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Efficacy of Zinc Oxide Nanoparticle Adding to Two Neonicotinoids Pesticides against *Spodoptera litura* (Lepidoptera: Noctuidae) and *Aphis gossypii* Glover

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ABSTRACT

In recent years, pesticide residues have become a danger to the food chain, the environment, and human health. Some nano metal oxides had been applied with pesticides to solve these problems and enhanced their insecticidal Activity. This new technology for the complete mineralization of pesticides is necessary to convert them to non-toxic forms. In this study, we investigated the synergistic effect of ZnO nanoparticles (NPs) with some neonicotinoid pesticides (Thioxam 25%WG and Actara 25% WP) against 4th instar larvae of *Spodoptera litura* (Lepidoptera: Noctuidae) and cotton aphid, *Aphis gossypii* Glover (Homoptera: Aphididea). These cotton pests were allowed to feed on castor leaves after impregnated with pesticides at recommended rate with the composite of ZnO (NPs). Observations showed an increase in mortality (48.57 %, 93.51 % increased mortality). The addition of the ZnO nanoparticles to insecticides increased the insecticidal activities and a reduction (%) against tested cotton pests. The synthesized nano metal oxide was characterized by different techniques as FTIR spectroscopy, TEM and UV spectroscopy used for a determination band gap of the prepared ZnO NPs. This present study suggests that nanoparticles metal oxide as (ZnO) applying as eco-friendly nanomaterial in the field of pest control.

INTRODUCTION

In all the world, the crops were raided by various insects' groups, causing sharp damage to plants (Wardle, *et al.*, 2004). Different groups of synthetic insecticides were much used to control these pests (Damalas and Koutroubas, 2016; Jameel, *et al.*, 2019). Pesticides are mostly used in agricultural production in controlling pests, fungi, and weeds. Therefore, there are extensively pesticide residues in soils, ground waters, and drinking waters (Nasrabadi, *et al.*, 2011; Gilliom, *et al.*, 1999).

Manganese, ferric, zinc, aluminum, magnesium, cerium, and titanium nanometal oxides are extremely effective adsorbents for a wide range of pesticides. Also, they have a high adsorption capacity, low cost, high surface area, and short diffusion distance (Armaghan and Amini, 2012; Moradi Dehaghi, *et al.*, 2014; Tavakkoli, Yazdanbakhsh, 2013; Cheng, 2013; Bardajee, and Hooshyar, 2013). In the last years, many nanometal particles are applied for progressing the insecticidal activity each alone or with the combined form against the different insects (Johnston, *et al.*, 2009; Malaikozhundan, et *al.*, 2017; Ishwarya, *et al.*, 2018). Instead of traditional insecticides (e.g., neonicotinoids) were

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introduced into the market because they have more resistant than most conventional insecticides on insect pests (such as aphids) became and subsequently replacing the organophosphates and methyl-carbamates (Tomizawa *et al.*, 2007). Abd-Ella *et al.,* (2015) determined the efficiency of acetamiprid, thiamethoxam, imidacloprid, dinotefuran, and malathion on cotton aphid. The results indicated that thiamethoxam, imidacloprid, acetamiprid and dinotefuran proved to be the most effective insecticides in reducing cotton aphid population up to 21 days after treatment. Mohd, *et al.,*2020 studied the insecticidal activity and persistence effect of adding ZnO NPs to thiamethoxam and thiamethoxam alone on *Spodoptera litura* and measured their effectiveness on in the environment. Nano metal oxides can be added to pesticides to reduce their dose (Perez and Rubiales, 2009) and increase their efficacy in pest control (Liu *et al.,* 2008; Werdin Gonza´lez *et al.,* 2014; Patil *et al.,* 2016). Nano metal oxide materials as aluminum oxide (ANP), zinc oxide (ZNP), titanium oxide (TNP), and silver NPs have shown insecticidal efficacy.

This study aimed to investigate the effect of adding ZnO nanoparticles (NPs) to Thioxam 25%WG and Actara 25% WP against 4^{th} instar larvae of *S. litura* (Lepidoptera: Noctuidae) and cotton aphid, *A.gossypii* Glover (Homoptera: Aphididea).

MATERIALS AND METHODS

Chemicals:

- The tested neonicotinoids pesticides are Thioxam 25%WG and Actara 25% WP.
- The tested metal oxide is zinc oxide (ZnO) from Sigma Aldrich.

Synthesis of Nano Metal Oxide (ZnO NPs):

Zinc oxide nanoparticles were synthesized by Shoeb et al. method. The nanometal was added to the tested pesticides (neonicotinoids) water solutions by (1:5 ratio) under continuous magnetic stirring for 1h at (25°C) room temperature. Then, the mixture was heated to 50°C for 1h (Shoeb, *et al.,* 2013). The white formed precipitate was washed two times with distilled water then it dried at 50°C in the oven for 2 hour (Mohd, *et al.,*2020).

Characterization of Nano Metal Oxide (ZnO NPs):

Nicolet 550 FTIR spectrometer with 128 scans and resolution 4 cm^{-1} in the mid IR (400-4000 cm^{-1}) region. The measured sample is prepared with KBr (1-100mg), and pressed into a thin wafer and then the spectrum was recorded. TEM images were determined by a (JEOL-JEM-2100) electron microscope by dipping onto a copper grip coated with holey carbon foil and dried at an ambient temperature. UV spectroscopy used for determination band gap of the prepared ZnO NPs from the energy fixed values of the orbitals involved in electronic transitions by the appearance of new bands at definite λmax (nm) by using Perkin-Elmer S52 Spectrophotometer.

Field Experimental Design:

The cotton seeds (Egyptian cultivar Giza 90) were planted in Experimental Farm (Mansoura, Egypt), on July 27, 2020. The field area was divided into three plots, 3 ×3.5 meters. A sampling of aphid, *A. gossypii* and *S.litura* were reared weekly on April, 15, 2020 till the pest's disappearance. The *S. litura* larvae were rearing around the agricultural fields. They were set in a jar of 15 × 20 cm size, in the laboratory conditions of 28 ± 2°C temperature, 65−70% humidity and a photoperiod (10 h dark: 14 h light) in an incubator.

Mode of Application of Nano Metal Zno With Different Concentrations in Each Thiamethoxam Pesticides*:*

Each tested pesticide was dissolved in distilled water to form four concentrations, Thioxam 25%WG (20, 40, 60, and 90 mg/L) against *S. litura* (4^{th} instar larvae) and Actara 25% WP (5, 10, 30, and 50 mg/L) against cotton aphid, *A. gossypii*. Each pesticide alone was considered as the control. The cotton leaves were sprayed with different concentrations of diluted pesticide and pesticide with ZnO NPs. The replicate concentrations were four and the control.

RESULTS AND DISCUSSION

Characterization of Nano Metal Oxide (ZnO NPs):

In Figure (1), the FTIR spectrum of Nano metal ZnO shows a strong absorptions peak at 508.4 cm^{-1} expressed oxygen gaps in ZnO. The band at 3459.4 cm^{-1} may show the vibration of the (H-OH) bones groups. The broadband at 1638.1 cm^{-1} represents the expansion vibration of the carbonyl peak. The stretching band at 2945.1,2854.9 cm^{-1} expressed the (N, C, S) vibration. In Figure (2), TEM image analyzed the traced of the insecticides on Nano metal ZnO surface. There are two separated phases on the scale of 100 nm. The dark phase belongs to Nano metal ZnO. In Figure (3), the quaternization of Nano metal ZnO was confirmed by the appearance of new bands at definite λmax at 338 nm by using Perkin-Elmer S52 Spectrophotometer.

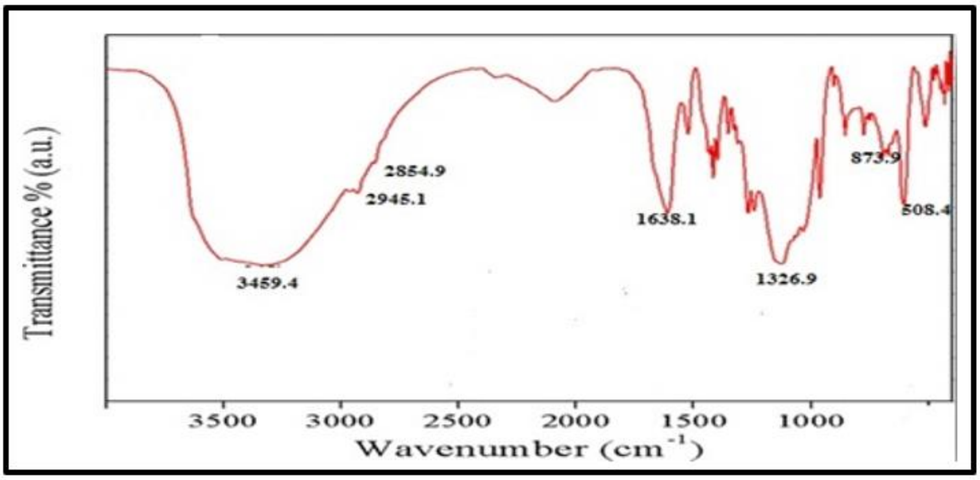

Fig. 1. FTIR spectrum pattern of Nano metal ZnO**.**

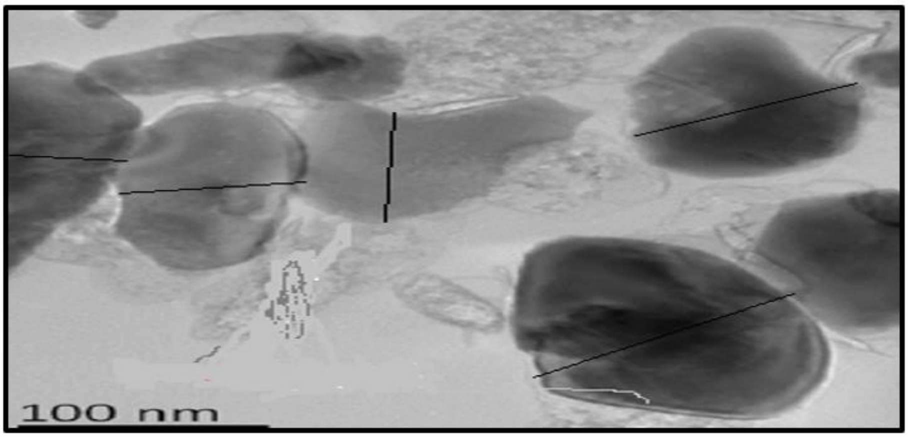

Fig. 2. TEM images of Nano metal ZnO on the scale of 100 nm.

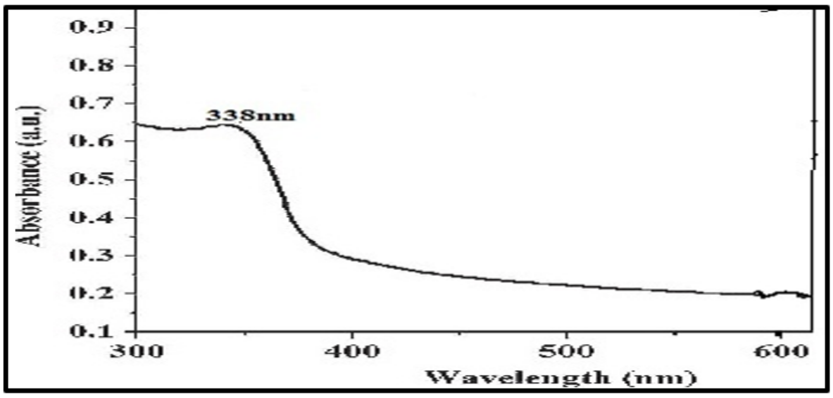

Fig. 3. UV spectroscopy of Nano metal ZnO using Perkin-Elmer S52 Spectrophotometer.

Effect of Adding Zno Nps/ Thioxam 25%WG and Zno Nps/ Actara 25% WP on Mortality Percentage and Mean Residual Effect:

1.Effect on Mortality Percentage:

Mortality percentage increases after treating Thioxam 25%WG and Actara 25% WP formulations with ZnO NPs (Table 1) in comparison with pesticides alone. Thioxam 25%WG (in the highest concentration) with ZnO NPs, we observed 28.3% increase in the mortality percentage than the groups treated with Thioxam 25%WG alone against *S. litura* (4^{th} instar larvae). Also, the highest Actara 25% WP concentration with ZnO NPs enhanced mortality 39.8% than Actara 25% WP alone against cotton aphid, *A. gossypii* Glover (Homoptera: Aphididea). These manifest differences in the mortality percentage showed the effect of adding the ZnO NPs to the two pesticides formulations. The obtained results in table (1) show that Thioxam 25%WG concentration with ZnO NPs showed the LC50's of 58.16 *mg/L*. While Actara 25% WP with ZnO NPs showed the LC50's of 14.95 mg/L. The insecticidal activity increases with adding ZnO NPs to pesticides solutions spray solutions. Nanometal oxides as zinc oxide were highly attractive toward showing modern methods for formulating the pesticides (active ingredients) in nanoscale sizes and easily delivering them, these are known as the implementation of nanotechnology in crop protection (Smith *et al.,* 2008; Yasur and Usha Rani, 2013; Benelli *et al.,* 2017).

Table 1: The effect of adding ZnO NPs to Thioxam 25%WG and Actara 25% WP on mortality percentage and insecticidal activity.

Cotton Pests	Treatment	LC_{50}(mg/L)	Mortality %
S. litura	Thioxam 25%WG	90.04	49.72
	Thioxam 25%WG/ ZnO NPs	58.16	78
A. gossypii	Actara 25% WP	28.46	50
	Actara 25% WP/ ZnO NPs	14.95	89.78

Effect on the Mean Residual Percentage:

Data presented in table (2) showed that; ZnO NPs improved the insecticidal action of Thioxam 25%WG and Actara 25% WP formulations at the complete recommended rate. Using Thioxam 25%WG in the complete recommended rate of an application gave 48.57 % initial larval mortality while the addition of ZnO NPs to Thioxam 25%WG gave a higher larval mortality 66.49%. Also, using Actara 25% WP at the complete recommended rate of an application gave 84.70% initial mortality while the addition of ZnO NPs to Actara 25% WP gave a higher mortality 93.51%. Generally, it was noticed that ZnO NPs increased the percentage of mortality at the recommended rate.

Table 2: The effect of adding ZnO NPs to Thioxam 25%WG and Actara 25% WP on the mean residual effect.

Cotton Pests	Treatments	The Residual effect					%of the mean residual effect
		Tested concentration (mg/L)	1 DAY	7 DAY	15 DAY	21 DAY	
S. litura	Thioxam 25%WG	180	84.31	56.13	38.73	15.10	48.57
	Thioxam 25%WG/ ZnO NPs		87.80	79.05	63.14	35.99	66.49
A. gossypii	Actara 25% WP	50	96.54	99.50	91.12	51.66	84.70
	Actara 25% WP/ZnO NPs		100	100	94.95	79.08	93.51

CONCLUSION:

The findings of this new study proposed that adding of ZnO nanoparticles to Thioxam 25%WG, and Actara 25% WP was used to control the population of *S.* ***litura*** (4^{th} instar larvae), and the cotton aphid, *A. gossypii* Glover. The adding of the ZnO nanoparticles composite to insecticide solutions played important roles in the insecticidal activity, and the reduction (%) of *S. litura* and cotton aphid and *A. gossypii* Glover.

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ARABIC SUMMARY

فاعلية إضافة جزيئات أكسيد الزنك النانوية علي اثنين من المبيدات (نيونيكوتينويد) ضد دوده ورق القطن ومن القطن

نيره سمير المصري
معهد بحوث وقاية النباتات –مركز البحوث الزراعية - مصر

في السنوات الأخيرة، أصبحت بقايا المبيدات تشكل خطراً على السلسلة الغذائية، والبيئة، وصحة الإنسان. تم استخدام بعض أكاسيد النانو المعدنية مع مبيدات الآفات لحل هذه المشاكل وتعزيز نشاط المبيدات الحشرية. هذه التكنولوجيا الجديدة للتمعدن الكامل لمبيدات الآفات ضرورية لتحويلها إلى أشكال غير سامة. في هذه الدراسة، درسنا التأثير التآزري للجسيمات النانوية لأكسيد الزنك مع بعض مبيدات عائله النيونيكوتينويد مثل ثيوكسام 25% وأكتارا 25% لمكافحه يرقات دوده ورق القطن (الطور الرابع) وحشره من القطن .وتم السماح لآفات القطن هذه بالتغذية على أوراق الخروع بعد تشريبها بالمبيدات الحشرية بالمعدل الموصى به ومع أضافه مركب أكسيد الزنك في حجم النانو.

أظهرت النتائج زيادة في معدل الوفيات (48.57٪، 93.51٪ زيادة في الوفيات). أدت إضافة الجسيمات النانوية لأكسيد الزنك إلى المبيدات الحشرية إلى زيادة أنشطة المبيدات الحشرية وتقليل نسبه آفات القطن المختبرة. وتم التعرف على شكل أكسيد الفلز النانوي المركب بتقنيات مختلفة مثل التحليل الطيفي (FTIR) و الميكرسكوب الألكتروني TEM) (و الأشعة فوق البنفسجية(UV) لتحديد شكل و نطاق الجسيمات النانوية لأكسيد الزنك المحضرة. تشير هذه الدراسة الحالية إلى أن أكسيد معدن الزنك يستخدم كمواد نانوية صديقة للبيئة في مجال مكافحة الآفات.