.3	53.3	66.6
		0.08	20	46.6	63.3
		0.06	16.6	50	60
		0.04	20	50	53.3
		0.02	6.6	43.3	46.6
	+3d	0.1	26.6	83.3	86.6
		0.08	13.3	76.6	83.3
		0.06	33.3	73.3	76.6
		0.04	10	63.3	66.6
		0.02	6.6	33.3	43.3
+3e	0.1	26.6	66.6	66.6	
	0.08	16.6	56.6	63.3	
	0.06	16.6	50	60	
	0.04	13.3	30	53.3	
	0.02	0	26.6	46.6	
+3f	0.1	23.3	63.3	70	
	0.08	13.3	50	63.3	
	0.06	16.6	53.3	60	
	0.04	10	30	50	
	0.02	6.6	23.3	40	
Control	0	0	0	0	

However, when locally produced surfactants are mixed with the pesticide under test, the physico-chemical characteristics of the evaluated insecticidal spray solutions were altered. These modifications result in improved field performance and insecticidal effectiveness. When used in direct mixtures with the tested pesticide, PEG 400 mono esters (3a-c) and PEG 600 mono esters (3d-f).

This outcome is in line with (Wasfy *et al.*, 2012) who reported that, the production of non-ionic surfactants like glycerol mono-laurate and diethylene glycol mono-laurate improved cotton leaf-worm control. Both soft water and hard water's pH were lowered by these surfactants. On the other hand, the physical and chemical characteristics of the spray solutions are changed when locally produced surfactants were added to the tested pesticides. These modifications improve pesticide efficiency and field performance. Additionally, concur with (Saad *et al.*, 2013) who reported that, the tested adjuvants (Ethoxylated octyl phenol acts as a wetting agent, and polyoxyethylene glycol (PEG 200) of laurate and oleate, butyl di-glycol, and Top Film as emulsifiers) increased the residual toxicity of the insecticides that were developed. When Lambda-cyhalothrin was combined with assessed adjuvants at the recommended rate, it proved more effective against

the *S. littoralis*. Also, agree with EL-Sisi *et al.*, (2011) who said that all the additives (PEG 600 di-laurate, PEG 600 mono-laurate, potassium sulfonate, Arabic gum, Glue, hydroxy methyl cellulose, poly-acryl amid, oxalic acid, citric acid, sulphonic acid, and tartaric acid) increased the 3/4 recommended rate's insecticidal effects on *S. littoralis* larvae in their 4th instar. The lethal concentrations of emamectin benzoate after mixing with surfactants to larvae of the cotton leaf-worm after 3 days from treatment are shown in Table 7. The highest ester that decreased the LC₅₀ and LC₉₀ of the investigated insecticide was 3d, with values of 0.021 and 0.129, respectively. Followed esters 3c and 3e, with respective LC₅₀ and LC₉₀ values of 0.024 and 0.304. Followed by 3f with LC₅₀ and LC₉₀ values of 0.033 and 0.729, respectively, followed by 3a, with LC₅₀ and LC₉₀ values of 0.048 and 0.358, respectively, and 3b, that showed high LC₅₀ and LC₉₀ values of 0.067 and 0.662, respectively. Additionally, the slope values for emamectin benzoate 1.9 % EC+ 3a, emamectin benzoate 1.9 % EC+3b, emamectin benzoate 1.9 % EC+3c, emamectin benzoate 1.9 % EC+3d, emamectin benzoate 1.9 % EC+3e and emamectin benzoate 1.9 % EC+3f were 1.468 ± 0.124, 1.292 ± 0.141, 0.647 ± 0.278, 1.618 ± 0.121, 0.647 ± 0.278 and 0.952 ± 0.190, respectively.

Table 7. Susceptibility of 4th instar larvae of cotton leaf-worm to emamectin benzoate 1.9% EC with mono-esters.

Tested formulation	LC ₅₀ (Lower-upper)	LC ₉₀ (Lower-upper)	Slope ± SE	R ²	
Emamectin benzoate 1.9 % EC	+ 3a	0.048 (0.027 ± 0.084)	0.358 (0.204 ± 0.627)	1.468 ± 0.124	0.970
	+3b	0.067 (0.036 ± 0.127)	0.662 (0.350 ± 1.251)	1.292 ± 0.141	0.985
	+3c	0.024 (0.007 ± 0.084)	2.304 (0.658 ± 8.070)	0.647 ± 0.278	0.968
	+3d	0.021 (0.012 ± 0.036)	0.129 (0.075 ± 0.223)	1.618 ± 0.121	0.998
	+3e	0.024 (0.007 ± 0.084)	2.304 (0.658 ± 8.070)	0.647 ± 0.278	0.968
	+3f	0.033 (0.014 ± 0.077)	0.729 (0.310 ± 1.716)	0.952 ± 0.190	0.964

Conclusion

A new group of esters was synthesized as surfactants, their chemical structures were elucidated, and they were evaluated both alone and in combination with the commercial pesticide that was recommended against the cotton leaf-worm. The results revealed that the newly prepared esters increased the pesticide's efficacy. Consequently, reducing the amount of pesticide used to manage pests and preserving the environment from the pollution hazards.

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تخليق بعض العوامل النشطة سطحياً الجديده وخصائصها الطبيعية والكيميائية وتأثيراتها التحسينية للإيمامكتين بنزوات ضد حشرة دودة ورق القطن

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تعد الإسترات من بين المركبات المنتجة في الصناعات في جميع أنحاء العالم وتعتبر منتجات مهمة بسبب استخدامها في إنتاج مبيدات الحشائش والمبيدات الحشرية والدهانات والمذيبات والبلستيك. الطريقة الأكثر استخداماً لتصنيع الإسترات هي تفاعل أسترة فيشر. تم تفاعل الأحماض الدهنية (الكابريك، اللوريك، والبالمتيك) مع البولي إيثيلين جلايكول (PEG 400 و PEG 600) لتصنيع مواد خافضة للتوتر السطحي غير الأيونية، ثم تم توضيح تركيبها بواسطة طيف الأشعة تحت الحمراء ومقياس الطيف الكتلي، ثم تم تقدير خواصها الطبيعية والكيميائية. مثل (CMC،HLB، الحموضة الحرة أو القلوية والذوبان). تم تقدير الخصائص الطبيعية والكيميائية لمحلول رش المبيدات بشكل منفصل وبعد خلطه مع الإسترات. بعد ذلك تمت معاملة يرقات العمر الرابع لدودة ورق القطن بتراكيز تسلسلية من الإسترات المحضرة وحدها والمبيد الموصى به وكذلك أيضاً تمت معاملتها بـ LC₅₀ للمبيد مع الإسترات. أظهرت النتائج أن المواد الخافضة للتوتر السطحي غير الأيونية المحضرة حسنت الخصائص الطبيعية والكيميائية لمحاليل الرش. ونتيجة لخلط المبيدات مع المواد الخافضة للتوتر السطحي، زادت لزوجة المبيدات و التوصيليه الكهربيه بينما انخفض التوتر السطحي وقيمة الرقم الهيدروجيني. أظهرت نتائج الاختبار الحيوي أيضاً أن جميع المواد الخافضة للتوتر السطحي خفضت قيمة LC₅₀ للمبيد الحشري emamectin benzoate 1.9% EC، إلا أن إضافة PEG 600 MC إلى المبيد كان له أفضل التأثيرات، حيث خفض قيمة LC₅₀ إلى 0.021 جزء في المليون. وبالتالي، بعد إكمال الدراسات اللازمة على هذه الإسترات، فمن الممكن ان يوصى بدمجها مع المبيدات الحشرية الموصى بها تجارياً من أجل تقليل كمية المبيدات المستخدمة وبالتالي تلوث البيئة.