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FILED EVALUATION OF THIAMETHOXAM INSECTICIDE AND ITS MIXTURES WITH CERTAIN ADJUVANTS IN CONTROLLING LEGUMINOUS APHID, Aphis craccivora (KOCH) AND THEIR RESIDUES IN PLANTS AND SOIL

Ahmed A.A. Aioub^{*}, A.E. Omar and A.E.A. El-Sobki

Plant Prot. Dept., Fac. Agric., Zagazig Univ., Egypt

ABSTRACT

Efficacy of thiamethoxam at 1/2 and 1 recommended dose alone as well as its mixtures with certain adjuvants, *i.e.*, Sylgard 309® and Agrimax-3H® in controlling leguminous aphid, *Aphis craccivora* (Koch) were evaluated in faba bean plants under field conditions during 2013 and 2014 seasons at Zagazig District, Sharkia Governorate, Egypt. Residues of thiamethoxam alone and in mixtures with the previous adjuvants were also determined in leaves of treated plants and soil during 2014 season. Results recorded reduction percentage of aphid populations during 2013 season which were 97.47, 57.20, 97.93 and 86.44% at recommended dose of thiamethoxam (20 g a.i./fad.), 1/2 dose alone, 1/2 dose + Sylgard 309® and 1/2 dose + Agrimax-3H®, respectively. No observed effect of each adjuvant alone against aphid. However, mixing 1/2 dose of thiamethoxam with Sylgard 309® increased the efficacy of the insecticide to be the same as 1 of the recommended dose. Same trend was noticed during 2014 season. Initial residues of insecticide on leaves of plants were 2.76, 1.67, 1.60 and 1.06 mg/kg with 1 dose of thiamethoxam alone, 1/2 dose alone, 1/2 dose + Sylgard 309® and 1/2 dose + Agrimax-3H®, respectively. Whereas, the initial residues of the insecticide in soil were 0.54, 0.48, 0.40 and 0.45 mg/kg, respectively. Amount of insecticide residues were gradually degraded to reach undetectable amounts (UND) in the end of experiment.

Key words: Thiamethoxam, Sylgard 309®, Agrimax-3H®, control, Aphis craccivora, residues.

INTRODUCTION

In the European Union, the use of the insecticide thiamethoxam had been approved by the year 2004. Thiamethoxam is approved for use as soil, folair and seed treatment for the control of sucking insects and some chewing species, because of its excellent absorption and translocation in plants (Maienfisch *et al.*, 2001).

An adjuvant is any compound that is added to a insecticide formulation or a tank mix to facilitate the mixing, application or effectiveness of the insecticide whereas reducing surface tension in the spray droplet, which ensures that the formulation spreads out and cover plants with a thin film rather than beading up (Coret *et* al., 1993; Kirkwood, 1999). Sylgard 309[®] is a non ionic surfactant (organosilicone) and specifically designed to enhance the efficacy of pesticide. Sylgard 309[®] is non toxic to mammals and was found to synergize pymetrozine against insects. (Acheampong and Stark, 2004). Also Sylgard .309[®] was found to enhance either formulation of malation and chloropyrifos against *Tribolium castaneum*, reduced their LC₅₀ and the fiducial limits became more narrow due to its synergistic effect (Saad *et al.*, 2011).

Agrimax-3H[®] is excellent adhesive polymer decreases remove of pesticide deposits from plant surface. The addition of an effective and synergistic agent will lead to reduce the insecticide dose and thus reduce both the environmental stress and the resistance pressure.

^{*}Corresponding author: Tel. : +201007456970 E-mail address: ahmedaioub@rocketmail.com

Surfactants reduce surface tension at interfaces and improve spreading and contact with the leaf surface. Surface tension influences droplet formation, transport, evaporation, impact and retention. Increasing surfactant concentration generally decreases dynamic surface tension and increases droplet retention and cuticle penetration (Green and Foy, 2003).

Addition of 0.2% BB5[®], an alkyl phenoxy polyethylene ethanol formulated to thiamethoxam resulted in a considerable increase of its toxicity to Bemisia tabaci (Gennadius) nymphs. According to LC_{50} values, the combined effect was over 25-fold higher than that of thiamethoxam alone. On the other hand, no appreciable combined effect was observed when BB5 and thiamethoxam were applied together on *B. tabaci* adults. In addition, BB5 reduces and buffers the spray solution at a lower pH to a level optimum for pesticide performance (Ishaaya et al., 2005).

Describe the synthesis of two new fluorescent derivatives of thiamethoxam and compared their toxicity on aphid *Acyrthosiphon pisum* and their mode of action on insect nicotinic acetylcholine receptors expressed on the sixth abdominal ganglion (Emiliane *et al.*, 2014).

A simple and low cost method based on solid-liquid extraction with low temperature partitioning (SLE-LTP) was optimized and validated for the residue analyses of the pesticides thiamethoxam, triadimenol and deltamethrin in pineapple samples using gas chromatography with electron capture detector (GC-ECD) Elisa Helena da Costa (Morais *et al.*, 2014).

This work aimed to evaluate the effect of addition Sylgard 309[®] and Agrimax-3H[®] adjuvants on thiamethoxam efficiency under field conditions against leguminous aphid, *Aphis craccivora* (Koch) and distribution of insecticide residues in leaves of faba bean plants and soil, alone and mixing with pervious adjuvants.

MATERIALS AND METHODS

Insecticide Used

Thiamethoxam (Actara 25% WG) (3-(2chloro-thiazol-5-ylmethyl-(1,3,5) oxidiazinan – 4- ylidene- N- nitro-amine) belongs to the neonictinoids group.

Adjuvants Used

- 1. Sylgard 309[®] organosilicone non ionic surfactant.
- 2. Agrimax-3H[®] (excellent adhesive) alkylated vinyl pyrrolidone polymers which alkyl groups are grafted surfactant and polymer properties combined polymeric surfactant.

Methods Used

Field experiments were conducted in two locations, Ibraheymia (season 2013) and Ghazala (season 2014) at Zagazig District, Sharkia Governorate, Egypt.

The experimental area of faba bean received routine agricultural practices was designed as complete randomized blocks, three plots as replicates were used to each treatment and each replicate was 42 m^2 . The insecticidal treatments were applied when plants were 40 days aged, using a knapsack sprayer.

The insecticide of thiamethoxam was evaluated alone and in mixture with Sylgard 309[®] and Agrimax-3H[®] each on *A. craccivora* on faba bean plants.

Field evaluation of thiamethoxam, with the two adjuvants and their binary mixture against the leguminous aphid, A. craccivora

Experimental plots of faba bean were sprayed with insecticide at the recommended dose (20 g a.i./fad.) and the half recommended dose alone and in mixture with adjuvants. Control plots were sprayed with water only in Novmber 2013 and December 2014. Mean number of aphids was recorded before spraying and after posttreatment periods, namely, 1, 3, 7, 10 and 14 days post-treatment. Percentages reduction of aphid population was calculated according to the equation of Henderson and Tilton (1955).

Determination of thiamethoxam residues

Soil and leaves samples of faba bean plants were collected at random (during season 2014) from treated and untreated plants after one hour of application and then after 1, 3, 7, 10 and 14 days post-treatment.

Sample preparation

Leaves and soil

Leaves sample of faba bean plants and soil were collected at random from treated and untreated plants after one hour of application and then after 1, 3, 7, 10 and 14 days posttreatment. Random samples of about 3 kg each were collected from the three replicates of each treatment.

Leaves and soil samples were extracted and cleaned up using QuEChERS methodology (Lehotay *et al.*, 2005; Abd-Alrahman, 2014).

GC conditions

Residues determination of thiamethoxam were carried out at Central Laboratory of Pesticides, Dokki, Giza with gas chromatography. (HEWLETT PACKAR series 11). Separation was carried out on a colum HP 624 ($30m \times 53$ mm $\times 3\mu$ m). Flow dose of 4ml/min. The retention time of thiamethoxam was about 3.22 min. Residues were estimated by comparison of peak area of standards with that of the unknown or spiked samples run under identical conditions.

Recovery test

Thiamethoxam was added to untreated control samples at three levels (0.05, 0.1 and 1.0 mg/kg). For method validation, control and fortified, samples were analyzed under the same conditions. The average recoveries ranged from 75% and 95% in soils and leaves, respectively.

RESULTS AND DISCUSSION

Efficacy of Thiamethoxam Against Leguminous Aphid, A. craccivora

Data concerning the efficiency of thiamethoxam alone and both tested adjuvants (Sylgard 309[®] and Agrimax-3H[®]) against A craccivora attacking faba bean plants under field conditions during two seasons (2013 and 2014) are illustdosed in Tables 1 and 2.

In the first season, percent reductions were 97.47, 57.20, 97.93 and 86.44% in case of the recommended dose of thiamethoxam, half dose of thiamethoxam

with Sylgard 309[®] and half dose of thiamethoxam with Agrimax-3H[®], respectively.

In the second season, percent reductions were 98.52%, 50.14%, 96.86% and 89.25% in case of the recommended dose of thiamethoxam, half dose of thiamethoxam, half dose of thiamethoxam with Sylgard 309[®] and half dose of thiamethoxam with Agrimax-3H[®], respectively.

It could be concluded that mixtures of thiamethoxam at half recommended dose with adjuvants (Sylgard 309[®] and Agrimax-3H[®]), specially Sylgard 309[®] increased the insecticide efficiency of insecticide against the leguminous aphid, *A craccivora*.

These results agree with Susanna and Stark (2004) who evaluated the effects of a purportedly selective aphicide, pymetrozine (Fulfill), and a commonly used agricultural adjuvant, Sylgard 309, which is often applied with pymetrozine, on the life-table parameters of the aphid parasitoid Diaeretiella rapae (McIntosh). They found that pymetrozine was not toxic to D. rapae at the evaluated concentration (0.212 g a.i./ha). Instead of reducing the intrinsic dose of increase (rm) of D. rapae after adult exposure to the recommended field dose for aphid control in broccoli, rm actually increased 11%. However, Sylgard 309 alone was toxic and reduced rm by 18% compared to the control. Sazo et al. (2008) studied the effect of a siliconate coadjuvant + poliether (Break) at 50, 75 and 100 ml/ha, along with standard insecticides, chlorpyrifos and diazinon on Pseudococcus viburni mealy bug control in two commercial vineyards. These vineyards were naturally infested with mealy bugs. On the basis of the results obtained, the application of Trisiloxane in combination with polyether (Break) did not improve the effectiveness of chemical control in areas with a modedose to high infestation of P. viburni.

Residues of Thiamethoxam Alone and in Mixture with Sylgard 309 and Agrimax-3H[®] Each in Leaves of Faba Bean Plants and Soil

Residues in leaves

Data presented in Table 3 show that the residues of thiamethoxam at the high dose in leaves

 Table 1. Efficacy of thiamethoxam, two adjuvants and their mixtures against leguminous aphid,

 Aphis craccivora attacking faba bean plants in the field at Ibraheymia district, Sharkia

 Governorate (season, 2013)

| Treatments | Count before | Initial effect | Reduct | tion (%) post-ti | at indica reatment | ated days | Means of | General averages |
|----------------------------------------------------|------------------------------------------------------|-------------------|--------|---------------------|-------------------------|-----------------------------|--------------------|---------------------|
| | spraying (A) | 1 | 3 | 7 | 10 | 14 | residual effect | of Red. (%) |
| Thiamethoxam* | - | 90.65 | 96.74 | 100 | 100 | 100 | 99.185 | 97.47 |
| Thiamethoxam** | - | 45.50 | 52.87 | 58.68 | 63.65 | 65.32 | 60.13 | 57.20 |
| Sylgard 309 | - | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Agrimax-3H | - | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Thiamethoxam ** | - | 89.97 | 99.71 | 100 | 100 | 100 | 99.92 | 97.93 |
| + Sylgard 309 | | | | | | | | |
| Thiamethoxam ** | - | 46.43 | 92.25 | 95.50 | 98.05 | 100 | 96.45 | 86.44 |
| + Agrimax 3H | | | | | | | | |
| Control (water) 24.88 | | 26.60 | 28.22 | 29.65 | 30.67 | 31.35 | 29.97 | 29.29 |
| LSD _{0.05} level for treatme | ents $(T) = 1.41$ | .6 | | LS | D _{0.05} level | for periods | (P) = 1.197 | |
| * Complete dose of inse (A) No. of nymphs and a | tion $(T \times P) =$ cticide. adults per plan | = 3.167 It. | | ** | Half dose Red | of insectici . (%)= Perc | de. entage of R | eduction. |

Table 2. Efficacy of thiamethoxam, two adjuvants and their mixtures against leguminous aphid,Aphis craccivora attacking faba bean plants in the field at Ghazala region, SharkiaGovernorate (season, 2014)

| Treatments | Count | Initial | Reduction | on (%) a | nd indica | ted days | Means | General |
|--------------------------------------|-------------------|---------|-----------|----------|--------------|------------|--------------------|----------------|
| | spraying (A) | 1 | 3 | 7 | 10 | 14 | residual effect | of Red. (%) |
| Thiamethoxam* | - | 94.63 | 97.97 | 100 | 100 | 100 | 99.49 | 98.52 |
| Thiamethoxam** | - | 38.40 | 45 | 51.08 | 55.90 | 60.33 | 53.07 | 50.14 |
| Sylgard 309 | | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Agrimax -3H | - | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Thiamethoxam** | - | 84.50 | 99.83 | 100 | 100 | 100 | 99.95 | 96.86 |
| + Sylgard 309 | | | | | | | | |
| Thiamethoxam** | · · - | 58.35 | 93.68 | 96.58 | 97.66 | 100 | 96.88 | 89.25 |
| + Agrimax-3H | | | | | | | | |
| Control (water) | 23.33 | 31.72 | 34.08 | 35.31 | 36.30 | 36.37 | 35.51 | 34.75 |
| LSD _{0.05} level for treatr | nents $(T) = 0.5$ | 70 | - | LSD | 15 level for | periods (F | P) = 0.38 | |

LSD_{0.05} level for treatments (1) = 0.570LSD_{0.05} level for interaction $(T \times P) = 1.274$

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* Complete dose of insecticide.

(A) No. of nymphs and adults per plant.

**Half dose of insecticide.

Red. (%) = Percentage of Reduction

| greimax-3H® adjuvants in leaves of faba bean plants | • |
|--------------------------------------------------------------------------------|----------------------------------------------------------------------------|
| Table 3. Residues of thiamethoxam alone and in mixture with Sylgard 309® and A | and soil in the field at Ghazala region, Sharkia Governorate (season 2014) |

| Days after application | | Thiamet | hoxam | | | Fhiamet | hoxam | * * | | Thiametl + Sylga | noxam rd 309 | * | | Thiametl + Agrin | 10xam ⁴ nax-3H | * |
|---------------------------|----------|-----------|--------|------------|---------|----------------|---------|------------|--------|---------------------|-----------------|----------|---------|---------------------|------------------------------|---------|
| | Le | aves | - | ioil | Le | aves | S | oil | Le | aves | | oil | Ľ | aves | N. | oil |
| | mqq | loss (%) | mqq | loss (%) | mqq | (%) SSO | h mqq | (%) SSO | mqq | 0%) SSO | mqq | loss (%) | mqq | loss (%) | mqq | (%) SSO |
| Initial | 2.67 | 0.00 | 0.54 | 0.00 | 1.67 | 0.00 | 0.48 | 0.00 | 1.60 | 0.00 | 0.40 | 0.00 | 1.06 | 0.00 | 0.45 | 0.00 |
| 1 | 2.05 | 23.22 | 0.35 | 22.22 | 1.22 | 26.94 | 0.32 | 33.33 | 0.58 | 63.75 | 0.30 | 25 | 0.88 | 16.98 | 0.30 | 33.33 |
| | 1.43 | 46.44 | 0.30 | 44.44 | 0.56 | 66.46 | 0.25 | 47,91 | 0.50 | 68.75 | 0.21 | 47.5 | 0.52 | 50.94 | 0.22 | 51.11 |
| 1 | 0.98 | 63.29 | 0.25 | 53.70 | 0.50 | 70.05 | 0.20 | 58.33 | 0.37 | 76.87 | 0.18 | 55 | 0.41 | 61.32 | 0.15 | 66.67 |
| 10 | 0.41 | 84.64 | 0.20 | 62.96 | 0.35 | 79.04 | 0.15 | 68.75 | 0.30 | 81.25 | 0.13 | 67.5 | 0.31 | 70.75 | 0.10 | 77.78 |
| 14 | UND | 100 | UND | 100 | 0.12 | 92.81 | QND | 100 | QND | 100 | QND | 100 | UND | 87.73 | UND | 100 |
| T% in days | 4. | 07 | 2 | . 0 | ŝ | 25 | 5. | 70 | 1. | 67 | Ś | 16 | 4 | .80 | 4 | 19 |
| The recommend | ded dose | of thiame | thoyam | L ** | he half | recomm | - nehre | lose of th | iameth | mexo | | 11 = UN | ndetect | able amo | uts | |

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of faba bean plants were 2.67, 2.05, 1.43, 0.98, 0.41 and UND mg/kg after one hour, 1, 3, 7, 10 and 14 days post-treatments where the residues of tested insecticide at the low dose were 1.67, 1.22, 0.56, 0.50, 0.35 and 0.12 mg/kg, respectively.

Mixing thiamethoxam at low dose with Sylgard 309 adjuvant decreases the amount of insecticide to reach 1.60, 0.58, 0.50, 0.37, 0.30 and UND mg/kg in leaves of plants.

Same trend was noticed in case of mixing of low dose of insecticide with the second adjuvant, Agrimax-3H. Residues were 1.06, 0.88, 0.52, 0.41, 0.31 and UND mg/kg in leaves of plants after one hour, 1, 3, 7, 10 and 14 days post-treatment, respectively.

Loss percentages of pesticide residues were ranged between 0.00 - 92.81, 0.00 - 100 and 0.00 - 87.73% with low doses of insecticide alone and mixing with Sylgard 309[®] and Agrimax-3H[®], respectively.

Residues in soil

The results compiled in Table 3 show that the amounts of insecticide residues at the high dose in soil were 0.54, 0.35, 0.30, 0.25, 0.20 and UND mg/kg after one hour, 1, 3, 7, 10 and 14 days post-treatment where the residues in the case of low dose were 0.48, 0.32, 0.25, 0.20, 0.15 and UND mg/kg, respectively.

Residues of insecticides; in case of low dose when mixing with Sylgard 309; in soil were 0.40, 0.30, 0.21, 0.18, 0.13 and UND mg/kg after one hour, 1, 3, 7, 10 and 14 days post-treatment where the residues of thiamethoxam with Agrimax-3H were 0.45, 0.30, 0.22, 0.15, 0.10 and UND mg/kg, respectively.

Dissipation percentages of insecticides in soil were ranged from 0.00 to 100% with all treatments during the experimental period.

In spite of Sylgard 309 and Agrimax-3H increase the efficiency of thiamethoxam against *A craccivora* until 14 days post-treatment (Tables 1 and 2), data presented in Table 3 show that faba bean leaves and soil treated with thiamethoxam (half dose) alone were appeared to carry higher quantities of residues than leaves and soil treated with mixtures of thiamethoxam with Sylgard

309[®] or with Agrimax-3H[®]. This trend was obtained at all time intervals of post-treatment.

This finding may be due to the chemical structure of Sylgard 309[®] and Agrimax-3H[®] as a complex structure of surfactant and polymer, acting as a chelating agent binding strongly with thiamethoxam have different Rt (Rations time) values in chromatogram analysis.

Table 3 show the residues half-life $(t^{1/2})$ values of the recommended dose of thiamethoxam, the half dose of thiamethoxam, the half dose of thiamethoxam with Sylgard 309 and the half dose of thiamethoxam with Agrimax-3H in leaves of faba bean plants. Values were 4.07, 3.25, 1.67 and 4.80 days, respectively.

Data presented in Table 3 show the residues half-life $(t\frac{1}{2})$ values of the recommended dose of thiamethoxam, the half dose of thiamethoxam, the half dose of thiamethoxam with Sylgard 309 and the half dose of thiamethoxam with Agrimax-3H in soil. Values were 7.04, 5.70, 5.91 and 4.19 days, respectively.

The allowable tolerance of thiamethoxam residues in leafy vegetables by CAC/PR (2011) is 3 mg/kg. Comparing this figure with those presented in Table 3 show that leaves of faba bean plants could be used for animal consumption directly after spraying.

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التقييم الحقلى لمبيد ثاياميثوكسام ومخاليطه مع بعض المواد المضافة في مكافحة من البقوليات على نباتات الفول البلدي ومتبقياته في النبات والتربة (Koch)

١- أ.د. محمد السعيد صالح أحمد الزميتي أستاذ المبيدات – كلية الزراعة – جامعة عين شمس.
 ٢- أ.د. رفعت مصطفى محمد شريف أستاذ المبيدات المتفرغ – كلية الزراعة – جامعة الزقازيق.

المحكمون: