# Appraising the role of anti-thermal activity of beta-cyclodextrin on selective insecticides against *Thrips tabaci* (Thysanoptera: Thripidae)

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# **RESEARCH ARTICLE**

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### ABSTRACT

Evaluation studies investigated the leverage effects of beta-cyclodextrin ( $\beta$ -CD) on the long-termed toxicity of cypermethrin 25% EC, sulfoxaflor 24% SC, acetamiprid 20% SL and chlorfenapyr 24% SC against adults of Thrips tabaci laboratory strain (Thysanoptera: Thripidae) (Lindeman, 1889) from 8 up to 40 °C. Laboratory studies showed no toxicity for  $\beta$ -CD alone at all tested concentrations. Concentrations of  $\beta$ -CD at 1.25 and 2.50 gm L<sup>-1</sup> had potent leverage effects on the  $LC_{50}$ s of cypermethrin within 30–35 °C and sulfuxoflor at 40 °C.  $\beta$ -CD at 0.5 gm L<sup>-1</sup> had no leverage effect on tested insecticides. All the tested concentrations of  $\beta$ -CD decreased the toxicity of acetamiprid. Semi-field trials ( $\geq$ 28 °C) along 12 days declared that  $\beta$ -CD (equivalent to 1.25 gm L<sup>-1</sup>) increased the overall mean mortality percentages of 0.5 FRs of cypermethrin (73.08%) and sulfoxaflor (54.74%) compared to their 0.5 FRs alone of 63.70 and 44.30%, respectively in season 2020. While in season 2021, only cypermethrin at 0.5 FR +  $\beta$ -CD (74.45%) surpassed its 0.5FR (61.83%). Lethal times (LT<sub>50</sub>) values in semi-field trials showed a prolonged residual toxicity periods for the 0.5 FRs of cypermethrin +  $\beta$ -CD (8.58 days) and sulfoxaflor +  $\beta$ -CD (4.80 days) compared to their 0.5 FRs of 6.65 and 3.24 days, respectively in season, 2020. Furthermore, LT<sub>50</sub> values of the 0.5 FRs of cypermethrin +  $\beta$ -CD (9.02 days) and sulfoxaflor +  $\beta$ -CD (7.34 days) exceeded their 0.5 FRs of 6.24 and 4.07 days, respectively in 2021. Thus  $\beta$ -CD could realize leverage efficacy and longer-termed toxicity for cypermethrin and sulfoxaflor in high temperatures.

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#### **KEYWORDS**

cyclodextrin, cypermethrin, sulfoxaflor, acetamiprid, chlorfenapyr

## INTRODUCTION

*Thrips tabaci* (see Lindeman, 1889) (Thysanoptera: Thripidae) has world-wide assistance and adaptation midst in high temperature regions (Hansen, 1989; Mound and Marullo, 1996; Murai and Toda, 2002; Deligeorgidis et al., 2005; Trdan et al., 2007; Sedaratian et al., 2010; Gill et al., 2015). It is a common dangerous insect pest for various cultivated hosts, such as, onion, tobacco and cotton, besides considerations to be suspicious vector of tomato spotted wilt Orthotospovirus (Jenser and Szénási, 2004).

In the current years, the main targets of integrated pest management strategies for sustainable agriculture are directed towards increasing insecticides efficiency and decreasing their field rate dosages. These targets measures would preserve the environment from excessive exposure to pollutants of pesticides (Green and Beestman, 2007; Delogu et al., 2019).

For nearly four decades ago, many safety profiles evaluated cyclodextrins (CDs) to own safe uses with agro-chemical manufacturing that should be submitted to environmental protection (Mitchell and Armstrong, 2004; Liu et al., 2011; Gruiz et al., 2011; Betrand et al., 2011). Particularly, betacyclodextrin ( $\beta$ -CD) follows the classification of small CDs macrocyclic oligo-sugars mainly consists of seven (CD) glucose units of [(C6H10O5) + 3H2O], which is produced by enzymatic conversion of starch (Jin, 2013; Amiri and Amiri, 2017; Morin-Crini et al., 2020).  $\beta$ -CD is relatively nontoxic and biodegradable (Campos et al., 2015). Since 1970s, many adequate toxicological publications proved that CDs were non-toxic (Szejtli, 2004; Jin, 2013). Moreover, CD-containing mobile phase in aqueous solution is safer than the organic or mixed solvent due to its non-toxicity and volatility (Dall'asta et al., 2003; Jin, 2013). Native CDs are chemically, thermally, and enzymatically stable and can be easily handled and stored as a powder (Shieh and Hedges, 1996; Astray et al., 2009; Delogu et al., 2019). Complex of  $\beta$ -CD with chloramidophos enhance the thermal stability and in vitro assay on acetylcholinesterase (AChE) showed no inhibitory effects on its bioefficacy (Sliwa and Girek, 2017). Native CDs likewise  $\beta$ -CD has the ability to form inclusion complexes with a wide variety of hydrophobic or hydrophilic agricultural chemical agents in insecticides, fungicides, herbicides, rodenticides, attractants, repellents, pheromones and growth regulators (Delogu et al., 2019). The biological proprieties of the guest molecules remain unchanged as a result for non-covalent interactions with CD. Thus, CDs complexes are used as amendments that usually aim to: (i) increase the solubility, dissolution rate and wettability (ii) increase thermal and UV stability (iii) improve homogeneity, uniformity of commercial formulations and the dispersion of active ingredients (iv) enhance bioavailability within a limited applied dosage as it increases the permeability of guest molecules into the biological membranes (v) reduce the costs of packing, storage and manufacturing processes (Szejtli and Szente, 1994; Komiyama and Monflier, 2006; Hashimoto, 2006; Amiri and Amiri, 2017; Delogu et al., 2019).

Insecticides that possess a positive temperature coefficient become more toxic with the increases of temperature, whereas those have negative temperature coefficient become more toxic within cold temperature range (Khan et al., 2013a, 2013b). Pyrethroid insecticides often have negative thermal effects (Miller and Adams, 1982) as in the case of deltamethrin that exhibited a regular negative temperature coefficient against *Anopheles funestus* strains as temperature kept rising (Glunt et al., 2018). The toxicity of fenpropathrin and lambda-cyhalothrin dramatically decreased with increasing temperature from 17 to 37 °C (Boina et al., 2009). Furthermore, over 60% of chlorfenapyr residues were dissipated when exposed to direct UV rays of sun light and high temperatures of 30, 40 and 50 °C after 144, 96 and 48 h (Kandil et al., 2011). However systemic insecticides that act on nicotinic acetylcholine receptor modulators such as acetamiprid (neonicotinoids) and sulfoxaflor (sulfoximines) possessed positive temperature coefficient, an obvious reductions in their toxicity were revealed against piercing insects in low ranges of temperature (Boina et al., 2009; Mansoor et al., 2015; Jiang et al., 2019; Etheridge et al., 2019).

In this respect, our objectives were directed to evaluate how long  $\beta$ -CD additions could improve anti-thermal activity of the selective insecticides of cypermethrin, sulfoxaflor, acetamiprid and chlorfenapyr, in laboratory according to their temperature-dependent toxicity against adult stage of laboratory strains of *T. tabaci*. In addition, semi-field trials were carried out on green bean plant during two successive seasons along forecasted and monitored periods of high temperature. The semi-field trials were subjected for further confirmations on the leverage effect of  $\beta$ -CD addition on the anti-thermal activity and residual toxicity of the tested insecticides. This study aimed to regain these tested insecticides, that may characterized by their negative thermal effects, to be more applicable however the temperatures keep rising during hot seasons.

## MATERIAL AND METHODS

### Insect rearing

Samples of adult *T. tabaci* were collected from different onion fields of El-Behira region and adapted in laboratory on green bean seedling plants, *Phaseolus vulgaris* L., in plastic pots (15 cm diameter (dia.)). Rearing conditions were optimized at  $25 \pm 2$  °C, ~ 70% RH and 12 : 12 light/ dark cycle in a purposed-built incubator chamber equipped with digital thermostat, hygrometer and automatic shut-off 24 h timer according to Insecticides Resistance Action Committee (IRAC) susceptibility test methods (2009). A susceptible laboratory strain (LS) of *T. tabaci* was obtained after approximate 6 successive generations.

## Tested insecticides

Cypermethrin [Super Alpha (25% EC)] belongs to pyrethroids, was applied with the field rate 75 mL 100 L<sup>-1</sup> was obtained from El-Helb Pesticides and chemical co. Both of chlorfenapyr (Capitol (24% SC) belongs to pyrroles was applied with the field rate 60 mL 100 L<sup>-1</sup>) and acetamiprid (Outlock (20% SL) belongs to neonicotinoids was applied with the applied field rate 30 mL 100 L<sup>-1</sup>). All three chemicals were obtained from Solitaire co. for industrial and agrochemical. Sulfoxaflor (Closer (24% SC) belongs to sulfoximines, applied field rate 40 mL 100 L<sup>-1</sup>) was obtained from Dow AgroScience.

## Tested chemical compound

Beta-Cyclodextrin hydrate ( $\beta$ -CD), 99%, a thermo scientific brand with catalog number 227281000, produced by Acros co. USA was chosen to evaluate its effect on the thermal stability of the tested insecticides.



# Laboratory studies on toxicity and anti-thermal activity of beta-cyclodextrin on the tested insecticides

Laboratory tests on toxicity and thermal effect on the tested insecticides were achieved according to the IRAC susceptibility test methods (2009). Toxicity tests of the tested insecticides were accomplished against adult stage of T. tabaci at 24 h of exposure. Series of the tested insecticides concentrations were prepared. Adequate numbers of bean sections (2 cm length) with vaseline-sealed ends immersed for 30 s in each concentration while distilled water was used for control treatment. Four replicates were used for each tested concentration. Treated bean sections were settled on warm mesh surface to dry. Reared adults of T. tabaci (6th generation) were collected from green bean seedling plants using aspiration device. Each replicate had 20 individuals of adult female and male thrips and three treated bean section placed in petri dish (7 cm dia.). The  $LC_{50}$  values of the tested insecticides were calculated with their 95% confidence limits. Likewise the toxic effect of  $\beta$ -CD was also investigated at different concentrations of 0.5, 1.25 and 2.5 gm  $L^{-1}$  at 24 h of exposure. On the other hand, toxic effects for the LC<sub>50</sub>s of the tested insecticides alone and in combine to  $\beta$ -CD at different concentrations of 0.5, 1.25 and 2.5 gm  $L^{-1}$  in association with elevated temperatures were evaluated against adults of T. tabaci. The experiment was carried out under sequenced temperatures of 8 °C in refrigerator and 15, 20, 25, 30, 35, 40 °C in Shel-lab incubator (model 15450, Sheldon Manufacturing, Inc.). Four replicates were used for each temperature. Each replicate had 20 adult thrips and three treated bean section inserted into petri dish (7 cm diameter). Mortality percentages at 24 h of exposure were corrected by the formula of Abbott (1925) and submitted to the probit analysis (Finney, 1971).

## Semi-field trials on long-termed toxicity

Two field experiments were conducted at the pod stage of green bean plant, Giza 4 in seasons of 2020 and 2021 at Abees Al-Satta, El-Behira, Egypt. The experimental period during the second half of June coincided with hot temperature above 28 °C was forecasted and monitored by the Egyptian Meteorological Authority. Agriculture field of green bean plant submited to the optimal agronomic procedures (Gul and Parlak, 2017). All treatments applied in micro-plots  $(50 \text{ m}^2)$  with four replicates were submitted to a randomized complete block design. All the selected insecticides were once only applied alone at their half, complete field rates (FRs) and in mixture with  $\beta$ -CD at 8.75 gm (=1.25 gm L<sup>-1</sup> in lab. test)/7 L/micro-plot by the Knapsack sprayer equipment (CP3). Control treatment was applied by water only. Adequate samples of bean pods for each treatment were collected from the treated plots in perforated bags and transferred to the laboratory. The sampling times of bean pods from the treated field were coincided with high hot days ( $\geq 28$  °C) along 12 DATs. These samples were collected at 0, 1, 3, 5, 7, 9 and 12 days post-treatments (DATs). The samples of bean pods were prepared in the form of sections with vaseline-sealed ends in laboratory. Each treatment was replicated 4 times in petri dish (7 cm diameter). Each replicate contained three sampled bean pods sections introduced immediately to 20 adults of T. tabaci (LS) at incubator (adjusted to the outdoor average temperature in the same day) for 24 h of exposure. Mortality percentages and residual toxicity of T. tabaci (LS) after 24 h of exposure to the treated bean pods at different interval times of exposure under the field conditions of high temperatures were determined according to Abbott equation (Abbott, 1925).



### Statistical analysis

All the obtained results of laboratory studies and semi-field trials were subjected to analysis of variance (ANOVA). Means were determined for significance at 0.05 by using LSD test (SAS software, 2002).

## RESULTS

# Toxicity of the selected insecticides and beta-cyclodextrin against adults of *Thrips* tabaci (LS)

Laboratory studies were carried out to determine the  $LC_{50}$  values of the selected insecticides on adults of *T. tabaci* under the laboratory condition of  $25 \pm 2$  °C, ~ 70% RH and 12 : 12 light/dark cycle (Table 1). Cypermethrin was the most toxic among the selected insecticides with  $LC_{50}$  value of 0.004 mg L<sup>-1</sup>. Acetamiprid had the least toxicity with  $LC_{50}$  value of 0.974 mg L<sup>-1</sup>. The  $LC_{50}$  values of chlorfenapyr and sulfoxaflor were 0.021and 0.051 mg L<sup>-1</sup>, respectively (Table 1). On the other hand, Laboratory studies showed no toxic effect for  $\beta$ -CD alone at 0.5, 1.25 and 2.5 gm L<sup>-1</sup> on adults of *T. tabaci* (LS) under the same laboratory condition.

# Effects of beta-cyclodextrin on the toxicity of the selected insecticides along sequential temperature degrees in laboratory

The obtained results in Table 2 showed that the additions of  $\beta$ -CD at 2.50 gm L<sup>-1</sup> significantly increased the mortality percentages of cypermethrin at LC<sub>50</sub> (53.70–52.65%) against *T. tabaci* (LS) to exceed its LC<sub>50</sub> alone (47.75–44.29%) within temperature ranges of 30–35 °C, respectively. Likewise,  $\beta$ -CD at 2.50 gm L<sup>-1</sup> could increase the mortality percentages of sulfoxaflor at LC<sub>50</sub> (63.07%) more than its LC<sub>50</sub> alone 45.31% at 40 °C. On the other hand, additions of  $\beta$ -CD at 1.25 gm L<sup>-1</sup> could brought out significant leverage effect on the mortality percentages of cypermethrin at LC<sub>50</sub> (51.72 and 51.25%) to exceed its LC<sub>50</sub> alone (47.75 and 44.29%) at 30 and 35 °C, respectively. Likewise,  $\beta$ -CD at 1.25 gm L<sup>-1</sup> could brought out significant leverage toxicity of sulfoxaflor at LC<sub>50</sub> (55.97%) to exceed its LC<sub>50</sub> alone (45.31%) at 40 °C. On contrary,  $\beta$ -CD at 0.5 gm L<sup>-1</sup> could not realize any leverage toxic effect on the tested insecticides. In addition,  $\beta$ -CD at all tested concentrations decreased the toxicity of acetamiprid at LC<sub>50</sub> (Table 2). It is noticeable that both concentrations of  $\beta$ -CD at 1.25 and 2.50 gm L<sup>-1</sup> had identical and

*Table 1.* Toxicity of the selected insecticides and on adults of *Thrips tabaci* at 24 h of exposure in the laboratory conditions

Tested insecticides	$LC_{50} (mg L^{-1})$	Confidence limits (mg $L^{-1}$ )	Slope $\pm SE^1$	$x^2$	df	N <sup>2</sup>
Cypermethrin 25% EC	0.004	0.003-0.004	$1.70 \pm 0.16$	0.391	6	720
Chlorfenapyr 24% SC	0.021	0.018-0.025	$1.50 \pm 0.14$	0.508	5	640
Sulfoxaflor 24% SC	0.051	0.045-0.058	$2.23 \pm 0.17$	9.902	5	640
Acetamiprid 20% SL	0.974	0.711-1.335	$1.72\pm0.16$	1.116	5	640

<sup>1</sup>Standard error

<sup>2</sup>Refer to total number of adult thrips used throughout the concentrations of each treatment



Tested insecticides + $\beta$ -CD (mg L <sup>-1</sup> )		Mortality $\%^1 \pm SE^2$ at 24 h of exposure along sequential range of temperatures (°C)								
		8 °C	15 °C	20 °C	25 °C	30 °C	35 °C	40 °C	mortality $\%^3 \pm SD^4$	RS <sup>5</sup>
Cypermethrin	_6	$57.05^{bac} \pm 2.84$	$57.50^{ba} \pm 4.68$	56.88 <sup>bac</sup> ± 2.13	$50.38^{dfc} \pm 3.03$	$47.75^{\text{gf}} \pm 2.93$	$44.29^{\text{gh}} \pm 2.27$	$27.65^{j} \pm 4.44$	48.79 <sup>cf</sup> ± 10.65	-
	+ 0.5	$57.05^{bac} \pm 11.87$	$57.50^{ba} \pm 11.83$	$56.88^{bac} \pm 11.78$	$49.75^{\text{gdf}} \pm 10.20$	$47.75^{\text{gf}} \pm 10.01$	$42.90^{\text{gh}} \pm 9.65$	$29.08^{j} \pm 6.44$	$48.70^{\text{ef}} \pm 10.28$	1.00
	+ 1.25	$56.41^{bdac} \pm 2.77$	$57.50^{ba} \pm 2.28$	$57.50^{ba} \pm 1.02$	$51.02^{bdfc} \pm 2.82$	$51.72^{bdac} \pm 2.26$	$51.25^{\text{edbac}} \pm 1.80$	$34.10^{ij} \pm 3.09$	$51.36^{ed} \pm 8.16$	1.05
	+ 2.5	$57.05^{bac} \pm 1.61$	$58.13^{a} \pm 1.20$	$56.88^{bac} \pm 1.88$	$51.02^{bdfc} \pm 0.64$	$53.70^{bdac} \pm 2.75$	$52.65^{ebdac} \pm 2.54$	$36.96^{ih} \pm 3.09$	$52.34^{edc} \pm 7.26$	1.07
Acetamiprid	-	$42.95^{\text{fedg}} \pm 4.84$	$46.88^{\text{fbedc}} \pm 4.72$	$45.00^{\text{fedcg}} \pm 2.70$	$52.29^{bac} \pm 2.17$	$53.70^{ba} \pm 1.71$	$58.91^{a} \pm 2.09$	$59.17^{a} \pm 3.18$	$51.27^{ed} \pm 6.53$	-
-	+ 0.5	$42.01^{\text{fedg}} \pm 2.47$	$44.38^{\text{fedcg}} \pm 3.44$	$45.63^{\text{fedcg}} \pm 2.13$	$51.53^{bac} \pm 1.39$	$53.63^{ba} \pm 1.29$	$47.69^{\text{bedc}} \pm 1.30$	$47.44^{bedc} \pm 0.82$	$47.47^{f} \pm 4.03$	0.93
	+ 1.25	$42.95^{\text{fedg}} \pm 9.14$	$46.88^{\text{fbedc}} \pm 9.73$	$46.25^{\text{fbedc}} \pm 10.60$	$49.75^{bdc} \pm 11.17$	$52.38^{bac} \pm 11.52$	$42.90^{\text{fedg}} \pm 9.26$	$44.84^{\text{fedcg}} \pm 9.71$	$46.56^{f} \pm 3.50$	0.91
	+ 2.5	$38.61^{g} \pm 1.88$	$41.25^{\text{feg}} \pm 1.61$	$41.88^{\text{fedg}} \pm 3.59$	$49.21^{bedc} \pm 4.14$	$45.93f^{bedg} \pm 1.93$	$40.16^{g} \pm 1.11$	$40.17^{\text{fg}} \pm 3.81$	$42.46^{g} \pm 3.76$	0.83
Sulfoxaflor	-	$53.61^{bdac} \pm 4.08$	$56.25^{bac} \pm 5.64$	$58.13^{bac} \pm 4.83$	$57.91^{bac} \pm 6.71$	$52.28^{bdc} \pm 7.71$	$51.09^{dc} \pm 4.00$	$45.31^{d} \pm 3.55$	$53.51^{bdc} \pm 2.87$	-
	+ 0.5	$53.25^{bdac} \pm 4.45$	$55.63^{bdac} \pm 2.58$	$58.75^{bac} \pm 3.31$	$58.78^{bac} \pm 1.39$	$55.10^{bdac} \pm 1.36$	$52.74^{bdac} \pm 1.85$	$54.57^{bdac} \pm 1.18$	$55.55^{bac} \pm 2.41$	1.02
	+ 1.25	$53.61^{bdac} \pm 4.08$	$56.88^{bac} \pm 1.20$	57.50 <sup>bac</sup> ± 3.39	$59.18^{bac} \pm 4.03$	$56.99^{bac} \pm 1.90$	$53.80^{bdac} \pm 4.84$	$55.97^{bac} \pm 2.96$	$56.28^{ba} \pm 2.01$	1.03
	+ 2.5	$56.19^{bac} \pm 3.79$	$57.50^{bac} \pm 1.77$	$57.50^{bac} \pm 1.77$	$61.73^{ba} \pm 4.54$	$59.01^{bac} \pm 2.54$	$56.52^{bac} \pm 2.48$	$63.07^{a} \pm 1.16$	$58.79^{a} \pm 2.65$	1.08
Chlorfenapyr	-	$45.35^{ef} \pm 1.72$	$44.86^{ef} \pm 5.04$	$44.18^{ef} \pm 3.73$	$52.37^{ebdac} \pm 3.94$	$51.98^{ebdcf} \pm 5.59$	$52.50^{ebdc} \pm 2.18$	$54.38^{bdac} \pm 4.73$	$49.37^{ef} \pm 4.36$	-
	+ 0.5	44.89 <sup>ef</sup> ± 9.49	$44.29^{ef} \pm 8.81$	$43.18^{f} \pm 8.81$	$52.81^{ebdac} \pm 8.02$	$54.25^{bdac} \pm 9.25$	$54.38^{bdac} \pm 7.95$	$56.25^{ba} \pm 8.39$	$50.01^{\text{edf}} \pm 5.62$	1.01
	+ 1.25	$47.58^{ebdcf} \pm 4.26$	$47.69^{ebdcf} \pm 3.61$	$46.73^{\text{edcf}} \pm 1.20$	$54.08^{bdac} \pm 3.29$	$55.54^{bac} \pm 2.80$	$56.25^{ba} \pm 1.71$	$58.13^{a} \pm 5.36$	$52.29^{edc} \pm 4.79$	1.06
	+ 2.5	$47.58^{ebdcf} \pm 3.72$	$47.69^{ebdcf} \pm 4.13$	$46.73^{\text{edcf}} \pm 1.20$	$54.08^{bdac} \pm 3.29$	$55.54^{bac} \pm 1.69$	$56.25^{ba} \pm 2.83$	$58.13^{a} \pm 5.36$	$52.29^{edc} \pm 4.79$	1.06

*Table 2.* Mortality percentages of the selected insecticides alone and in combine with  $\beta$ -CD at 24 h of exposure to LC<sub>50</sub>s against adults of *T. tabaci* (LS) along sequential range of temperatures

 $^{1}$ Mortality percentages with the same letter are not significantly different according to the LSD<sub>0.05</sub> for the interactions between each tested insecticide separately and temperature degrees.

<sup>2</sup>Mortality percentages displayed with error bars using standard error.

<sup>3</sup>Overall mean mortality percentages with the same letter are not significantly different according to the LSD<sub>0.05</sub>.

<sup>4</sup>Standard deviation.

<sup>5</sup>RS: relative superiority that based on ratios of mortality percentages of insecticide  $+\beta$ -CD/insecticide alone, where values >1 fold considered as relative superiority.

<sup>6</sup> -: the tested insecticide alone.

simultaneous potent effects on the tested insecticides. Thus, the  $\beta$ -CD at 1.25 gm L<sup>-1</sup> might be considered to be an initial concentration that gave a regular leverage effect on the toxicity of cypermethrin and sulfoxaflor. This concentration could be selected later as an appropriate concentration in the semi-field trials.

Regarding to the results of the overall mean of mortality percentages in Table 2, Only addition of  $\beta$ -CD at 2.5 gm L<sup>-1</sup> had the highest significant leverage on overall mean mortality percentage of sulfoxaflor at LC<sub>50</sub> (58.79%) compared to its LC<sub>50</sub> alone (53.51%). All the tested concentrations of  $\beta$ -CD had no significant differences between the on the overall mean of mortality percentages of each tested insecticide of cypermethrin, sulfoxaflor and chlorfenapyr. Contrariwise, all tested concentrations of  $\beta$ -CD had negative adverse effects on overall mean of mortality percentages of acetamiprid.

Given the data based on the ratios between the overall mean mortality percentages of the insecticide +  $\beta$ -CD to the same insecticide alone, the only values exceeded 1 fold were considered to possess relative superiority. Thus, relative superiority for sulfoxaflor (1.08 folds) > cypermethrin (1.07 folds) > chlorfenapyr (1.06 folds) whenever mixed with  $\beta$ -CD at 2.5 gm L<sup>-1</sup>. On the other hand, relative superiority for cypermethrin (1.05 folds) > chlorfenapyr (1.06 folds) > sulfoxaflor (1.03 folds) whenever mixed with  $\beta$ -CD at 1.25 gm L<sup>-1</sup>. On the other hand, the addition of  $\beta$ -CD at 0.5 gm L<sup>-1</sup> only realized relative superiority for sulfoxaflor (1.02 folds) and chlorfenapyr (1.01 folds).

#### Semi-field assessment of beta-cyclodextrin on the toxicity of the selected insecticides

The obtained results of semi-field trials in season 2020 and 2021 were carried out only on the tested insecticides that have shown a positive leverage effect and relative superiority whenever mixed  $\beta$ -CD in the laboratory trials. Thence, the semi-field trials in season 2020 and 2021 were carried out on the toxic effect of cypermethrin, sulfoxaflor and chlorfenapyr at different field rates alone and in combine with  $\beta$ -CD against adults of *T. tabaci* (LS) exposed after 24 h of treatment (Tables 3 and 4). The suitable concentration of  $\beta$ -CD for field trial spray was deducted from the data in Table 2 to be equivalent to 1.25 gm L<sup>-1</sup>. This concentration was chosen according to its regular leverage effect on the toxicity of cypermethrin and sulfoxaflor besides fulfillment to the relative superiority term (values >1 fold) for chlorfenapyr. Therefore, the suitable spray concentration of  $\beta$ -CD for semi-field applications was 8.75 gm =1.25 gm L<sup>-1</sup> in lab./7 L/micro-plot (50 m<sup>2</sup>) in combine with the 0.5 FRs of cypermethrin, sulfoxaflor and chlorfenapyr.

The results of the leverage toxic effect of  $\beta$ -CD in season 2020 revealed through the mortality percentages against *T. tabaci* (LS) in cypermethrin at 0.5 FRs+  $\beta$ -CD (55.88 and 54.55%) that surpassed its 0.5 FRs (41.18 and 18.18%) at 7 and 9 DATs, respectively. In addition, leverage toxic effects of sulfoxaflor at 0.5 FRs +  $\beta$ -CD (97.22, 66.67, 30.30 and 15.15%) surpassed its 0.5 FRs (86.11, 52.78, 12.12 and 5.30%) against *T. tabaci* (LS) within the periods of 1, 3, 9 and 12 DATs, respectively (Table 3). Finally, the results of overall mean mortality percentages in season 2020 declared that 0.5 FRs +  $\beta$ -CD of cypermethrin (73.08%) and sulfoxaflor (54.74%) significantly surpassed their 0.5 FRs of 63.70 and 44.30%, respectively. No significant differences for overall mean mortality percentages between 0.5 FR +  $\beta$ -CD and 0.5 FR in chlorfenapyr treatment (Table 3).

Comparably, the results in season 2021 showed that cypermethrin and chlorfenapyr had no significant differences between mortality percentages of their 0.5 FR in compare to 0.5 FR +  $\beta$ -CD



Table 3. Mortality percentages of adult Thrips tabaci (LS) in semi-field trials at 24 h of exposure to the selected insecticides at different field rates alor
and in combine with $\beta$ -CD in season 2020

			Mean of mortality % $\pm$ SD <sup>1</sup> at 24 h of exposure along 12 DATs								
Treatments		0 DAT	1 DAT	3 DAT	5 DAT	7 DAT	9 DAT	12 DAT	$\pm SD^1$	${\rm LT_{50}}^2$	
Cypermethrin	FR	$97.22^{ba} \pm 5.56$	$97.22^{ba} \pm 5.56$	$97.22^{ba} \pm 5.56$	$91.18^{bac} \pm 5.88$	85.29 <sup>c</sup> ± 11.26	$66.67^{d} \pm 24.99$	$33.33^{f} \pm 7.00$	$81.16^{a} \pm 23.77$	11.82	
71	0.5 FR	$91.67^{bac} \pm 5.56$	$97.22^{ba} \pm 5.56$	$97.22^{ba} \pm 5.56$	$85.29^{\circ} \pm 5.88$	$41.18^{f} \pm 9.61$	$18.18^{g} \pm 6.06$	$15.15^{g} \pm 0.00$	$63.70^{dc} \pm 37.49$	6.65	
	$0.5 \text{ FR} + \beta \text{CD}$	$100.00^{a} \pm 0.00$	$97.22^{ba} \pm 5.56$	$94.44^{bac} \pm 6.42$	$88.24^{bc} \pm 0.00$	$55.88^{ed} \pm 5.88$	$54.55^{f} \pm 6.06$	$21.21^{g} \pm 7.00$	$73.08^{b} \pm 29.79$	8.58	
Sulfoxaflor	FR	$88.89^{bac} \pm 9.07$	$91.67^{ba} \pm 10.64$	$80.56^{\circ} \pm 5.56$	$67.65^{d} \pm 5.88$	$70.59^{d} \pm 6.79$	$39.39^{ m gf} \pm 0.00$	$21.21^{ji} \pm 7.00$	$65.71^{\circ} \pm 26.93$	8.58	
	0.5 FR	$86.11^{bc} \pm 5.56$	$86.11^{bc} \pm 5.56$	$52.78^{\circ} \pm 5.56$	$38.24^{\text{gfh}} \pm 5.88$	29.41j <sup>h</sup> ± 9.61	$12.12^{j} \pm 6.06$	$5.30^{k} \pm 6.72$	$44.30^{f} \pm 32.62$	3.24	
	$0.5 \text{ FR} + \beta \text{CD}$	$94.44^{ba} \pm 11.11$	$97.22^{a} \pm 5.56$	$66.67^{d} \pm 0.00$	$41.18^{f} \pm 0.00$	$38.24^{\text{gfh}} \pm 5.88$	$30.30^{gih} \pm 6.06$	$15.15^{j} \pm 0.00$	$54.74^{e} \pm 32.33$	4.80	
Chlorfenapyr	FR	$100.00^{a} \pm 0.00$	$97.22^{ba} \pm 5.56$	$97.22^{ba} \pm 5.56$	$88.24^{\circ} \pm 0.00$	$85.29^{dc} \pm 5.88$	78.79 <sup>ed</sup> ± 6.06	36.36 <sup>hi</sup> ± 11.61	$83.30^{a} \pm 22.05$	5.97	
	0.5 FR	$91.67b^{ac} \pm 5.56$	$97.22^{ba} \pm 5.56$	69.44f ± 5.56	$44.12^{hg} \pm 5.88$	$50.00^{g} \pm 11.26$	$33.33^{i} \pm 7.00$	$15.15^{j} \pm 0.00$	$57.28^{\circ} \pm 30.28$	5.32	
	$0.5 \text{ FR} + \beta \text{CD}$	$88.89^{bc} \pm 0.00$	$97.22^{ba} \pm 5.56$	$72.22^{\text{ef}} \pm 6.42$	$47.06^{g} \pm 6.79$	$47.06^{g} \pm 6.79$	$33.33^{i} \pm 7.00$	$18.18^{j} \pm 6.06$	$57.71^{de} \pm 29.23$	5.49	

<sup>1</sup>Standard deviation.

 $^{2}LT_{50}$ : lethal time queried to kill 50% of insect individuals calculated within intervals of 1, 3, 5, 7, 9 and 12 DATs.

• Mortality percentages, for each insecticide at FR, 0.5FR and 0.5FR+  $\beta$ -CD, with the same letter are not significantly different according to the LSD<sub>0.05</sub> for the interactions between treatments and DATs.

• Means of overall mortality percentages with the same letter are not significantly different according to the LSD<sub>0.05</sub> between treatments.

against *T. tabaci* (LS) along the 12 DATs (Table 4). The leverage toxic effect of sulfoxaflor at 0.5 FR +  $\beta$ -CD (67.65%) exceeded its 0.5 FR (52.94%) at 3 DAT (Table 4). The obtained results in season 2021 declared that the overall mortality percentage of 0.5 FR +  $\beta$ -CD for cypermethrin (74.45%) could significantly surpassed its 0.5FR (61.83%). No significant differences for overall mean mortality percentages between 0.5 FR +  $\beta$ -CD and 0.5 FR in sulfoxaflor and chlorfenapyr (Table 4).

Eventually, the obtained data of  $LT_{50}$ s in Tables 3 and 4 expressed the certain times at which the residual efficacy of each treatment could achieve 50% toxic effects against adults of *T. tabaci*.  $LT_{50}$ s in season 2020 for cypermethrin, sulfoxaflor and chlorfenapyr at 0.5 FRs +  $\beta$ -CD were 8.58, 4.80 and 5.49 days, respectively more than their 0.5FRs, which were 6.65, 3.24 and 5.32 days, respectively. On the other hand,  $LT_{50}$ s in season 2021 for cypermethrin and sulfoxaflor at 0.5 FRs +  $\beta$ -CD were 9.02 and 7.34 days, respectively exceeded their 0.5FRs, which were 6.24 and 4.07 days, respectively. Oppositely,  $LT_{50}$  of chlorfenapyr at 0.5 FR +  $\beta$ -CD (5.05 days) was less than its 0.5FR (5.17 days).  $LT_{50}$ s in the two seasons for all selected insecticides at 0.5 FRs +  $\beta$ -CD were less than their FRs along all the DATs.

## DISCUSSION

The efficiency of insecticides used for controlling agro-pests encountering obstacles regarding thermal effect within wide range of high temperatures during hot seasons as well as in hot temperature zones. Many studies were carried out to enhance the efficacy on insecticides based on temperature-dependent toxicity correlations (Boina et al., 2009; Teja et al., 2018; Glunt et al., 2018). Therefore, the outlook of this study refers to evaluate the addition of  $\beta$ -CD to the tested insecticides having a varied temperature-dependent toxicity to improve their efficacy in controlling *T. tabaci* within wide range of high temperatures. This claim are supported by the investigations carried out on CDs usages, which had been grown since 1980 in different disciplines especially in agro-chemical industries. The broad usages of CDs are a reflection to its ability to form inclusion complexes with various molecules through host-guest interactions. Moreover, it improves the bioavailability of chemical compounds by increasing their solubility, dissolution, permeability and thermal stability (Menges and Armstrong, 1991; Szente and Szejtli, 1999; Mitchell and Armstrong, 2004; Morillo, 2006; Kayaci et al., 2013; Sliwa and Girek, 2017; Delogu et al., 2019; Morin-Crini et al., 2020).

Data of toxicity studies in this research showed no toxic effect for  $\beta$ -CD alone in the concentration limits of 0.5, 1.25 and 2.50 gm L<sup>-1</sup> on adults of *T. tabaci* (LS). These data may be demonstrated by the facts about the safety and toxicity profiles of CDs that assessed it to be natural, relatively nontoxic, depending upon the dose and administration routes in drug-based compounds researches. Of all the CD derivatives available, hydroxy propyl - $\beta$ -CD is the safest, as it does not penetrate the membranes of cell organs (Fromming and Szejtli, 1994; Loftsson, 2002; Amiri and Amiri, 2017). In addition, CDs are co-friendly to environment and may be used in environmental protection, as it efficiently removes polar organic pollutants from aqueous environments by selective adsorption (Liu et al., 2011; Gruiz et al., 2011; Betrand et al., 2011; Sliwa and Girek, 2017).

The data of our research for each cypermethrin and sulfoxaflor alone at  $LC_{50}$  in laboratory tests showed negative toxic effects against adults of thrips in the temperature range above 30 °C.



Table 4. Mortality percentages of adult Thrips tabaci (LS) in semi-field trials at 24 h of exposure to the selected insecticides at different field rates	s alone
and in combine with $\beta$ -CD in season 2021	

			Mean of mortality % $\pm$ SD <sup>1</sup> at 24 h of exposure along 12 DATs								
Treatments		0 DAT	1 DAT	3 DAT	5 DAT	7 DAT	9 DAT	12 DAT	$\pm SD^1$	${\rm LT_{50}}^2$	
Cypermethrin	FR	$97.14^{a} \pm 5.71$	$96.97^{a} \pm 6.06$	$97.06^{a} \pm 5.88$	93.94 <sup>ba</sup> ± 7.00	$86.49^{ba} \pm 5.41$	65.63 <sup>ed</sup> ± 11.97	$33.33^{\text{gf}} \pm 7.00$	$81.51^{a} \pm 24.08$	11.95	
71	0.5 FR	$94.29^{a} \pm 6.60$	$96.97^{a} \pm 6.06$	$97.06^{a} \pm 5.88$	$78.79^{bdc} \pm 11.61$	$37.84^{ed} \pm 5.41$	$18.75^{ef} \pm 7.22$	$9.09^{h} \pm 7.00$	$61.83^{dc} \pm 38.79$	6.24	
	$0.5 \text{ FR} + \beta \text{CD}$	$97.14^{a} \pm 5.71$	$96.97^{a} \pm 6.06$	$97.06^{a} \pm 5.88$	$84.85^{bac} \pm 6.06$	$67.57^{edc} \pm 22.20$	$59.38^{\circ} \pm 23.27$	18.18 <sup>gh</sup> ± 12.99	$74.45^{b} \pm 29.10$	9.02	
Sulfoxaflor	FR	$91.43^{a} \pm 10.94$	$93.94^{a} \pm 7.00$	$76.47^{bc} \pm 16.64$	$69.70^{\circ} \pm 7.00$	$67.57^{\circ} \pm 8.83$	$34.38^{ef} \pm 6.25$	15.15 <sup>gh</sup> ± 9.90	$64.09^{\circ} \pm 29.19$	8.81	
	0.5 FR	$88.57^{a} \pm 9.33$	$96.97^{a} \pm 6.06$	$52.94^{d} \pm 9.61$	$36.36^{ef} \pm 6.06$	$40.54^{e} \pm 6.24$	$18.75^{\text{gh}} \pm 7.22$	$12.12^{h} \pm 11.61$	$49.47^{e} \pm 32.63$	4.07	
	$0.5 \text{ FR} + \beta \text{CD}$	$94.29^{a} \pm 6.60$	$96.97^{a} \pm 6.06$	$67.65^{\circ} \pm 5.88$	$42.42^{ed} \pm 6.06$	$45.95^{ed} \pm 8.83$	$25.00^{\text{gf}} \pm 10.21$	$18.18^{\text{gh}} \pm 6.06$	$55.78^{de} \pm 31.51$	7.34	
Chlorfenapyr	FR	$97.14^{a} \pm 5.71$	$96.97^{a} \pm 6.06$	$88.24^{a} \pm 9.61$	$84.85^{ba} \pm 6.06$	$89.19^{a} \pm 8.83$	$71.88^{bc} \pm 15.73$	$33.33^{h} \pm 15.65$	$80.23^{ba} \pm 22.36$	13.46	
	0.5 FR	$85.71^{a} \pm 9.33$	$93.94^{a} \pm 6.06$	$61.76^{dce} \pm 9.61$	$42.42^{\text{gfh}} \pm 9.90$	$51.35^{dfe} \pm 5.41$	$34.38^{h} \pm 6.25$	$18.18^{i} \pm 9.90$	$55.39^{de} \pm 27.25$	5.05	
	$0.5 \text{ FR} + \beta \text{CD}$	$88.57^{a} \pm 5.71$	$96.97^{a} \pm 7.00$	$64.71^{dc} \pm 5.88$	39.39 <sup>gh</sup> ± 18.18	$54.05^{dfe} \pm 6.24$	$34.38^{h} \pm 6.25$	$15.15^{i} \pm 6.06$	$56.17^{d} \pm 29.53$	5.17	

<sup>1</sup>Standard deviation.

 $^{2}LT_{50}$ : lethal time queried to kill 50% of insect individuals calculated within intervals of 1, 3, 5, 7, 9 and 12 DATs.

•Mortality percentages, for each insecticide at FR, 0.5FR and 0.5FR +  $\beta$ -CD, with the same letter are not significantly different according to the LSD<sub>0.05</sub> for the interactions between treatments and DATs.

• Means of overall mortality percentages with the same letter are not significantly different according to the LSD<sub>0.05</sub> between treatments.

These data meet the findings of toxicity declinations of zeta-cypermethrin by 1.80 fold in against *Diaphorina citri* (Hemipetra, Lividae) (Kuwayama, 1908) over the 17–27 °C range, and further declination by 5.33 fold occurred along the 27–37 °C range. Thus, zeta-cypermethrin could be most effective during the cooler winter seasons in Florida region (Boina et al., 2009). On the other hand, our data of acetamiprid and chlorfenapyr alone at LC<sub>50</sub> have positive toxic effects within the temperature range above 25 °C. These data were symmetrical to previous research studies confirmed that chlorfenapyr had strong positive temperature–dependent toxic effects above 30 °C against diamondback moth, *Plutella xylostella* (Linnaeus, 1758) (Lepidoptera, Plutellidae) (Teja et al., 2018) as well as against the susceptible KISUMU strain of *Anophilus gambiae* at temperature range of 21–29 °C (Oxborough et al., 2015; Glunt et al., 2018). acetamiprid showed also a positive temperature dependent toxicity correlations against *D. citri* adults over the temperature range of 17–27 °C and 27–37 °C, respectively (Boina et al., 2009).

The obtained data in laboratory tests showed that  $\beta$ -CD at 1.25 and 2.50 gm L<sup>-1</sup> in high range  $\geq$ 30 °C consistently had equalized effects in increasing the toxicity of cypermethrin, sulfoxaflor and chlorfenapyr. In the same mode the results of overall mortality percentages in semi-field trials which carried out along 12 successive days of high temperature ( $\geq 28$  °C) declared that 0.5 FRs +  $\beta$ -CD of cypermethrin could regularly alter their 0.5 FRs along the two successive seasons as well as sulfoxaflor in season 2020. Data of  $LT_{50}$ s in the two seasons for cypermethrin and sulfoxaflor at 0.5 FRs +  $\beta$ -CD regularly exceeded their 0.5FRs as well as chlorfenapyr in season 2021. These findings could be elucidated by some researches and reviews throughout two main pivots. Firstly, the role of CDs in improving thermal stability and controlled time-released activity for incorporated insecticides. Inclusion complex formation entraps guest molecules in CD cavity, which limits its diffusion from CD, stabilizes and delays its mass decomposition, protects it from reacting with active agents and thus improving stability (Arias et al., 2000; Arima et al., 2001; Amiri and Rahimi, 2014, 2015). This was also evidently verified by differential scanning calorimetry thermo-gram for imidacloprid that exhibits a sharp endothermic peak nearby 133 °C (melting temperature) and another large exothermic peak centered at 260 °C referring to its decomposition. Obvious shifting had been occurred in thermo-gram of imidacloprid- $\beta$ -CD inclusion complex that hardly owned inconspicuous peak of melting temperature that reached up to 300 °C (decomposition of  $\beta$ -CD). In accordance, imidacloprid inclusion complex with  $\beta$ -CD into polypropylene and filament yarns improved thermal stability and prolonged release activity compared to the neat imidacloprid (Giordano et al., 2001; Turan et al., 2017). Moreover, inclusion complexes of eugenol in polyvinyl alcohol/  $\beta$ -CD nanofibers have much slower release and thermal evaporation shifted to higher temperature when compared to pure eugenol (Kayaci et al., 2013). Secondly, justifications for maintaining the residual efficacy of insecticides/CD complexes could be deducted via their acquired selective toxicity by contact absorption that targeted the herbivorous pests except nontarget insects. For instance, in vitro assay by acetyl choline esterase inhibition and acute aquatic toxicity accomplished on chloramidophos insecticide complex with  $\beta$ -CD had great improvement on its thermal stability without adverse effects on its bio-efficacy compared to neat chloramidophos (Jin, 2013; Li and Jin, 2013). In contrary, the data of our research in laboratory tests on acetamiprid at  $LC_{50} + \beta$ -CD against adults thrips showed obvious decline in the toxicity in compare to acetamiprid alone along temperature range from 25 up to 40 °C. So far, no reviews could investigate or justify phenomenon of negative result with acetamiprid.



### Conclusion

 $\beta$ -CD realized a regular increases in toxicity of sulfoxaflor and cypermethrin against adults of *T. tabaci* (LS) within high temperatures exceeded 30 °C in laboratory tests. Furthermore in semi-field trails, addition of  $\beta$ -CD exhibited regular increases of long-termed toxicity of sulfoxaflor and cypermethrin at  $\geq 28$  °C. The highest LT<sub>50</sub> in  $\beta$ -CD + 0.5 FRs of cypermethrin (9.02 days) and sulfoxaflor (7.34 days) exceeded their 0.5 FRs in the two successive seasons. Oppositely,  $\beta$ -CD had negative effects on acetamiprid toxicity. However  $\beta$ -CD had an absence role on the toxicity of chlorfenapyr in laboratory studies, it could attain the superiority relative term. Moreover, no role for  $\beta$ -CD could be realized on chlorfenapyr during semi-field trails in the two seasons. Eventually, there are an urgent need for more prospect studies that may develop the usage of CDs complex inclusion with insecticides formulations to maintain their anti-thermal activity in hot seasons and high temperature regions particularly in field conditions.

Finally, more prospect studies are needed in future to investigate whether pesticides with appropriate formulations with  $\beta$ -CD would have a toxic impact on the predators of thrips species under field conditions.

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