

ORIGINAL ARTICLE

## Role of etofenprox nanoformulation in suppression of the silver whitefly, *Bemisia tabaci* and its residue in eggplant fruits

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### Abstract

The normal formulation of etofenprox was developed to nanoformulation and used against the adults of silver whitefly, *Bemisia tabaci* in eggplant fields. Three concentrations of both the normal and nanoformulations were used. The concentrations of etofenprox nanoformulation were one-fifth of the normal formulation. The nanosize of etofenprox ranged from 225 to 489 nm. The loading capacity of etofenprox was  $60.7 \pm 5.7\%$ . The obtained results showed that the  $LC_{50}$  of the normal formulation was four times more than the nanoformulation. The  $LC_{50}$  for the nanoformulation was 0.9 and 3.5 ppm for the normal formulation of etofenprox. This means that the nanoformulation of etofenprox was more effective than the normal. The residues of both nano and normal formulations were determined in eggplant fruits after three applications. The obtained results showed that the residue of nanoformulation after 1 hour of treatment was  $0.51 \pm 0.03$  compared with  $0.62 \pm 0.03 \text{ mg} \cdot \text{kg}^{-1} \pm \text{SD}$  in normal formulation. After 1 hour of treatment the residue of etofenprox was reduced to  $0.11 \pm 0.1$  and  $0.22 \pm 0.02 \text{ mg} \cdot \text{kg}^{-1} \pm \text{SD}$  in nano and normal formulations, respectively. The dissipation rates of both nano and normal formulations after 1 hour were 78.3 and 64.5%, respectively. The degradation rate ( $K$ ) in nanoformulation and normal etofenprox was 1.33 and  $0.73 \text{ mg} \cdot \text{kg}^{-1} \pm \text{SD}$ , respectively. The residue half-life ( $LR_{50}$ ) was 0.52 and 1 day, respectively. The preharvest interval (PHI) was 6 days for both nano and normal etofenprox formulations. The results confirmed that nanoetofenprox was more effective against *B. tabaci* adults, with lower persistence and lower residue than the normal formulation of etofenprox.

**Keywords:** *Bemisia tabaci*, eggplant, nanoformulations, reduction percentages, residues

## Introduction

Silver whitefly, *Bemisia tabaci* (Gennadius) (Hemiptera : Aleyrodidae) is considered to be one of the major pests on eggplant, *Solanum melongena* (Solanales : Solanaceae). This pest can infest eggplant leaves and reduce eggplant yield (Touhidul and Shunxiang 2009). It is primarily found sucking the sap from the undersides

of leaves and covering them with sticky honeydew. This allows black sooty mold to grow over the honeydew which lowers the photosynthetic capacity of the plant and makes the fruit unattractive (Hirano *et al.* 1995). The efficacy of *B. tabaci* on eggplant is not only as a sucking pest but also as a virus vector (Sani *et al.*

2020). The economic damage caused by *B. tabaci* can reach up billions of USD in many crops (Hasan *et al.* 2019). Many efforts have been carried out to suppress the population of this pest. Chemical control has been used as an effective agent to reduce and eradicate the silver whitefly such as neonicotinoids (Palumbo *et al.* 2001), avermectin and pyriproxyfen (Wang *et al.* 2020). Due to the extensive use of normal pesticide formulations, this pest has acquired resistance to all normal chemical formulations (Sani *et al.* 2020).

Although there are side effects of chemical control application these methods is still the most effective method of pest control. Therefore, there is a dire need to find innovative strategies that create new formulations which are less toxic on nontarget organisms and at the same time are very effective against the target pest. As a result, the use of nanoformulations of pesticides is the only solution. Nanopesticide formulations have a potentially bright future for the development of more effective and safer pesticides/biopesticides (Deka *et al.* 2021). Nanopesticides can reduce the required amount of pesticide for pest control by increasing the durability and efficacy of chemicals (Demir 2020).

Etofenprox, a pyrethroid ethyl, is a new pesticide used against a wide range of insect pests such as thrips, whiteflies and aphids (Sabry *et al.* 2018). The nanoformulation of etofenprox was used against *Spodoptera litura* (Mohd *et al.* 2017) and it was more toxic than the normal formulation (Sabry and Hussein 2022).

Lee and Im (2021) determined the residue of etofenprox in rice, while Malhat *et al.* (2012) determined the residue of etofenprox in tomato fruits.

This work aimed to use a new strategy of integrated pest management as a promising trend for silver whitefly control that would reduce the insecticide residues by using nanoformulation of etofenprox.

## Materials and Methods

### Tested insecticide

Etofenprox (Infinity 5% EC) is produced by Astranova, Turkey. The recommended field rate is 200/feddan (feddan = 2400 m<sup>2</sup>). This means that the recommended field rate is 25 ppm. Three concentrations were used: the recommended field rate (25 ppm), half of the recommended field rate (12.5 ppm) and one-fourth of the recommended field rate (6.25 ppm). Three concentrations of etofenprox nanoformulations were used. The nanoformulation concentrations were one-fifth of the normal concentrations (5, 2.5 and 1.25 ppm).

### Tested insect

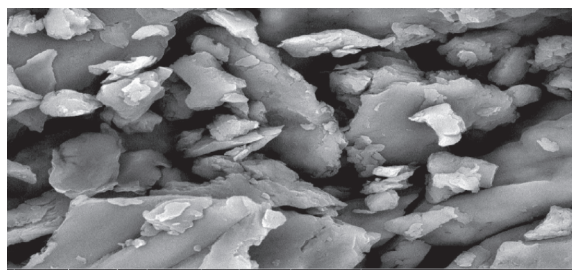
This experiment was carried out under semifield conditions. The adults of silver whitefly, *B. tabaci* in eggplant fields were tested against both the normal and nanoformulation of etofenprox. This work was carried out in the eggplant field of the Agriculture Research Center. Only the adults of *B. tabaci* were tested against both tested formulations (nano and normal formulations of etofenprox).

### Preparation of nanoformulations

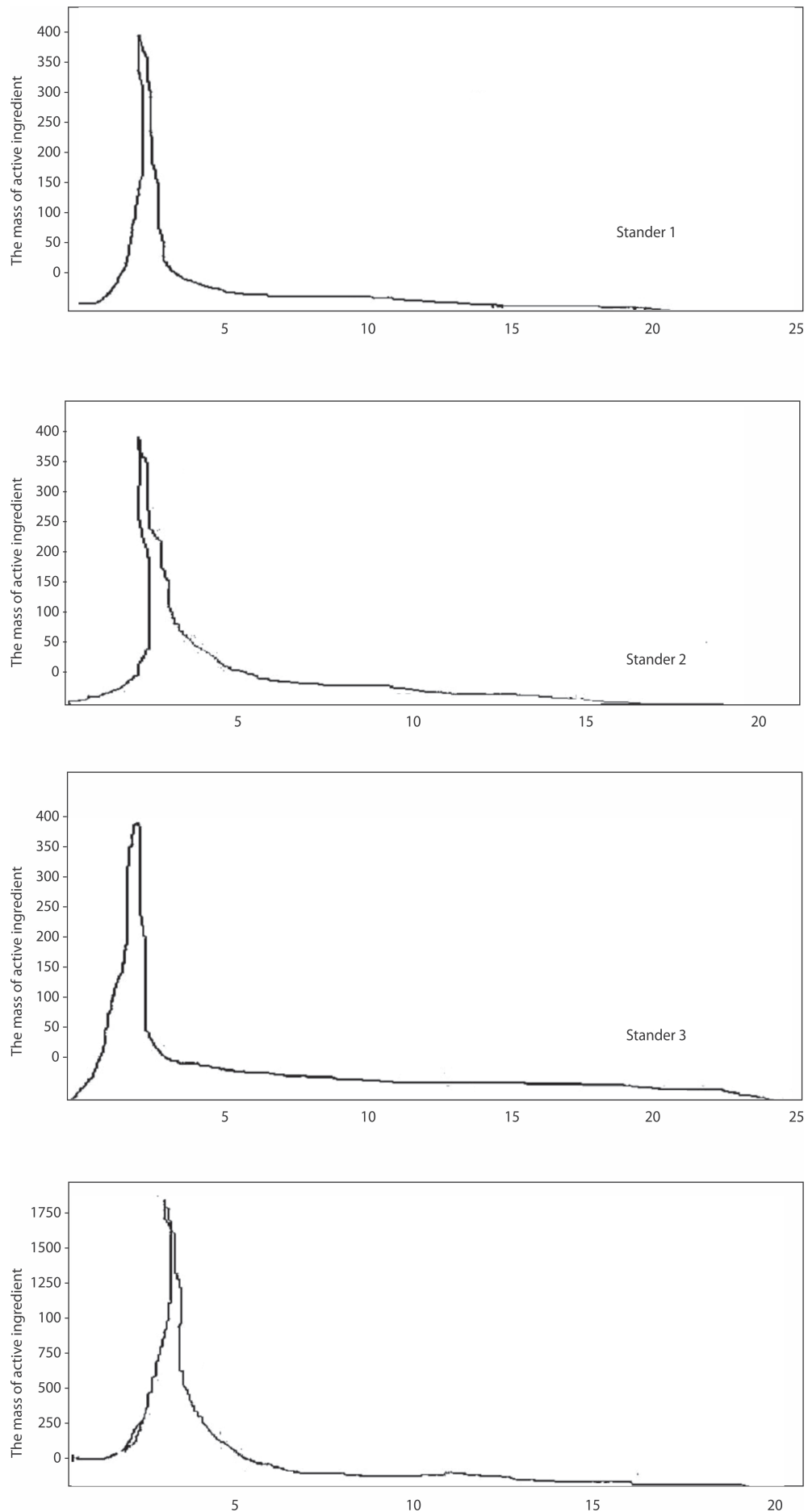
It was known that chitosan does not dissolve in water. So, 2% acetic acid solution (with distilled water) was used for dissolving chitosan (120 ml). This solution (chitosan and acetic acid 2%) was stirred with a magnetic stirrer for 25–30 min followed by sonication until the solution became transparent. When the solution became transparent it meant that the particles of chitosan had converted into nano size. Tripolyphosphate 0.8% (w/v) was dissolved in conductive water (120 ml normal water). One-fifth ml of etofenprox was added to the tripolyphosphate 0.8% solution. The obtained solution was added dropwise to the previous solution (acetic acid 2% acetic acid 2% and chitosan) with continuous stirring for 20–40 min. The suspension was centrifuged at 10 000 rpm for 30 min. The pellet was collected and lyophilized to obtain a nanoparticle formulation (Vaezifar *et al.* 2013). The obtained nanoparticles were photographed under SEM (scanning electron microscope) (Fig. 1).

### Loading capacity measurement

To make sure that the insecticide formulation (etofenprox) was loaded on the carrier or polymer used (chitosan) the loading capacity was tested. After preparing the etofenprox nanoformulations the loading capacity was determined according to He *et al.* (2017). This determination was carried out by using about 30 mg of the obtained sample (etofenprox nanoparticle formulation) and dissolved in 50 ml of acetonitrile. This mixture was placed in a shaking



**Fig. 1.** Etofenprox nanoparticles under scanning electron microscope (SEM)



**Fig. 2.** Loading capacity of etofenprox nanoparticles

tank overnight at a constant temperature to completely dissolve the carrier material. The obtained solution was filtered and the mass concentration of the tested pesticide in acetonitrile was examined by HPLC (high performance liquid chromatograph) [The HPLC system was equipped with an XTerra RP18 column, 5  $\mu\text{m}$  particle size, 4.6 mm internal diameter  $\times$  250 mm length (Waters®, USA)] under a detection wavelength of 278 nm (Fig. 2). The loading capacity was calculated by dividing the mass of loading pesticide (etofenprox) on the mass of etofenprox nanoparticles  $\times$  100.

According to this formula the loading capacity of etofenprox on chitosan nanoparticles was  $60.7 \pm 5.7\%$  (Fig. 2).

## Field bioassay

This experiment was carried out under semifield conditions in the Agricultural Research Center, Egypt, from August 2021 to March 2022. The temperature ranged between 20 and 35°C, whereas relative humidity was moderate, ranging between 49 and 75%. An area of 300 m<sup>2</sup> was cultivated, at the beginning of August with eggplant variety Anann. This experiment was divided into seven plots (three for the nanoformulation). Concentrations, three for normal concentrations and one for control (treated with water). Each concentration had three replicates. Each replicate was 6  $\times$  7 m. All plots were treated with the tested formulations three times at 1 week intervals. A 10 l knapsack sprayer was used in field applications. Eggplants were sprayed with both of the tested formulations (normal and nanoformulations). The numbers of silver whitefly adults per 10 eggplant leaves were counted randomly 24 h after each treatment in each replicate. Corrected efficacy of adult whitefly was calculated according to Henderson and Tilton (1955) as follows:

$$\text{Corrected mortality [\%]} =$$

$$= 1 - \frac{\text{No. whitefly in control before treatment} \times \text{No. whitefly in treatment after treatment}}{\text{No. whitefly in control after treatment} \times \text{No. whitefly in treatment after treatment}} \times 100.$$

The percentage of adult reduction was calculated by: the original number of adults before treatments – the new number after treatment/original number  $\times$  100. The lethal concentration for 50% of insect population LC<sub>50</sub> was determined. The percentages of adult mortality (corrected mortality) were calculated after each treatment and the LC<sub>50</sub> was calculated by Proban Software Program Version 4.4.

## Residue determination in eggplant fruits

### Sample extraction and cleanup method

QuEChERS method was conducted by using 10 g of well homogenized eggplant samples applicated by etofenprox in 10 ml of acetonitrile, then shaken well horizontally for 1 min. Mixed salts consisted of 4 g magnesium sulfate, 1 g sodium chloride, 1 g sodium citrate dibasic sesquihydrate and 0.5 g sodium citrate tribasic dehydrate supplied from Interchim (USA) and were added to the tube, which were centrifuged 5000 rpm speed for 5 min, and finally 2 ml of supernatant were filtered by a PTFE filter 0.22  $\mu\text{m}$  in glass vials to be injected.

### Instrumentation

High-performance liquid chromatography (Agilent HPLC1260) using UV-detector set at wave length 225 nm. Column Eclipse XDB-C18 (5  $\mu\text{m}$ , 4.6  $\times$  250 mm), and the mobile phase acetonitrile/water (85 : 15, v/v) at flow rate: 0.7 ml  $\cdot$  min<sup>-1</sup>. These conditions resulted in good separation and high sensitivity was obtained.

The recovery rate and precision of the method [expressed as relative standard deviation (RSD), %] were measured by analyzing replicate pesticide-free eggplant, which were fortified at concentrations of 0.01, 0.5 and 3 mg  $\cdot$  kg<sup>-1</sup>. The results were corrected depending on the recovery rate. The mean recovery percentages of etofenprox ranged from 89.42 to 98.5% as shown in Table 1. The sensitivity was evaluated by determining the limit of detection (LOD) and limit of quantification (LOQ) according to Lehotay *et al.* (2010). The LOD value recorded was 0.01 mg  $\cdot$  kg<sup>-1</sup>, while the LOQ value was 0.03 mg  $\cdot$  kg<sup>-1</sup>.

## Kinetic studies

The degradation rate was calculated mathematically according to Timme and Frehse (1980).

The first order kinetic using common logarithms as in the following equation:

$$\log R = \log R_0 - 0.434 Kt,$$

where:  $R_0$  – residue level at the initial time (zero time),  $R$  – residue level at interval (days) after application,  $Kt$  – degradation rate constant at the successive intervals in days.

Residue half-life value (RL<sub>50</sub>) was calculated mathematically according to Moye *et al.* (1987) from the following equation:

$$RL_{50} = \ln 2 / K = 0.6932 / K,$$

where:  $K$  – rate of decomposition.

## Statistical analysis

Data were subjected to the analysis of variance test (ANOVA) via Randomized Complete Block Design (RCBD) (F-test) and analysis of variance (one way classification ANOVA) followed by a least significant difference (LSD) at 5% (Costat 1990)

## Results and Discussion

The adults of silver whitefly, *B. tabaci* were tested with three concentrations of both normal and nanoformulations.

### Toxicity of both normal and nanoformulations against the adults of silver whitefly adults, *Bemisia tabaci*

The lethal concentration of 50% of the tested insect population was determined with both normal and nanoformulations (Table 1).

Data showed that the  $LC_{50}$  after the first treatment were 3.6 and 12.5 ppm for the nano and normal formulations, respectively (Table 2). This means that the nanoformulation was more effective (four times) against adults of *B. tabaci*. This efficacy was increased with the second treatment to five times.

The  $LC_{50}$ s were 1.7 and 8.9 ppm, respectively. The same results were obtained after the third treatment. The efficacy of nanoformulation was more effective than the normal formulation. These results showed

that the nanoformulation of etofenprox was more potent than the normal formulation.

### The reduction effect of the first concentration (recommended field rate) of both normal and nanoformulations against silver whitefly adults, *Bemisia tabaci*

The percentages of adult reduction were calculated after each treatment. After the first treatment the percentages of adult reduction were 66.8 and 68.6% with the nano and normal formulations, respectively (Table 3).

As mentioned in Table 3 after the first treatment the population of silver whitefly decreased. The percentage of reduction was 66.8 and 68.6% for nano and normal formulation, respectively. After the second treatment the percentage of reduction increased to 90.3 and 85.8% in both formulations, respectively. The efficacy of both formulations also increased after the third application. The percentage of silver whitefly reduction reached 96.6 and 97.6% for nano and normal formulations. The statistical analysis (*F*-test) showed that there was no significant difference between the normal and nanoformulation. However, there was a very significant difference between each formulation and the control.

### The reduction effect of the second concentration (half of recommended field rate) of both normal and nanoformulations against silver whitefly adults, *Bemisia tabaci*

The obtained results showed that the efficacy of the second concentration was approximately the same as the first concentration (Table 4). The percentages of reduction after the first treatment were 59.7 and 58.1% for nano and normal formulations, respectively. These percentages reached 92.6 and 89.6%, respectively, after the third treatment. Statistically there was no difference between the efficacy of nano and normal formulations.

**Table 1.** Recovery rate of etofenprox

| Level<br>[mg · kg <sup>-1</sup> ] | Etofenprox    |     |       |          |
|-----------------------------------|---------------|-----|-------|----------|
|                                   | Rec % ± SD    | RSD | LOD   | LOQ      |
| 3                                 | 98.5 ± 0.28   | 2.8 |       |          |
| 0.5                               | 89.42 ± 0.043 | 5.3 | 0.010 | 0.032242 |
| 0.01                              | 92.6 ± 0.001  | 2.5 |       |          |

RSD – relative standard deviation; LOD – limit of detection; LOQ – limit of quantification; Rec – recovery rate

**Table 2.** Toxicity of etofenprox normal and nanoformulation against the adults of *Bemisia tabaci*

| Etofenprox         | The lethal concentration for 50% of insect population ( $LC_{50}$ ) |                              |                       |                              |                       |                              |
|--------------------|---------------------------------------------------------------------|------------------------------|-----------------------|------------------------------|-----------------------|------------------------------|
|                    | after 1st application                                               |                              | after 2st application |                              | after 3st application |                              |
|                    | Slope ± SE                                                          | $LC_{50}$ and fiducial limit | Slope ± SE            | $LC_{50}$ and fiducial limit | Slope ± SE            | $LC_{50}$ and fiducial limit |
| Normal formulation | 1.1 ± 0.3                                                           | 12.5<br>(4.3 – 18.1)         | 1.4 ± 0.7             | 8.9<br>(5.1 – 12.6)          | 1.6 ± 0.5             | 3.5<br>(0.4 – 6.8)           |
| Nanoformulation    | 1.5 ± 0.3                                                           | 3.6<br>(2.5 – 4.5)           | 1.8 ± 0.3             | 1.7<br>(0.8 – 2.3)           | 1.9 ± 0.5             | 0.9<br>(0.2 – 1.5)           |

**Table 3.** Role of both nano and normal formulations of etofenprox against *Bemisia tabaci* in eggplant by the first concentration (recommended field rate)

| Formulations | No. of <i>B. tabaci</i> /leaves |                     |                        |                |                     |                        |                |                     |                        |                |
|--------------|---------------------------------|---------------------|------------------------|----------------|---------------------|------------------------|----------------|---------------------|------------------------|----------------|
|              | before treatment                | after 1st treatment | corrected efficacy [%] | % of reduction | after 2nd treatment | corrected efficacy [%] | % of reduction | after 3rd treatment | corrected efficacy [%] | % of reduction |
| Nano         | 38.3 ± 2.5                      | 12.7 ± 4.6 b        | 72.5                   | 66.8           | 3.7 ± 1.2 b         | 92.1                   | 90.3           | 1.3 ± 1.5 b         | 97.2                   | 96.6           |
| Normal       | 42.3 ± 12.2                     | 13.3 ± 2.1 b        | 73.9                   | 68.6           | 6.0 ± 1.0 b         | 88.5                   | 85.8           | 1.0 ± 1.0 b         | 98.1                   | 97.6           |
| Control      | 45.0 ± 8.0                      | 54.3 ± 6.0 a        |                        |                | 55.3 ± 9.3 a        |                        |                | 54.0 ± 6.2 a        |                        |                |
| F-values     |                                 | 82.7*               |                        |                | 86.4*               |                        |                | 197.8*              |                        |                |
| LSD          |                                 | 9.1                 |                        |                | 10.9                |                        |                | 7.5                 |                        |                |

Means under each treatment sharing the same letter in a column are not significantly different at ( $p = 0.05$ ); \*there are significant difference among all treatments

**Table 4.** Role of both nano and normal formulations of etofenprox against *Bemisia tabaci* in eggplant by second concentration (half of the recommended field rate)

| Formulations | No. of <i>B. tabaci</i> /leaves |                     |                        |                |                     |                        |                |                     |                        |                |
|--------------|---------------------------------|---------------------|------------------------|----------------|---------------------|------------------------|----------------|---------------------|------------------------|----------------|
|              | before treatment                | after 1st treatment | corrected efficacy [%] | % of reduction | after 2nd treatment | corrected efficacy [%] | % of reduction | after 3rd treatment | corrected efficacy [%] | % of reduction |
| Nano         | 44.7 ± 6.5                      | 18.0 ± 3.0 b        | 62.3                   | 59.7           | 10.7 ± 1.5 b        | 79.3                   | 76.1           | 3.3 ± 1.5 b         | 93.4                   | 92.6           |
| Normal       | 41.3 ± 2.5                      | 17.3 ± 3.1 b        | 60.8                   | 58.1           | 13.3 ± 1.5 b        | 72.1                   | 67.8           | 4.3 ± 0.6 b         | 90.6                   | 89.6           |
| Control      | 38.7 ± 2.5                      | 41.3 ± 2.5 a        |                        |                | 44.7 ± 7.0 a        |                        |                | 43.0 ± 9.6 a        |                        |                |
| F-values     |                                 | 68.2*               |                        |                | 59.6*               |                        |                | 48.1*               |                        |                |
| LSD          |                                 | 5.7                 |                        |                | 8.5                 |                        |                | 11.3                |                        |                |

Means under each treatment sharing the same letter in a column are not significantly different at ( $p = 0.05$ ); \*there are significant difference among all treatments

### The reduction effect of the third concentration (one-fourth of the recommended field rate) of both normal and nanoformulations against silver whitefly adults, *Bemisia tabaci*

The reduction percentages with the third concentration (one-fourth of the recommended concentration) were 46.5 and 36.1% after the first treatment for the nano and normal formulations, respectively (Table 5). These percentages increased after the second treatment to 76.1 and 67.8%, respectively. After the third treatment the percentages of reduction reached 81.4 and 80.4%, respectively. The obtained results showed that the first concentration (recommended field rate) and the second concentration (half of the recommended field rate) were more effective against adults of *B. tabaci* than the third concentration.

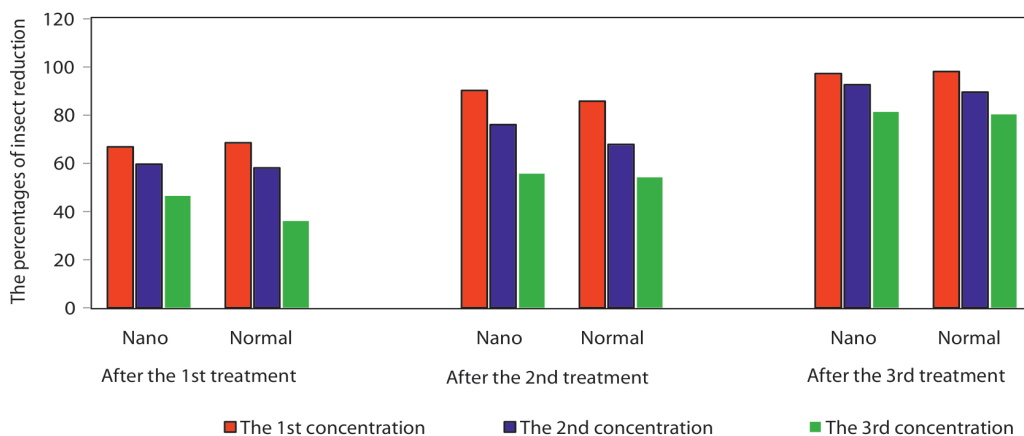
The first concentration of nanoformulation was approximately equal to the second concentration, 96.6 and 92.6%, respectively (Fig. 3) after the third application. This means that the second concentration could be used as an effective concentration against *B. tabaci* to reduce the concentration and environmental

contamination. The efficacy of the third concentration after the third treatment was 81.4% with nanoformulation. This percentage was more efficient to suppress the silver whitefly population in the field. The third concentration was 1.25 ppm. On the other hand, the third concentration with the normal formulation was 6.25 ppm. This means that one-sixth of the normal formulation could be used and get the same results. This result was consistent with Mohd *et al.* (2017) who got the same results with the tobacco caterpillar, *Spodoptera litura*. The nanoformulation of etofenprox was more effective than the normal formulation. The  $LC_{50}$  were 0.0175 and 0.0390%, respectively. Zaki *et al.* (2019) tested the nanoformulation of deltamethrin against the greenhouse whitefly, *Trialeurodes vaporariorum*. The obtained results showed that the nanoformulation of deltamethrin caused 82.95% mortality compared to 38.77% with the normal formulation. Shahid *et al.* (2022) used a nanosilver formulation against 3rd instar nymphs and adults of *B. tabaci*. Their results showed that the nanoformulation of silver can be used as an effective agent in integrated pest management (IPM) for the silver whitefly. Shanmugapriya *et al.* (2019) used

**Table 5.** Role of both nano and normal formulations of etofenprox against *Bemisia tabaci* in eggplant by the third concentration (one-fourth of the recommended field rate)

| Formulations     | No. of <i>B. tabaci</i> /leaves |                     |                        |                |                     |                        |                |                     |                        |                |
|------------------|---------------------------------|---------------------|------------------------|----------------|---------------------|------------------------|----------------|---------------------|------------------------|----------------|
|                  | before treatment                | after 1st treatment | corrected efficacy [%] | % of reduction | after 2nd treatment | corrected efficacy [%] | % of reduction | after 3rd treatment | corrected efficacy [%] | % of reduction |
| Nano             | 46.7 ± 9.1                      | 25.0 ± 3.0 b        | 49.9                   | 46.5           | 20.7 ± 3.8 b        | 63.3                   | 55.7           | 8.7 ± 0.6 b         | 84.7                   | 81.4           |
| Normal           | 44.3 ± 8.0                      | 28.3 ± 2.9 b        | 40.2                   | 36.1           | 20.3 ± 1.5 b        | 62.1                   | 54.2           | 8.7 ± 0.6 b         | 83.9                   | 80.4           |
| Control          | 40.0 ± 9.6                      | 42.7 ± 7.2 a        |                        |                | 48.3 ± 8.3 a        |                        |                | 48.7 ± 2.1 a        |                        |                |
| <i>F</i> -values |                                 | 11.4*               |                        |                | 27.1*               |                        |                | 960.0*              |                        |                |
| LSD              |                                 | 9.6                 |                        |                | 10.7                |                        |                | 2.6                 |                        |                |

Means under each treatment sharing the same letter in a column are not significantly different at ( $p = 0.05$ ); \*there are significant difference among all treatments

**Fig. 3.** Efficacy of both nano and normal formulations of etofenprox against *Bemisia tabaci* adults after different treatments

azadirachtin loaded in silica nanoparticles against *B. tabaci*. The obtained results showed that azadirachtin loaded in silica nanoparticles can be used as an alternative agent to conventional pesticide formulations against *B. tabaci*.

On the other hand, Sabry and Hussein (2022) found that the  $LC_{50}$  of etofenprox normal formulation and nanoformulation were 14.9 and 20.3 ppm against adults of the chocolate banded snail, *Eobania vermiculata*. This means that the normal formulation of etofenprox was more toxic than nanoformulation.

### Residue determination of both normal and nanoformulations of etofenprox in eggplant fruits

The residue of etofenprox in both normal and nanoformulations were determined in eggplant fruits (Table 6).

The present study was conducted to investigate the residual levels and dissipation behavior of the

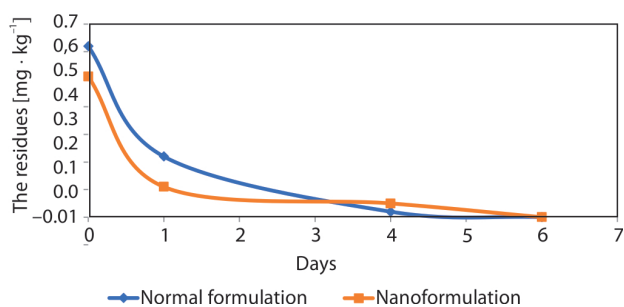
conventional formulation of etofenprox and its nanoformulation in eggplant, *S. melongena* L. fruits under greenhouse conditions. The insecticide residues were estimated after application of both tested insecticides with three replications at the recommended rate. Residual amounts, rate of degradation ( $K$ ), preharvest interval (PHI), kinetic equation; half-life ( $RL_{50}$ ) and Regression coefficient ( $R$ ) of insecticide residue dissipation were calculated and summarized in Table 6.

The concentration of etofenprox residues in eggplant after application with a normal formulation were 0.62, 0.22, 0.02 and 0.0 mg · kg<sup>-1</sup> at 1 hour, 1, 4, and 6 days after application, respectively, while for the nanoformulation they were 0.51, 0.11, 0.05 and 0.0 mg · kg<sup>-1</sup> at 1 hour, 1, 4 and 6 days after application, respectively. As reported, the average of etofenprox residue in eggplant declined with time and was non-detectable 6 days after application. The results showed that the residue of nanoetofenprox was less than the normal formulation after 1 hour and 1 day

**Table 6.** Residue of etofenprox in both nano and normal formulations in eggplant fruits after different times

| Time [days]                      | Normal formulation<br>[mg · kg <sup>-1</sup> ] | RSD<br>[%] | Nanoformulation<br>[mg · kg <sup>-1</sup> ] | RSD<br>[%] |
|----------------------------------|------------------------------------------------|------------|---------------------------------------------|------------|
| After 1 hour                     | 0.62 ± 0.03                                    | 4.8        | 0.51 ± 0.03                                 | 5.8        |
| After 1 day                      | 0.22 ± 0.02                                    | 9.2        | 0.11 ± 0.10                                 | 12.1       |
| Dissipation%                     | 64.5%                                          |            | 78.3%                                       |            |
| After 4 days                     | 0.02 ± 0.003                                   | 13.4       | 0.05 ± 0.009                                | 17.4       |
| Dissipation%                     | 96.7%                                          |            | 90.2%                                       |            |
| After 6 days                     | 0.0                                            |            | 0.0                                         |            |
| Dissipation [%]                  | 100%                                           |            | 100%                                        |            |
| MRL [mg · kg <sup>-1</sup> ]     |                                                |            | 0.01                                        |            |
| PHI [days]                       | 6                                              |            | 6                                           |            |
| Regression equation              | $y = -0.3449x - 0.247$                         |            | $y = -0.3596x - 0.5671$                     |            |
| Regression coefficient ( $R^2$ ) | $R^2 = 0.9956$                                 |            | $R^2 = 0.98$                                |            |
| $K$                              | 0.73                                           |            | 1.33                                        |            |
| RL <sub>50</sub> [days]          | 1                                              |            | 0.52                                        |            |

MRL – maximum residue limit; PHI – preharvest interval;  $K$  – rate of decomposition; RSD – relative standard deviation

**Fig. 4.** The residues in both normal and nanoformulations

after application. The residue in normal formulation sharply decreased after 4 days compared to the nanoformulation (Fig. 4).

The dynamics of etofenprox residue could be described by the equation:  $y = -0.3449x - 0.247$ , with  $R^2 = 0.9956$  and  $y = -0.3596x - 0.5671$ , with  $R^2 = 0.98$  for conventional and nanoetofenprox, respectively.

The obtained results showed that etofenprox normal and nanoformulations in eggplant fruits were not detected 6 days after application, where the degradation rates ( $K$ ) were 0.73 and 1.33 day<sup>-1</sup>, in normal and nanoformulation (Table 6). This means that the degradation rate of nanoformulation was more than the normal formulation. This observation may explain why the residue in nanoformulation was less than the normal formulation. The half-life (RL<sub>50</sub>) time values were 1 and 0.52 days, respectively. This observation showed that the half-life of nanoformulation was half of the normal formulation. European Union MRL for etofenprox in eggplant was 0.01 mg · kg<sup>-1</sup>. The obtained data revealed that eggplant fruits could be consumed

safely after 6 days of the application depending on the maximum residue limit (MRL) of etofenprox in eggplant. Watanabe and Baba (2015) determined the etofenprox residue in eggplant fruit by using (HPLC-FLD). Hwang *et al.* (2015) determined the dissipation rate of etofenprox in spring onion under greenhouse conditions. The obtained results showed that the half-life of etofenprox in spring onion was 9.5 days.

## Conclusions

The normal formulation of etofenprox was developed to the nanoformulation. The main benefit of this development was to reduce the concentration used, reduce the cost of application, reduce the insecticide residues and therefore reduce the environmental contamination. The LC<sub>50</sub> of nanoformulation was 3 to 6 times lower than the normal formulation. This means the efficacy of nanoformulation was more toxic than the normal one against the target insect. Using nanoformulation can achieve the difficult formula by decreasing the insecticide concentration and increasing efficacy. The residue of etofenprox nanoformulation was lower than the normal formulation in eggplant fruits after zero time (1 hour), 1 day. The persistence of the nanoformulation was less than the normal formulation. The obtained results concluded that the nanoformulation of etofenprox was more effective than the normal formulation. The results also concluded that the persistence of nanoformulation in eggplant fruits was less than the normal formulation.



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