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Toxicity and Biochemical Impacts of Garlic and Camphor Essential Oils against *Aphis gossypii* and *Gynaikothrips ficorum* Compared to Chitosan and Mineral Oil

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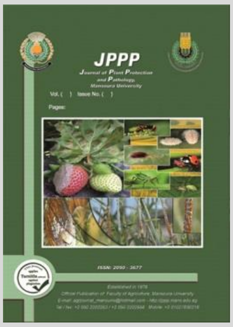

ABSTRACT

Aphis gossypii (Glover) (Hemiptera: Aphididae) and *Gynaikothrips ficorum* Marchal (Thysanoptera: Phlaeothripidae) are important economic insect pests in Egypt. They cause economic losses to many crops. Plant essential oils are less harmful to the environment, hypotoxic to mammals, and lower in cost in comparison with synthetic insecticides, and they have biological activity. This study aimed to investigate the efficiency of garlic and camphor essential oils, mineral oils, and chitosan against *A. gossypii* and *G. ficorum* as well as, to study their effects on the activity of some detoxifying enzymes such as GPT, GOT, GS-T, ALP and ACP. The obtained results indicated that garlic and camphor essential oils were more toxic against tested insects, *A. gossypii* and *G. ficorum*, compared with mineral oil and chitosan. According to the toxicity index of LC_{50} and LC_{90}, the treatments were ranked first as camphor oil then garlic oil, mineral oil and chitosan in case of *A. gossypii*, while they ranked garlic oil then camphor oil, mineral oil and chitosan in case of *G. ficorum.* In general, all tested treatments caused high mortality percentages, ranging from 70 to 97% against *A. gossypii* and 68 to 93% against *G. ficorum* at 10000 ppm after 48 hours of treatment. In addition, most of the estimated enzymes were inhibited by all treatments except some. Thus, the essential oils of garlic and camphor are gaining acceptance as a strategy for integrated pest management due to their safety and their high toxicity against *A. gossypii* and *G. ficorum*.

Keywords: Cotton aphid, Ficus thrips, Natural insecticides, Detoxifying enzymes

INTRODUCTION

Aphids are cosmopolitan pests that occur in different temperate regions of the world. These pests are direct plant sap-sucking and can cause serious problems on many crops even at low densities because of transmitting plant viruses (Munster, 2020). The cotton aphid, *Aphis gossypii* is a polyphagous pest attacking more than 92 plant families including field, vegetable, fruit, and ornamental crops (Somar *et al*., 2019). *Aphis gossypii* infests leaves, stems and fruits and causes direct significant economic damage in addition to its major indirect damage, which is deposition of sooty mold and secretion of honeydews (Srivastava and Shukla, 2021). Sooty mold hinders the plant respiration and decreases the photosynthetic rate, causing crop weakening (Fontes *et al.*, 2006). Unfortunately, *A. gossypii* exhibited high levels of resistance to many types of insecticides in the field (Chen *et al*., 2017).

The Ficus thrips or the Cuban laurel thrips (Paine, 1992), *Gynaikothrips ficorum* Marchal (Thysanoptera: Phlaeothripidae) is a monophagous insect pest recorded widely in all regions where its host plant, *Ficus microcarpa* (Marchal) (Moraceae), has been cultivated in urban and interior landscape plant species across all continents (Tavares *et al*., 2013) except Antarctica (Mound, 2009). It is a major pest of ficus trees, which preferred feeding on tender young leaves and induces leaf-fold galls (Tree and Walter, 2009). *Gynaikothrips ficorum* does not kill the infested trees, but the galls reduce the photosynthetic activity of the plants and the ornamental value and quality of the plants are reduced markedly due to discolored and curled leaves (Dang *et al*., 2021). In addition, adults of *G. ficorum* can be annoying and biting people, causing skin irritation (Piu *et al*., 1992).

The high and injudicious uses of synthetic chemical insecticides enhanced the resurgence and resistance of many pests. In addition, the high cost of producing insecticides and legal restrictions have focused on using alternative approaches to control pests (Dubey and Sharma, 2022). To decrease the harmful impacts of the chemical insecticides on the environment and human health, there is insistent need for new effective substrates in the programs of integrated pest management (IPM) (Rodríguez-González *et al*., 2019). In addition, to cope with sustainable agriculture, attention is directed toward expansion in organic farming, which is the production extension system that completely or largely avoids the use of artificial chemicals such as pesticides, synthetic fertilizers and growth regulators (Behera *et al*., 2012). Therefore, the secondary metabolites (such as phenols, flavonoids, quinones, terpenoids, alkaloids, tannins and etc.), which produced by plants to protect themselves against herbivorous and microbial attacks could be extracted and used for control many plant pests (Liu *et al*., 2021). So, botanical extracts which are less harmful to the environment, hypotoxic to mammals, and lower in cost in comparison with insecticides (Dougoud *et al.*, 2019) are commonly used for pest control due to their adverse effects on different life stages of insect pests (Ahmed *et al*., 2020). These extracts can reduce the viability of insect eggs,

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slowing the growth of insect and can cause insect mortality (Bedini *et al*., 2020). Moreover, botanical extracts as well as mineral oils are not likely to cause pesticide resistance among pests due to their molecule complexity (Bedini *et al*., 2020) and do not generate dangerous residues in the water and soil, which in chemical pesticides can cause substantial environmental pollution (Kundu *et al.*, 2020)

Garlic (*Allium sativum* L.) essential oil was demonstrated to possess insecticidal activity against many insect pests [i.e. *A. gossypii*, *Tuta absoluta* (Lepidoptera: Gelechiidae), *Lycoriella ingénue* Dufour (Diptera: Sciaridae), *Reticulitermes speratus* Kolbe (Isoptera: Rhinotermitidae) and several grain storage insects as *Ephestia kuehniella* Zeller (Lepidoptera: Pyralidae), *Sitophilus oryzae* Linnaeus, *Sitophilus zeamais* Motschulsky (Coleoptera: Curculionidae), *Tribolium castaneum* Herbst (Coleoptera: Curculionidae), and *Tenebrio molitor* L. (Coleoptera: Tenebrionidae) (Huang *et al.,* 2000; Plata-Rueda *et al.,* 2017). The major bioactive components responsible for the benefits of garlic are assumed to be allylic sulfur compounds (Banerjee and Maulik, 2002). The camphor tree, *Cinnamomum camphora* (L.) is a medicinal plant, which have a camphor-like aroma. For pest control, most studies have focused on the insecticidal and repellent activity of camphor essential oil (Jiang *et al.,* 2016). Camphor oils can be developed as larvicides against insect pests such as *Lucilia sericata* (Diptera: Calliphoridae) (Shalaby *et al*., 2016). Mineral oils are the distillation products of petroleum consisting of four compounds' types: naphthene, paraffins, aromatics and olefins (Nile *et al.,* 2019). The insecticidal properties of these oils were recognized as it had been used to control insect pests of fruit trees (Vincent *et al.,* 2003) and management of vector-virus complexes in potato crops (Wróbel, 2012). In addition to essential oils and mineral oils, chitosan is produced from chitin, a natural amino polysaccharide, which is extracted from the exoskeleton of crustaceans, insect, fungal cell walls, present in abundant numbers and known as nontoxic and biodegradable properties (Jia *et al*., 2016). Chitosan application on insects had mostly focused on their effect as insecticides, such as those for *A. gossypii, Callosobruchus maculatus* (Sahab *et al.,* 2015), and *Spodoptera littoralis* (Badawy *et al.,* 2005).

Keeping in view the previous information, the present work aims to evaluate the toxicity of the two essential oils of camphor and garlic plants as well as chitosan in comparison with the paraffin mineral oil against *A. gossypii* and *G. ficorum* under laboratory conditions. In addition, to investigate the impact of the median lethal concentration (LC_{50}) of each treatment on the activities of some detoxifying enzymes in treated *A. gossypii* and *G. ficorum.*

MATERIALS AND METHODS

The insect pests

Cotton aphids, *Aphis gossypii* (Glover) (Aphidoidea: Hemiptera), and black thrips, *Gynaikothrips ficorum* (Thysanoptera: Phlaeothripidae), were collected from free-pesticide zucchini plants and ficus trees, respectively.

The identification of both insects was confirmed at the Plant Protection Research Institute, Agriculture Research Center, Egypt.

The evaluated materials as natural pesticides:

Two essential oils of camphor (*Cinnamomum camphora*) and garlic (*Allium sativum*) were obtained with their chemical analysis results from the Pure Life Company, Cairo, Egypt. Table 1 shows the composition of the essential oils. The commercial formulation of Agre-Blue mineral oil (Paraffin oil, 83% EC) was obtained from the Plant Protection Research Institute, Agriculture Research Center, Egypt. Also, chitosan solution ≥75% deacetylated (Sigma-Aldrich/ CAS no. 9012-76-4) was obtained from El-Nasr Company, Egypt. All oils and chitosan were used to investigate their efficacy in controlling *A. gossypii* and *G. ficorum* under laboratory conditions.

Table 1. The composition of camphor and garlic oils

No.	Camphor oil		Garlic oil	
	Compound	%	Compound	%
1	Tricyclene	1.04	Acids	
2	Thujene <alpha>	3.05	Myristic acid	1.0
3	Pinene <alpha>	8.59	Palmitic acid	10.0
4	Fenchene <alpha>	0.27	Palmitoleic acid	1.0
5	Camphene	2.98	Stearic acid	4.0
6	Sabinene	12.76	Oleic acid	20.0
7	Pinene <beta>	6.17	Linoleic acid	50.0
8	5-Hepten-2-one, 6-methyl-	0.17	Arachidic acid	4.0
9	Menthene < 3-p>	0.04	Gadoleic acid	1.0
10	Myrcene	11.93	Behenic acid	3.5
11	2,3-Dehydro-1,8-cineole	0.04	Erueic acid	1.5
12	2-Carene	0.95		
13	Phellandrene <alpha>	2.64	Active ingredients	
14	3-Carene	0.11	Diallyl disulfide	3.0
15	Terpinene	0.59	Diallyl trisulfide	24.0
16	Cymene <para->	0.91	Alliin	
17	Limonene	10.16	Allicin	39.0
18	Phellandrene <beta>	0.44	Sulfoxid	
19	1,8-Cineole (Eucalyptol)	45.38	Alliinase	
20	Terpinene <gamma>	0.04	Peroxidase	
21	Fenchol <endo>	0.03	Myrosinase	
22	Camphor	30.38		
23	Borneol	0.50		

Bioassay procedure

Four concentrations (2500, 5000, 7500, and 10000 ppm) were prepared from each treatment (camphor oil, garlic oil, chitosan, and mineral oil). 0.05% of Tween 80 was added to prepare the concentrations of camphor and garlic oils to make an emulsion of oil with water, but chitosan and mineral oil (Commercial formulation) were prepared directly in water. The control treatment was prepared by the mixture of water and 0.05% Tween 80. Ten individuals from the adult stage of the tested insect (*A. gossypii* or *G. ficorum*) were transferred to a petri dish (9 cm in diameter), which contains filter paper to absorb the high humidity. The Petri dishes were provided with fresh leaves of zucchini (for *A. gossip*) and ficus (for *G. ficorum*). Two ml of each concentration were sprayed on the insects in each petri dish. Each treatment was repeated four times. The dead insects were counted and recorded daily.

Estimating the change in the insect enzymes after treatment

The median lethal concentrations (LC50) of the evaluated oils and chitosan were calculated according to the Finny method (Finney, 1971). The LC50 of each treatment was prepared and sprayed on the tested insects.

After 24 hours, the live insects in each treatment and control were collected and weighted in eppendorf tubes. All the eppendorf tubes were frozen (-20 oC). The activities of five detoxifying enzymes were estimated colorimetrically by a UV visible spectrophotometer (model V1200, China) at the Plant Protection Research Institute (Mansoura branch), Agriculture Research Center, Mansoura, Egypt. The glutamate pyruvate transaminase (GPT) and glutamate oxaloacetic transaminase (GOT) activities were estimated at 505 nm as described by Reitman and Frankel (1957), glutathione S-transferase (GST) activities were estimated at 340 nm as described by Pan *et al.* (2016), alkaline phosphatase (ALP) and acid phosphatase (ACP) activities were estimated at 510 nm as described by Powell and Smith (1954).

Statistical analysis

Mortality percentages of the treated insects by the evaluated oils and chitosan were corrected by Abbot's formula (Abbot, 1925). The results of the bioassay test and insect enzyme activity were subjected to analysis of variance (ANOVA) and calculate the standard error (SE) by using CoHort software (CoHort, 2004). Median lethal concentration and slope values were calculated by the Finney method according to Finney (1971) by using LDP-line software. Toxicity index was calculated according to the Sun equation (Sun, 1950).

RESULTS AND DISCUSSION

1. Insecticidal activity of evaluated oils and chitosan against *A. gossypii* and *G. ficorum*:

a) ***A. gossypii:***

The results illustrated in Table (2) show that the camphor oil was significantly the most effective treatment against *A. gossypii*, followed by garlic oil and mineral oil. While chitosan showed the lowest effects on *A. gossypii* adults, with significant differences compared with other tested treatments. The mortality percentage of *A. gossypii* increased significantly after 48 hours compared to 24 hours, where the mortality percentages reached 97, 90, 87, and 70% in the treated insects by camphor, garlic, mineral oils, and chitosan after 48 hours, respectively, while they were 87, 83, 77, and 63% after 24 hours, respectively, at 10000 ppm concentration.

In addition, the results in Table (3) show the toxicity of the tested treatments against *A. gossypii* after 48 hours of treatment. The most toxic treatment was camphor oil, whose LC_{50} and LC_{90} were 1379 and 7527 ppm, respectively, followed by garlic oil (3302 and 10553 ppm) and mineral oil (3321 and 12964 ppm), while the least toxic treatment was chitosan, whose LC_{50} and LC_{90} were 5506 and 18606 ppm, respectively. The toxicity index at LC_{50} and LC_{90} arranged the treatments in descending order as follows: camphor oil, followed by garlic oil, mineral oil, and chitosan.

Table 2. Mortality % of the treated *A. gossypii* at different concentrations of tested treatments under laboratory conditions

Treatments	Mortality % of *A. gossypii* at different concentrations ±SE							
	After 24 hours				After 48 hours			
	2500 ppm	5000 ppm	7500 ppm	10000 ppm	2500 ppm	5000 ppm	7500 ppm	10000 ppm
Chitosan	$7^{c}\pm1.15$	$43^{c}\pm1.73$	$53^{b}\pm1.73$	$63^{c}\pm1.73$	$17^{c}\pm1.15$	$53^{c}\pm1.73$	$63^{c}\pm1.73$	$70^{c}\pm2.88$
Mineral oil	$33^{b}\pm1.73$	$47^{c}\pm1.15$	$73^{a}\pm1.73$	$77^{b}\pm4.04$	$43^{b}\pm1.73$	$57^{bc}\pm1.15$	$80^{b}\pm2.88$	$87^{b}\pm1.15$
Camphor oil	$60^{a}\pm2.88$	$70^{a}\pm2.88$	$80^{a}\pm2.88$	$87^{a}\pm1.15$	$70^{a}\pm2.88$	$80^{a}\pm2.88$	$87^{a}\pm1.15$	$97^{a}\pm1.15$
Garlic oil	$30^{b}\pm1.15$	$63^{b}\pm1.73$	$77^{a}\pm4.04$	$83^{ab}\pm1.73$	$40^{b}\pm1.15$	$63^{b}\pm1.73$	$83^{ab}\pm1.73$	$90^{b}\pm1.15$
F. test	***	***	***	***	***	***	***	***
LSD (5%)	6. 10	6.45	9.02	7.93	6.10	6.45	6.45	5.72

Values with different letters in the same column are significantly different according to Fisher's test at *$P < 0.05$*.

Table 3. Toxicity of tested treatments against the adult stage of *A. gossypii* under laboratory conditions after 48 hours

Treatments	LC_{50} (ppm)	Confidence limit (95%)		LC_{90} (Ppm)	Confidence limit (95%)		*Slope ± SE*	x^2	Toxicity index (%)	
		Lower ppm	Upper Ppm		Lower ppm	Upper ppm			LC_{50}	LC_{90}
Chitosan	5506	4839	6241	18606	14338	28195	2.42 ±0.31	3.29	25.0	40.45
Mineral oil	3321	2633	3902	12964	10257	18987	2.16 ±0.30	3.91	41.5	58.06
Camphor oil	1379	619	2022	7527	6033	10968	1.73±0.33	4.40	100	100
Garlic oil	3302	2722	3805	10553	8791	13869	2.54 ±0.31	1.39	41.8	71.32

b) *G. ficorum*

On the other hand, garlic oil was the most effective treatment against *G. ficorum* compared with other tested treatments. The mortality percentages of treated *G. ficorum* with garlic oil reached 73, 93, and 96% after 24, 48, and 72 hours, respectively, at 10000 ppm. The next effective treatment was camphor oil, followed by mineral oil and chitosan as shown in Table (4).

The data and statistical analysis in Table (4) indicate that there isn't a significant difference between chitosan, mineral oil, and camphor oil at most concentrations. In general, the mortality percentages of *G. ficorum* were high after 48 and 72 hours with all treatments.

The results of toxicity analysis by Log Dose-Probit line (LD-P line) indicate that the most toxic treatment against *G. ficorum* was garlic oil followed by camphor oil, then mineral oil, and chitosan as shown in Table (5).

The LC50 and LC90 of garlic oil after 48 hours of treatment were 4245 and 9909 ppm, respectively, but the LC50 and LC90 of chitosan were 7011 and 22219 ppm after 48 hours of treatment. According to the toxicity index at LC50 and LC90 values, the treatments could be arranged in descending order as follows: garlic, then camphor, mineral oils, and chitosan, respectively.

Table 4. Mortality % of treated *G. ficorum* by different concentrations of tested treatments under laboratory conditions

Treatments	Mortality % of *Gynaikothrips ficorum* at different concentrations ± SE											
	After 24 hours				After 48 hours				After 72 hours			
	2500 ppm	5000 ppm	7500 ppm	10000 ppm	2500 ppm	5000 ppm	7500 ppm	10000 ppm	2500 ppm	5000 ppm	7500 ppm	10000 ppm
Chitosan	3[b] ±0.57	27[c] ±1.15	33[d] ±1.73	47[c] ±1.15	14[c] ±1.15	32[c] ±1.15	54[c] ±2.30	68[b] ±4.61	21[b] ±0.57	39[c] ±2.88	64[c] ±2.30	75[c] ±2.89
Mineral oil	7[b] ±1.15	37[b] ±4.04	43[c] ±1.73	57[b] ±3.48	18[bc] ±1.73	43[b] ±1.75	61[b] ±0.58	75[b] ±2.88	29[ab] ±5.19	50[b] ±2.89	68[bc] ±4.61	82[bc] ±1.15
Camphor oil	13[a] ±1.73	40[ab] ±2.31	53[b] ±1.73	57[b] ±1.15	21[ab] ±0.57	46[b] ±3.46	64[b] ±2.31	79[b] ±5.19	36[a] ±3.46	54[ab] ±2.30	75[b] ±2.88	86[b] ±3.46
Garlic oil	17[a] ±2.88	47[a] ±2.88	63[a] ±1.73	73[a] ±1.73	25[a] ±2.88	54[a] ±2.30	82[a] ±1.15	93[a] ±1.73	39[a] ±2.30	61[a] ±0.57	89[a] ±2.30	96[a] ±0.58
F. test	**	**	***	***	*	***	***	*	*	**	**	**
LSD (5%)	5.87	9.12	5.64	6.87	5.87	7.58	5.72	12.59	10.89	7.70	10.35	7.64

Values with different letters in the same column are significantly different according to Fisher's test at *P < 0.05*.

Table 5. Toxicity of tested treatments against the adult stage of *G. ficorum* under laboratory conditions after 48 hours

Treatments	LC_{50} (ppm)	Confidence limit (95%) Lower ppm	Confidence limit (95%) Upper ppm	LC_{90} (ppm)	Confidence limit (95%) Lower Ppm	Confidence limit (95%) Upper Ppm	*Slope ± SE*	x^2	Toxicity index (%) LC_{50}	Toxicity index (%) LC_{90}
Chitosan	7011	6228	8057	22219	16776	34929	2.55±0.32	0.80	60.54	44.59
Mineral oil	5868	5221	6608	18040	14193	26175	2.62±0.32	0.15	72.34	54.92
Camphor oil	5282	4663	5938	16567	13157	23650	2.58±0.31	0.39	80.36	59.81
Garlic oil	4245	3802	4669	9909	8674	11860	3.48±0.33	2.88	100	100

c) Comparison between *A. gossypii* and *G. ficorum*

Data illustrated in Figure (1) show that *A. gossypii* was more sensitive to all of the evaluated treatments than *G. ficorum*, where the values of LC_{50} against *A. gossypii* were lower than their counterparts against *G. ficorum*. On the other hand, *A. gossypii* was relatively more sensitive to camphor oil and less sensitive to chitosan, while *G. ficorum* was relatively more sensitive to garlic oil and less sensitive to chitosan.

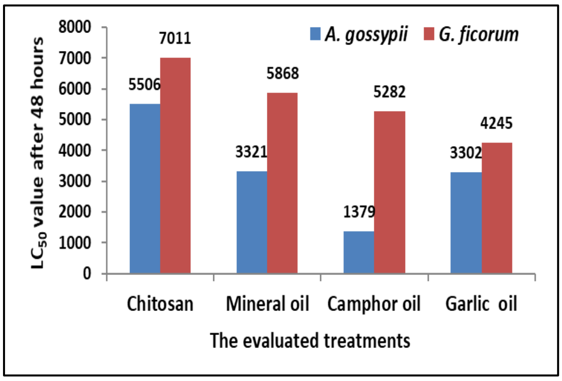

Fig. 1. The sensitivity of both tested insects against the evaluated treatments after 48 hours of treatment

2. Impact of evaluated treatments on the activities of some detoxifying enzymes in *A. gossypii* and *G. ficorum*

a) *A. gossypii*

As shown in Table (6), all tested treatments led to significant inhibition of the GOT, ACP, and ALP enzyme activity in treated *A. gossypii* compared with untreated insects. Also, all tested treatments caused significant inhibition of the GPT enzyme activity in treated *A. gossypii*, except chitosan, which led to activation compared with untreated insects. As well, chitosan and camphor oil caused significant inhibition of the GST enzyme activity, but mineral oil and garlic oil led to significant activation of the GST enzyme in treated insects compared with untreated insects (Table 6).

b) *G. ficorum*

On the other hand, the activity of estimated enzymes in treated *G. ficorum* with chitosan, mineral oil, camphor oil, and garlic oil showed significant inhibition compared with untreated insects. The inhibition percentage ranged from 38 to 62% in GOT, 49 to 84% in GPT, 12 to 63% in ACP, 25 to 84% in ALP, and 6 to 53% in GST, as shown in Table (7). Also, the higher inhibition percentages were in the treated *G. ficorum* by garlic oil in all estimated enzymes except ACP.

c) Comparison between *A. gossypii* and *G. ficorum*

As results in Tables (6 and 7) and Figure (2), chitosan and mineral oil led to inhibition of estimated enzymes in *G. ficorum* higher than *A. gossypii*, except with ALP and GST enzymes, which had the opposite effect. In addition, the GPT and GST enzymes were activated in *A. gossypii*, but they were inhibited in *G. ficorum* by using chitosan and mineral oil. Also, camphor oil caused inhibition in all estimated enzymes, but the inhibition percentage in *G. ficorum* was higher than in *A. gossypii*, except with the ALP and GST enzymes. The garlic oil led to inhibition in all estimated enzymes in both insects except GST in *A. gossypii*, but the inhibition percentages were higher in *G. ficorum* than in *A. gossypii* except with ACP and GST enzymes.

Table 6. Effect of the tested treatments on the activities of some detoxifying enzymes in adults of *A. gossypii*

Treatments	Enzymes activity ± SE									
	GOT U/ml	Ch %	GPT U/ml	Ch %	ACP U/ml	Ch %	ALP U/ml	Ch %	GST Mmol sub. conjugated/min. mg protein	Ch %
Control	4.48[a]±0.03	00	4.95[b]±0.02	00	0.75±0.02	00	1.82[a]±0.03	00	7.84[c]±0.04	00
Chitosan	3.53[b]±0.02	-21	5.44[a]±0.03	+9	0.43[c]±0.03	-42	0.33[c]±0.01	-81	5.14[e]±0.10	-34
Mineral oil	2.78[c]±0.03	-37	4.04[c]±0.03	-18	0.67[b]±0.01	-10	0.38[c]±0.01	-79	9.36[a]±0.06	+19
Camphor oil	2.01[d]±0.01	-55	2.27[d]±0.02	-54	0.37[c]±0.01	-50	0.58[b]±0.02	-68	5.39[d]±0.02	-31
Garlic oil	1.83[e]±0.02	-59	2.10[e]±0.04	-57	0.36[c]±0.02	-52	0.33[c]±0.01	-81	8.85[b]±0.03	+12
F. test	***		***		***		***		***	
LSD (5%)	0.08		0.10		0.06		0.06		0.08	

Ch %: Change percentage compared with control, (-): inhibition.
Values with different letters in the same column are significantly different according to Fisher's test at *P < 0.05*.

Table 7. Effect of the tested treatments on the activities of some detoxifying enzymes in treated *G. ficorum* adults

Treatments	Enzymes activity ± SE									
	GOT U/ml	Ch %	GPT U/ml	Ch %	ACP U/ml	Ch %	ALP U/ml	Ch %	GST Mmol sub. conjugated/min. mg protein	Ch %
Control	7.12^{a}±0.01	00	2.68^{a}±0.03	00	1.03^{a}±0.01	00	0.44^{a}±0.01	00	6.41^{a} ±0.01	00
Chitosan	4.36^{b}±0.03	-38	1.36^{b}±0.02	-49	0.38^{d}±0.03	-63	0.26^{c}±0.01	-40	5.31^{c}±0.01	-17
Mineral oil	3.53^{c}±0.02	-50	1.18^{c}±0.03	-55	0.43^{c}±0.02	-58	0.14^{d}±0.01	-68	4.30^{d}±0.02	-32
Camphor oil	2.79^{d}±0.03	-60	0.93^{d}±0.02	-65	0.40^{cd}±0.02	-61	0.33^{b}±0.01	-25	5.98^{b}±0.03	-6
Garlic oil	2.68^{e}±0.02	-62	0.42^{e}±0.01	-84	0.90^{b}±0.02	-12	0.07^{e}±0.001	-84	2.95^{e}±0.02	-53
F. test	***		***		***		***		***	
LSD (5%)	0.08		0.08		0.07		0.004		7.27	

Ch % : Change percentage compared with control, (-) : Inhibition, (+) : Activation.
Values with different letters in the same column are significantly different according to Fisher's test at *P* < *0.05*.

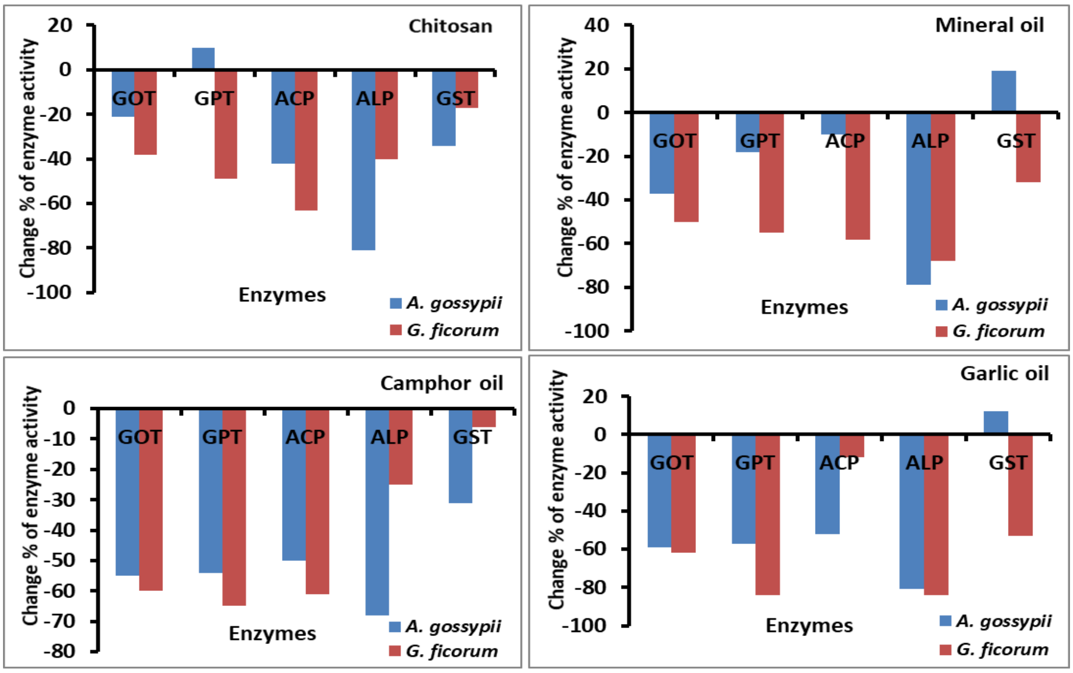

Fig. 2. Effect of evaluated oils and chitosan on the enzymes activity in *A. gossypii* and *G. ficorum*

More than 92 plant families, including field, vegetable, fruit, and ornamental crops, are attacked by the polyphagous cotton aphid, *Aphis gossypii* (Glover) (Hemiptera: Aphididae). (Somar *et al*., 2019). According to Tavares *et al.* (2013), the thrips, *G. ficorum* (Thysanoptera: Phlaeothripidae), is a monophagous insect pest that has been extensively documented in all regions where its host plant, *F. microcarpa* (Marchal), has been cultivated in urban and interior landscape plant species nationwide. The plant-based compounds are gaining acceptance as a strategy for integrated pest management due to their low toxicity to non-target organisms and ecosystems, as well as the slow evolution of insect resistance (Zhao *et al.*, 2013). Many previous studies mentioned the toxic effects of different plant essential oils against several insect pests such as *S. littoralis* (Ali *et al*., 2017), *Phthorimaea operculella* (Zell.) and *Agrotis ipsilon* (Huf.) (Adel *et al*., 2015), and *Trichoplusia ni* (Tak and Isman, 2016). In this study, garlic oil, camphor oil, mineral oil, and chitosan were evaluated as insecticides against *A. gossypii* and *G. ficorum* under laboratory conditions. The results of this study indicated that garlic oil and camphor oil were more toxic against tested insects, *A. gossypii* and *G. ficorum*, followed by mineral oil and chitosan. In general, all tested treatments caused high mortality percentages, ranging from 70 to 97% against *A. gossypii* and 68 to 93% against *G. ficorum* at 10000 ppm after 48 hours of treatment. The higher percentages of insect mortality caused by garlic and camphor oils may be because of their active ingredients, such as diallyl disulfide (3%), diallyl trisulfide (24%), allicin (39%), and many fatty acids in garlic oil, as well as camphor (30.38%), 1,8-cineole (also known Eucalyptol) (45.38%), sabinene (12.76%), limonene (10.16%), and pinene (14.76%) in camphor oil, as shown in Table (1).

In the previous studies, Yang *et al.* (2012) reported that diallyl disulfide and diallyl trisulfides are two of the major active components of garlic essential oil. Also, garlic essential oil, diallyl disulfide, and diallyl trisulfide had significant toxicity against *Sitotroga cerealella*. Similar results were mentioned by Plata-Rueda *et al.* (2017) who reported that garlic essential oil and their compounds caused lethal and sublethal effects on *Tenebrio molitor* (Coleoptera: Tenebrionidae) and, therefore, have the potential for pest control. Also, Omar and Zayed (2021) estimated the impact of garlic and mandarin essential oils against two stored product insects. They found that garlic oil was more effective compared with mandarin. Also, they mentioned that the major contents of garlic oil were diallyl sulfide, diallyl disulfide, diallyl trisulfide, dimethyl tetrasulfide, and allyl methyl trisulfide. Likewise, garlic essential oil is also rich in secondary metabolites, including tannins, alkaloids, steroids, and saponins, which have been shown to act as antifeedants against a variety of insect orders, including

Diptera, Lepidoptera, and Coleoptera, and may influence the developmental process (Ali *et al.*, 2017; Zayed *et al.*, 2009). Mousa *et al.* (2013) evaluated garlic and camphor oils, dimethoate, and pestban against some piercing-sucking insect pests, and the results showed that garlic oil caused a high reduction in the population of leaf hoppers and plant hoppers in the field application, but camphor oil was the least effective treatment.

In our study, the mortality percentage of *A. gossypii* and *G. ficorum* increased with the concentration of treatments and the exposure time. Similar results were mentioned by Alghamdi (2018), who studied the effect of four plant essential oils, *Moringa oleifera*, *Eruca sativa*, *Raphanus sativus*, and *Allium sativum*, on the mortality of *Macrosiphum rosae* and *Aphis fabae*. The results revealed that mortality percentages increased with the concentrations tested and the exposure times. Also, the garlic oil at 2% concentration caused 86% mortality in *M. rosae* and 80% in *Aphis fabae* after 48 hours of treatment.

As well, Guo *et al.* (2016) studied the chemical composition and insecticidal activity of the camphor essential oil, which were found to possess strong toxicity against *Tribolium castaneum* and *Lasioderma serricorne* adults. It was also mentioned that the major components, such as camphor (30.38%) and 1,8-cineole (45.38%), had more toxicity against the tested insects. Similar results were found with Xu *et al.* (2020), who found that the camphor oil showed strong, dose-dependent larvicidal activities against *Anopheles stephensi*, and the onset of larvicidal efficacy was rapid. Additionally, it has been demonstrated that the widely accessible substance α-pinene possesses larvicidal properties against *Aedes aegypti* (Freitas *et al.*, 2010). Likewise, Rafea *et al.* (2022) evaluated the camphor oil (bulk and nanoemulsion) against *Spodoptera littoralis*, who found that the LC_{50} was 20232 ppm in the case of bulk emulsion but 1664 ppm in the case of nanoemulsion of camphor oil. Also, they found that the main component in the camphor oil was eucalyptol (1,8-cineole). Ahmed *et al.* (2021) evaluated camphor essential oil against the green peach aphid, *Myzus persicae* (Aphididae: Hemiptera), in the laboratory. They mentioned that the camphor essential oil significantly reduced and controlled *M. persicae* population and caused higher mortality. Also, they reported that the essential oil formulation was effective in reducing the mortality of aphids. In addition, camphor essential oil is thought to be non-polluting, environmentally benign, and to have little to no toxicological effect. It has a broad range of pesticide qualities, such as insecticidal, insect repellent, herbicidal, acaricidal, fungicidal, and anti-microbial (Ben-Issa *et al.*, 2017). Insecticidal effects of camphor essential oil include contact, antifeeding, oviposition inhibition, repellence, and fumigant. Cotton leafhoppers and cotton stainers are effectively combatted by camphor leaf extract through the inhibition of oviposition method, which also works against aphids through the contact test method and antifeeding. Camphor essential oil fumigant repels house flies, and potatoes are shielded from potato tuber moths by the oil and dry powder extract (Hammer *et al*., 2006).

In our study, 1.8-cineole (Eucalyptol), alpha-pinene, beta-pinene, terpinene, camphor, sabinene, and limonene were the major chemical compositions in the camphor essential oil, as shown in Table 1. Many previous studies reported that 1.8-cineole has insecticidal activities against many insects (Ben-Issa *et al*., 2017); alpha-pinene, beta-pinene, and camphor exhibit pesticide action that is used in pest control (Isman, 2006). Moreover, terpinene had insecticidal properties and affected insect enzymes such as AChE, GST, and CarE (Isman, 2000).

In our present study, garlic and camphor oils led to inhibition of estimated detoxifying enzyme activities such as GOT, GPT, ALP, ACP, and GST. Similar results were found by Halliwell and Gutteridge (1999); Singh and Singh (1996), who demonstrated that garlic compounds inhibit acetylcholinesterase, an enzyme that functions alone or in concert with diallyl disulfide, diallyl trisulfide, and allicin. In addition, the toxic effect in *T. molitor* may be caused by diallyl sulfide, a compound found in garlic, which also has the ability to cross-link with essential thiol compounds in enzyme structures, changing the functional shape of the protein and denaturalizing it.

Chitosan's biocompatibility and biodegradability make it a viable alternative to pesticides in pest management. Several studies have proven the potential use of chitosan to control insect pests. Badawy *et al.* (2005) used chitosan against *Spodoptera littoralis*, and Sahab *et al.* (2015) used chitosan against *Aphis gossypii*, *Callosobruchus maculatus,* and *Callosobruchus maculatus* insects. In addition, Rabea *et al.* (2005) discovered that chitosan exhibits potent insecticidal activity against some insect pests. They discovered that a chitosan derivative has insecticidal activity against the larvae of *Spodoptera littoralis*. Also, Abdullah and Sukar (2021) studied the efficiency of a chitosan mixture with *Beauveria bassiana* metabolites against the 2nd instar larvae of *S. littoralis*. Who found that the mortality percentage reached 57% when using chitosan alone but was 86% when using the mixture. Also, they mentioned that the growth rate of larvae was affected when treated with chitosan alone or in a mixture.

Finally, the present study indicated that both garlic and camphor essential oils showed a significant insecticidal impact on *A. gossypii* and *G. ficorum* and exhibited inhibition effects on some detoxifying enzymes in the treated insects. The obtained results thus suggested the efficiency of these essential oils in being used as natural insecticides. Also, the compounds in garlic and camphor essential oils are potential sources of insecticidal compounds and warrant further exploration.

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السمية والتأثيرات البيوكيميائية لزيوت الثوم والكافور العطرية ضد *Aphis gossypii* و *Gynaikothrips ficorum* مقارنة بالشيتوزان والزيوت المعدنية

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معهد بحوث وقاية النباتات – مركز البحوث الزراعية – الدقي – الجيزة - مصر

الملخص

تعتبركلا من *Aphis gossypii* و *Gynaikothrips ficorum* من الآفات الحشرية الاقتصادية الهامة في مصر. حيث أنهما يسببان خسائر اقتصادية للعديد من المحاصيل. تعتبر الزيوت العطرية النباتية أقل ضررًا على البيئة، وهي أقل سمية للثدييات، وأقل تكلفة مقارنة بالمبيدات الحشرية المصنعة، ولها نشاط بيولوجي فعال. تهدف هذه الدراسة إلى معرفة كفاءة زيت الثوم والكافور العطري والزيوت المعدنية والشيتوزان ضد *A. gossypii* و *G. ficorum* وكذلك دراسة تأثيرها على نشاط بعض الإنزيمات المزيلة للسموم مثل GPT, GOT, GS-T، ALPو ACP . أشارت النتائج المتحصل عليها إلى أن زيت الثوم والكافور العطري كانا الأكثر سمية ضد الحشرات المختبرة *A. gossypii* و *G. ficorum* مقارنة بالزيوت المعدنية والشيتوزان. وحسب مؤشر السمية عند LC_{50} و LC_{90} فقد رتبت المعاملات في المرتبة الأولى زيت الكافور ثم زيت الثوم والزيت المعدني والكيتوزان في حالة *A. gossypii* ، بينما رتبت المعاملات زيت الثوم ثم زيت الكافور والزيت المعدني والكيتوزان في حالة *G. ficorum* . و بشكل عام، تسببت جميع المعاملات المختبرة في ارتفاع نسب الموت، حيث تراوحت بين ٧٠ إلى ٩٧٪ ضد *A. gossypii* و٦٨ إلى ٩٣٪ ضد *G. ficorum* عند التركيز ١٠٠٠٠ جزء في المليون بعد ٤٨ ساعة من المعاملة. بالإضافة إلى ذلك، تم تثبيط نشاط معظم الإنزيمات المقدرة في جميع المعاملات باستثناء بعضها. وبناءا علي ما تقدم، فإن زيت الثوم والكافورالعطري يكتسبان القبول كاستراتيجية للإدارة المتكاملة للآفات بسبب أمانهما علي الانسان والبيئة وكذلك سميتهما العالية ضد *A. gossypii* و *G. ficorum* .