Larvicidal and adulticidal effects of some Egyptian oils against *Culex pipiens*

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Mosquitoes and mosquito-borne diseases represent an increasing global challenge. Plant extract and/or oil could serve as alternatives to synthetic insecticides. The larvicidal effects of 32 oils (1000 ppm) were screened against the early 4th larvae of *Culex pipiens* and the best oils were evaluated against adults and analyzed by gas chromatography-mass spectrometry (GC mass) and HPLC. All oils had larvicidal activity (60.0–100%, 48 h Post-treatment, and their Lethal time 50 (LT₅₀) values ranged from 9.67 (*Thymus vulgaris*) to 37.64 h (*Sesamum indicum*). Oils were classified as a highly effective group (95–100% mortalities), including *Allium sativum*, *Anethum graveolens*, *Camellia sinensis*, *Foeniculum vulgare*, *Nigella sativa*, *Salvia officinalis*, *T. vulgaris*, and *Viola odorata*. The moderately effective group (81–92% mortalities) included *Boswellia serrata*, *Cuminum cyminum*, *Curcuma aromatic*, *Allium sativum*, *Melaleuca alternifolia*, *Piper nigrum*, and *Simmondsia chinensis*. The least effective ones were *C. sativus* and *S. indicum*. *Viola odorata*, *Anethum graveolens*, *T. vulgaris*, and *N. sativa* provide 100% adult mortalities PT with 10, 25, 20, and 25%. The mortality percentages of the adults subjected to 10% of oils (H group) were 48.89%, 88.39%, 63.94%, 51.54%, 92.96%, 44.44%, 72.22%, and 100% for *A. sativum*, *A. graveolens*, *C. sinensis*, *F. vulgare*, *N. sativa*, *S. officinalis*, *T. vulgaris*, and *V. odorata*, respectively. *Camellia sinensis* and *F. vulgare* were the most potent larvicides whereas *V. odorata*, *T. vulgaris*, *A. graveolens* and *N. sativa* were the best adulticides and they could be used for integrated mosquito control.

Mosquitoes are an ancient nuisance pest and mosquito-borne diseases represent an increasing global health challenge, threatening over 40% of the world’s population and it is expected that almost half of the world’s population will be at risk of arbovirus transmission by 2050¹. *Culex pipiens* (Diptera: Culicidae) is widely distributed, transmitting dreadful diseases leading to severe morbidity and sometimes mortality to humans and animals²–⁵. Vector control is the primary method for reducing public concerns about mosquito-borne diseases⁶–¹¹. Controlling adults and larvae through repellents and insecticides¹²,¹³, are the most effective approach for reducing mosquito bites. Using synthetic insecticides led to insecticide resistance, environmental pollution, and health hazards to human health and non-target organisms.

Searching for eco-friendly alternatives in botanicals such as essential oils (EOs) is a curtail need. EOs are volatile components found in many plant families like Asteraceae, Rutaceae, Myrtaceae, Lauraceae, Lamiaceae, Apiaceae, Piperaceae, Poaceae, Zingiberaceae, and Cupressaceae¹⁴. EOs contain complicated mixtures of products as phenols, sesquiterpenes, and monoterpenes¹⁵. EOs have antibacterial, antiviral, and antifungal activities. They also possess insecticidal effect interfering with insects’ physiological, metabolic, behavioral, and biochemical functions through inhalation, ingestion, or skin absorption of EOs inducing a neurotoxic action¹⁶. EOs act as adulticides, larvicides, deterrents, and repellents. They are less toxic, biodegradable, and overcome insecticidal resistance¹⁵,¹⁷,¹⁸. EOs have higher popularity with organic growers and environmentally conscious consumers and suitability for urban areas, homes, and other sensitive areas.

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The role of EOs in mosquito control has been discussed. This study aimed to screen and evaluate the lethal time values of the larvicidal effects of thirty-two oils and evaluate the adulticidal effect and phytochemical analyses of the most effective ones against *Cx. pipiens*.

### Materials and methods

#### Plant oils

Thirty-two oils were purchased from EL CAPTAIN Company for extracting natural oils, plants, and cosmetics "Cap Pharm," El Obor, Cairo, Egypt, and Harraz for Food Industry & Natural products, Cairo, Egypt (Table 1).

#### Culex pipiens

* Culex pipiens* (an autogenous strain) was provided from the colony reared at the Department of Entomology, Faculty of Science, Benha University, Egypt, and maintained at 27 ± 2 °C, 75–85% RH and 14:10 h (L/D) photoperiod.

#### Larvicidal efficacy

Thirty-two oils were screened for their larvicidal efficacy against the early fourth instar larvae, *Cx. pipiens*. Oils were added to a solvent (emulsifier) consisting of dechlorinated water plus 1.0 mL of 0.5% Tween-20, through a shaker plate to yield a homogenous solution. Oils were added to a solvent consisting of dechlorinated water plus 5% Tween 20. For each oil, twenty larvae were placed in a 500 mL glass beaker containing 250 mL of 1000 ppm. The experiment and the control group, treated with the solvent only, were replicated three times. Larval mortalities were recorded 0.5, 2, 8, 24, and 48 h post-treatment (PT).

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Table 1. Plants species screened (oil No = 32) used for larvicidal activity. *Plant oils purchased from EL CAPTAIN company for extracting natural oils, plants and cosmetics "Cap Pharm".* Plant oils purchased from Harraz for Food Industry & Natural products.

| No. | Oil name       | Plant oils                   | Order    | Family         | English name      |
|-----|----------------|------------------------------|----------|----------------|-------------------|
| 1   | *Allium sativum* | Asparagales Amaryllidaceae   | Garlic   |                |                   |
| 2   | *Anethum graveolens* | Apiaceae         | Dill     |                |                   |
| 3   | *Argania spinosa*  | Sapotaceae         | Argan    |                |                   |
| 4   | *Boswellia serrata R.* | Sapindales Bursaraceae | Olibanum |                |                   |
| 5   | *Brassica carinata* | Brassicales Brassicaceae | Mustard  |                |                   |
| 6   | *Camellia sinensis* | Theaceae         | Green Tea |                |                   |
| 7   | *Cedrus libani A.* | Pinaceae           | Cedar wood |                |                   |
| 8   | *Citriulis colocynthis L.* | Cucurbitales Cucurbitaceae | Bitter apple |                |                   |
| 9   | *Crocus sativus L.* | Asparagales Iridaceae | Saffron crocus |                |                   |
| 10  | *Cucurbita maxima D.* | Cucurbitales Cucurbitaceae | Pumpkin |                |                   |
| 11  | *Cuminum cyminum* | Apiales Apiaceae | Camin |                |                   |
| 12  | *Cupressus sempervirens* | Pinales Cupressaceae | Italian cypress |                |                   |
| 13  | *Curcuma aromatica S.* | Zingiberales Zingiberaceae | Zarcuma |                |                   |
| 14  | *Curcuma longa L.* | Zingiberales Zingiberaceae | Common turmeric |                |                   |
| 15  | *Foeniculum vulgare M.* | Apiales Apiaceae | Sweet fennel |                |                   |
| 16  | *Gadus morhua* | Gadiformes Gadidae | Cod Liver |                |                   |
| 17  | *Lepidium sativum L.* | Brassicales Brassicaceae | Garden pepperwort |                |                   |
| 18  | *Linum usitatissimum* | Apiales Apiaceae | Linaceae | Common flax |                |
| 19  | *Melaleuca alternifolia* | Myrtales Myrtaecae | Tea tree |                |                   |
| 20  | *Nigella sativa* | Ranunculales Ranunculaceae | Black cumin |                |                   |
| 21  | *Panax ginseng* | Apiales Araliaceae | Chinese ginseng |                |                   |
| 22  | *Piper nigrum L.* | Piperales Piperaceae | Black pepper |                |                   |
| 23  | *Prunus dulcis* | Rosales Rosaceae | Almond |                |                   |
| 24  | *Ruta chalepensis L.* | Sapindales Rutaceae | Rues |                |                   |
| 25  | *Salvia officinalis L.* | Lamiales Lamiaceae | Sage |                |                   |
| 26  | *Sesamum indicum* | Lamiales Pedaliaceae | Sesame |                |                   |
| 27  | *Simmondsia chinensis* | Caryophyllales Simmondsiaceae | Jojoba |                |                   |
| 28  | *Syzgium aromaticum* | Myrtales Myrtaecae | Clove |                |                   |
| 29  | *Tilia americana L.* | Malvales Malvaceae | Tilia |                |                   |
| 30  | *Thymus vulgaris L.* | Lamiales Lamiaceae | Garden |                |                   |
| 31  | *Viola odorata L.* | Malpighiales Violaceae | Sweet violet |                |                   |
| 32  | *Zingiber officinale* | Zingiberales Zingiberaceae | Ginger |                |                   |
Adulticidal efficacy. Susceptibility tests for adult mosquitoes were performed for the promising larvicidal oils through the CDC bottle bioassays with modifications. For each concentration, three bottles were coated. Several concentrations for each oil were prepared using pure ethanol as a solvent. The bottles were coated with the desired concentrations and left overnight at 27±2°C for solvent evaporation. Adult mosquitoes (15–10, aged 3–4 days) fed on 10% sucrose solution were released to each bottle using a hand aspirator. The exposure time was set to 30 min. The mosquitoes were removed from the bottles. Mosquito groups were added to separate transparent paper cups (10 × 9 × 6 cm) having 10% sucrose solution and mortalities were checked after 24 h. Three replicates were made for each concentration.

GC/MS analysis. A Thermo Scientific Trace GC Ultra/ISQ Single Quadrupole MS, TG-5MS fused silica capillary column was used for the GC/MS study (0.1 mm, 0.251 mm and 30 m film thickness). An electron ionisation device with a 70 eV ionisation energy was employed for GC/MS detection. At a constant flow rate of 1 mL/min, helium gas was used as the carrier gas. Temperatures were established at 280°C for the injector and MS transfer line. The oven temperature was set at 50°C (hold for 2 min), then increased to 150°C at a rate of 7°C per minute, then to 270°C at a rate of 5°C per minute (hold for 2 min), and finally to 310°C at a rate of 3.5°C per minute (hold 10 min). A percent relative peak area was used to explore the quantification of all of the discovered components. The components were tentatively identified by comparing their respective retention times and mass spectra to those of the NIST, WILLY library data from the GC/MS instrument. The identification was done using mass spectra and a computer search of user-generated reference libraries. To check peak homogeneity, single-ion chromatographic reconstruction was used. When identical spectra could not be identified, only the structural type of the relevant component was provided based on its mass spectral fragmentation. When possible, reference compounds were co-chromatographed to confirm GC retention durations.

Data analysis. Data were analyzed through one-way analysis of variance (ANOVA), Duncan's multiple range tests, and Probit analysis for calculating the lethal concentration (LC) and lethal time (LT) values using the computer program PASW Statistics 2009 (SPSS version 22). The relative efficacies (RE) were calculated according to the following formula:

\[ RE = \frac{LT_{50} \text{ or } LC_{50}}{LT_{90} \text{ or } LC_{90}} \]

where \( LT \) is the lethal time and \( LC \) is the lethal concentration. The Chi-square, significance, and regression equations were provided for all tested oils (Table 3).

Results

The larvicidal effect of 32 oils was screened against the early 4th larvae, Cx. pipiens. The results showed that all plant oils had larvicidal activity (60.0–100%, 48 h PT) and their Lethal time 50 (LT50) values ranged from 9.67 (Thymus vulgaris) to 37.64 h (Sesamium indicum), Tables 2 and 3.

The efficacy of oils could be classified, 48 h post-treatment (PT) as the highly effective group (H group) inducing 95–100% mortalities, including eight oils: Allium sativum, Anethum graveolens, Camellia sinensis, Foeniculum vulgare, Nigella sativa, Salvia officinalis, T. vulgaris, and Viola odorata. Camellia sinensis and F. vulgare provided 100%, 24 h PT (Table 2).

The LT50 values of the H group ranged from 9.67 (T. vulgaris) to 19.91 (An. graveolens) hours and those of LT50 values ranged from 29.97 (Foeniculum vulgare) to 55.32 (An. graveolens). The relative efficacies (RE) of such oils according to LT50 values were 2.7, 1.9, 2.9, 3.7, 2.4, 2.4, 2.4, 2.4, and 3.6 times, respectively, times than S. indicum; whereas those of LT50 values were 2.1, 1.8, 2.4, 3.3, 3.0, 3.0, 3.0, 3.0, and 3.0 times, respectively, than C. sativus. The Chi-square, significance, and regression equations were provided for all tested oils (Table 3).

The moderately effective (M group) group of oils resulted in 81–92% mortalities 48 h PT, including B. serrata, C. cyanium, C. aromatic, L. sativum, M. alternifolia, P. nigrom, and S. chinensis. They provided 63.33–71.67% mortalities, 24 h PT (Table 2).

The LT50 values of M group ranged from 19.00 (S. chinensis) to 22.65 (C. cyanium) hours and those of LT90 values ranged from 57.95 (S. chinensis) to 66.22 (M. alternifolia) (Table 3). Their RE: regarding the LT50 values were 1.8, 1.7, 1.8, 1.9, 1.7, 1.9, and 1.9 times than S. indicum, respectively, whereas those of LT90 values were 1.7, 1.6, 1.6, 1.7, 1.5, 1.6, and 1.8 times than C. sativus, respectively (Table 3).

The least effective group (L group) included the other 17 oils, and the least effective ones were C. sativus, and S. indicum, providing 62.33 and 60.00% mortalities, 48 h PT, whereas their LT50 values were 37.07 and 37.64 h and their LT90 values were 96.88 and 92.89 h, respectively (Table 3).

Furthermore, the Kruskal–Wallis test was performed to compare the mean differences of more than two groups followed by the Mann–Whitney test to compare the mean differences between the effective oil groups.

Vioila odorata, A. graveolens, T. vulgaris, and N. sativa provide 100% adult mortality PT with 10. 25, 20, and 25%. The mortality percentages of the adults subjected to 10% of oils (H group) were 48.89%, 88.39, 63.94, 51.54, 92.96, 44.44, 72.22, and 100.0% for A. sativum, An. graveolens, C. sinensis, F. vulgare, N. sativa, S. officinalis T. vulgaris, and V. odorata, respectively. Their adulticidal LC50 values, 24 h PT, were 15.57, 2.42, 9.01, 15.07, 3.42, 20.46, 3.08, and 1.88%, whereas their LC90 values were 38.86, 9.47, 32.18, 33.34, 5.44, 50.76, 16.08, and 7.37%, respectively. Salvia officinalis followed by A. sativum were the least effective oils against adults. According to LC50, N. sativa, V. odorata and An. graveolens killed mosquitoes 9.3, 6.9, and 5.4 times more than S. officinalis (Table 6).
Oil phytochemical analysis. Phytochemical analysis of oils of *F. vulgaris* Mill., *A. graveolens* L., *V. odorata* L., *T. vulgaris* L., *A. sativum*, *S. officinalis* and *C. sinensis* by GC/MS and HPLC analysis revealed their major compounds. *F. vulgaris* oil contains Estragole (70.36%); Limonene (8.96%) and 1,3,3-trimethyl Bicyclo [2.2.1] heptan-2-one (2.81%) (Table 7 and Fig. 1).

Anethum graveolens showed abundance of 4-Pyrindinecarbaldehyde-4-propyl-3-thiosemicarbazone (32.13%); 1,5-dimethyl-1,5-Cyclooctadiene (17.19%); Dihydrocarvone (5.98%); 3a(1H)-Azulenol,2,3,4,5,8,8a-hexahydro-6,8-adimethyl-3-{1-methyl(ethyl),3R-[3a,3a,8a-a]} (Carrotol) (21.26%); and tricyclic compound Daucol (2.39%) (Table 8 and Fig. 2).

*V. odorata* L. oil contains Diphenyl ether (42.04%); alpha.-Iodone (11.87%); (Z)-5-(4-tert-Butyl-1-hydroxyccylohexyl)-3-methylpent-2-ene-4-yne (7.22%); 2,3,4,5,4,6,7,8,9a-decahydro-3,5a,9-trimethyl-7,9a-peroxy Naphtho-[1,2-b]furan-2-one (6.67%); 2-hexyl-1-Decanol (4.15%); and hexacahydro-Pyrene (2.79%) (Table 9 and Fig. 3).

*Thymus vulgaris* oil included 2-Ethynyl-3-hydroxypyridine (12.37%); 2-á-pinene(8.92%); 2,5-Dipropoxybenzaldehyde-4-propyl-3-thiosemicarbazone (32.13%); 1,5-dimethyl-1,5-Cyclooctadiene (17.19%); Dihydrocarvone (5.98%); 3a(1H)-Azulenol,2,3,4,5,8,8a-hexahydro-6,8-adimethyl-3-{1-methyl(ethyl),3R-[3a,3a,8a-a]} (Carrotol) (21.26%); and tricyclic compound Daucol (2.39%) (Table 8 and Fig. 2).

*Allium sativum* contains many effective chemical compounds including the 9-Octadecenamide, (Z)- (29.07%), Trisulfide, di-2-propenyl (14.86%), and isochiapin B%2 < (8.63%) compounds (Table 11 and Fig. 5).
**Table 3.** Lethal time values of applied oils (1000 ppm) against *Culex pipiens* larvae. RE Relative efficacy. Significant values are in [bold].

| Oil name               | LT$_{90}$ (lower-upper) | RE (LT$_{90}$) | LT$_{99}$ (lower-upper) | RE (LT$_{99}$) | Chi (Sig) | Regression equation |
|------------------------|--------------------------|----------------|--------------------------|----------------|-----------|--------------------|
| *Allium sativum*       | 3.2 (3.16–54.44)         | 2.7            | 2.2                      | 2.1            | 39.30     | $y = 0.86 + 0.06^\times$ |
| *Anethum graveolens*   | 19.90 (11.30–36.52)      | 1.9            | 1.8                      | 1.8            | 23.13     | $y = 1.23 + 0.06^\times$ |
| *Argania spinosa*      | 33.02 (22.75–55.92)      | 1.1            | 1.1                      | 1.1            | 13.91     | $y = 1.31 + 0.04^\times$ |
| *Boswellia serrata*    | 20.78 (12.05–37.26)      | 1.8            | 1.7                      | 1.7            | 22.42     | $y = 1.27 + 0.06^\times$ |
| *Brassica carinata*    | 32.09 (21.04–59.25)      | 1.2            | 1.1                      | 1.1            | 17.05     | $y = 1.33 + 0.04^\times$ |
| *Camellia sinensis*    | 13.02 (3.56–56.12)       | 2.9            | 2.5                      | 2.4            | 40.31     | $y = 0.96 + 0.07^\times$ |
| *Cedrus libani A*      | 26.87 (17.55–44.77)      | 1.4            | 1.3                      | 1.3            | 16.60     | $y = 1.24 + 0.05^\times$ |
| *Citrullus colocynthis*| 26.08 (12.80–65.61)      | 0.0            | 0.0                      | 0.0            | 32.23     | $y = 1.25 + 0.05^\times$ |
| *Crocus sativus*       | 37.07 (25.39–68.56)      | 1.0            | 1.0                      | 1.0            | 14.35     | $y = 1.41 + 0.04^\times$ |
| *Cucurbita maxima*     | 30.90 (22.00–47.60)      | 1.2            | 1.2                      | 1.2            | 12.91     | $y = 1.44 + 0.05^\times$ |
| *Camellia sinensis*    | 22.65 (13.54–140.07)     | 1.7            | 1.6                      | 1.6            | 22.68     | $y = 1.39 + 0.06^\times$ |
| *Capsicum annuum*      | 34.67 (26.87–47.96)      | 1.1            | 1.0                      | 1.0            | 18.16     | $y = 1.41 + 0.05^\times$ |
| *Crocus sativus*       | 20.49 (10.77–39.97)      | 1.8            | 1.6                      | 1.6            | 25.53     | $y = 1.14 + 0.05^\times$ |
| *Curcuma longa*        | 33.89 (24.46–52.94)      | 1.1            | 1.1                      | 1.1            | 11.35     | $y = 1.37 + 0.04^\times$ |
| *Foeniculum vulgare*   | 10.22 (5.29–21.14)       | 3.7            | 3.3                      | 3.3            | 21.56     | $y = 1.06 + 0.1^\times$ |
| *Galium verum*         | 27.64 (16.47–54.29)      | 1.4            | 1.3                      | 1.3            | 21.54     | $y = 1.2 + 0.04^\times$ |
| *Lepidium sativum*     | 20.06 (11.18–36.90)      | 1.9            | 1.7                      | 1.7            | 22.42     | $y = 1.11 + 0.05^\times$ |
| *Linum usitatissimum*  | 26.78 (12.80–77.92)      | 1.4            | 1.3                      | 1.3            | 31.75     | $y = 1.18 + 0.04^\times$ |
| *Malva sylvestris*     | 22.36 (9.11–58.90)       | 1.7            | 1.5                      | 1.5            | 36.44     | $y = 1.12 + 0.05^\times$ |
| *Nigella sativa*       | 15.67 (5.25–46.57)       | 2.4            | 2.1                      | 2.1            | 36.89     | $y = 1.01 + 0.06^\times$ |
| *Panax ginseng*        | 30.16 (19.05–57.39)      | 1.2            | 1.2                      | 1.2            | 18.86     | $y = 1.25 + 0.04^\times$ |
| *Piper nigrum*         | 20.14 (9.84–81.44)       | 1.9            | 1.6                      | 1.6            | 27.10     | $y = 1.07 + 0.05^\times$ |
| *Prunus dulcis*        | 26.75 (19.88–36.78)      | 2.6            | 1.4                      | 1.4            | 21.11     | $y = 1.2 + 0.04^\times$ |
| *Bauhinia variegata*   | 25.12 (14.60–50.27)      | 1.5            | 1.4                      | 1.4            | 24.68     | $y = 1.24 + 0.05$     |
| *Salvia officinalis*   | 15.42 (5.38–41.36)       | 2.4            | 2.1                      | 2.1            | 32.84     | $y = 0.89 + 0.06^\times$ |
| *Sesamum indicum*      | 37.64 (32.87–113.44)     | 1.0            | 1.0                      | 1.0            | 8.60      | $y = 1.54 + 0.04^\times$ |
| *Simmondsia chinensis* | 19.00 (14.03–25.19)      | 1.9            | 1.8                      | 1.8            | 4.20      | $y = 1.23 + 0.06^\times$ |
| *Soyuzgum aromaticum*  | 32.14 (21.00–44.85)      | 1.2            | 1.1                      | 1.1            | 16.81     | $y = 1.26 + 0.04^\times$ |
| *Tilia americana*      | 26.03 (19.61–33.79)      | 1.4            | 1.3                      | 1.3            | 16.6 (0.47a) | $y = 1.24 + 0.05^\times$ |
| *Thymus vulgaris*      | 9.67 (3.58–33.79)        | 3.9            | 3.2                      | 3.2            | 33.04     | $y = 0.88 + 0.09^\times$ |
| *Vicia faba*           | 10.31 (3.88–25.82)       | 3.6            | 3.2                      | 3.2            | 29.95     | $y = 0.96 + 0.09^\times$ |
| *Zingiber officinale*  | 29.27 (19.73–48.49)      | 1.3            | 1.2                      | 1.2            | 14.90     | $y = 1.26 + 0.04^\times$ |

*Reference oil: Sesamum indicum, Crocus sativus.*
Table 4. Kruskal–Wallis test for larval mosquito mortality (%) of plant oil groups at 1000 ppm. *Means produced by non-parametric analysis (Kruskal–Wallis, p 0.05). **The X^2 value is sig. at significant level 1% H: The highly effective group (95–100% mortality) are 8 oils (A. sativum, A. graveolens, C. sinensis, F. vulgaris, N. sativa, S. officinalis, T. vulgaris, and V. odorata). M: The moderately effective group (81–92% mortalities) are 7 oils (B. serrata, C. cymimum, C. aromatic, L. sativum, M. alternifolia, P. nigrum, and C. officinalis). L: The moderately effective group are included the rest of oils, 17 oils (A. spinosa, B. carinata, C. libani, C. colocynthis, C. sativus, C. maxima, C. sempervirens, C. longa, G. morhua, L. usitatissimum, P. ginseng, P. dulcis, R. chalepensis, S. indicum, S. aromaticum, T. americana, and Z. officinale).

| Oil groups | Mortality % (mean ± SD)* | 0.5 h | 2 h | 8 h | 24 h | 48 h |
|------------|--------------------------|-------|-----|-----|------|------|
| Low        | 4.2 ± 0.847              | 12.3 ± 2.278 | 25.980 ± 6.590 | 49.4 ± 7.838 | 71.6 ± 7.39 |
| Medium     | 5.0 ± 1.361              | 13.8 ± 4.050 | 35.950 ± 2.864 | 69.5 ± 2.841 | 88.3 ± 3.191 |
| High       | 7.5 ± 1.260              | 22.7 ± 1.527 | 54.792 ± 6.389 | 87.1 ± 8.533 | 98.3 ± 1.992 |
| Chi-Square | 16.909**                 | 18.152** | 23.037** | 25.391** | 25.098** |
| df         | 2                        | 2       | 2    | 2    | 2    |
| Asymp. Sig | 0.001                    | 0.001   | 0.001 | 0.001 | 0.001 |

Table 5. Friedman test for larval mosquito mortality (%) of plant oil groups at 1000 ppm. **The X^2 value is sig. at significant level 1%

### Table 5

| Oil groups | 0.5 h | 2 h | 8 h | 24 h | 48 h | Chi^2 | Df = 4 |
|------------|-------|-----|-----|------|------|-------|--------|
| Low        | 4.2 ± 0.847 | 12.3 ± 2.278 | 25.980 ± 6.590 | 49.4 ± 7.838 | 71.6 ± 7.39 | 68** |
| Medium     | 5.0 ± 1.361 | 13.8 ± 4.050 | 35.950 ± 2.864 | 69.5 ± 2.841 | 88.3 ± 3.191 | 28** |
| High       | 7.5 ± 1.260 | 22.7 ± 1.527 | 54.792 ± 6.389 | 87.1 ± 8.533 | 98.3 ± 1.992 | 31.7** |
| total      | 5.21 ± 1.735 | 15.21 ± 5.111 | 35.36 ± 1.337 | 63.23 ± 1.761 | 81.93 ± 1.992 | 127.6** |

### Discussion

EOs could serve as suitable alternatives to synthetic insecticides because they are relatively safe, available, and biodegradable\(^{35}\). In this study, 32 oils were evaluated against Cx. pipiens. *Thymus vulgaris* and *C. sinensis* were the most effective larvicides (100% mortality 24 h PT). The larvicidal effect of the H group could be arranged according to their LT_{50} values (h) as follows: *T. vulgaris* (9.67), *F. vulgaris* (10.22), *V. odorata* (10.31), *C. sinensis* (13.02), *A. sativum* (13.95), *S. officinalis* (15.42). *N. sativa* (15.67), then *An. graveolens* (19.90). On the other hand, their LT_{50} values ranged from 29.77 (F. vulgaris) to 55.31 (An. graveolens).

In this study, the most effective oils against adults were *An. graveolens* and *V. odorata* followed by *F. vulgaris* then *N. sativa*. The data revealed that *F. vulgaris* is a highly potent larvicide. Similarly, its oil controlled *Anopheles arabiensis*, *Culex quinquefasciatus*\(^{28,29}\), and *Aedes aegypti*\(^{30}\). Despite its effectiveness as larvicide in this study, *F. vulgaris* was the least effective adulticide. In contrast, it induced adulticidal properties against *Cx. quinquefasciatus*\(^{21}\).

Our data indicated that *C. sinensis* was a highly effective larvicide and the less effective adulticide. Comparatively, the chemical extracts of *C. sinensis* induced larvicidal and adult repellent effects against *C. pipiens* providing the highest protection (100%) from the bites of starved females at the dose of 6 mg/cm\(^2\)\(^{29}\). Moreover, its leaf extract showed larvicidal effect against *Anopheles arabiensis* and *Anopheles gambiae* (s.s.)\(^{31}\).

*Thymus vulgaris* *An. graveolens* showed potent larvicidal and adulticidal effects in this work. Likewise, *T. vulgaris* has both effects against *Cx. quinquefasciatus*\(^{28,29}\) and *A. aegypti*\(^{30}\). *Thymus vulgaris* exhibited larvicidal properties, 100% mortality, against *Cx. pipiens* larvae, at 200 ppm, whereas the LC_{50} and LC_{90} values indicated no effect on AChE activity, activation of the detoxification system, as indicated by an increase in GST activity and a decrease in GSH rate\(^{30}\).

Our findings agree with another study found that the most potent EOs out of 53 oils against larvae were *F. vulgaris*, *T. vulgaris*, *Citrus medica* (lime), and *C. sinensis* (LC_{50} = 27.5, 31.6, 51.3, 53.5 ppm, respectively). *C. sinensis* was the most efficient EOs enhancing the efficacy of deltamethrin, co-toxic factor = 316.67, over than PBO, the positive control, co-toxic factor = 283.35\(^{33}\).

Some oils applied in this study showed a similar larvicidal effect against *C. pipiens* as *N. sativa*\(^{34,35}\) and *S. officinalis*\(^{36}\). Some essential oils such as *T. vulgaris*, *S. officinalis*, *C. sempervirens* and *A. graveolens* had a larvicidal effect against mosquito larvae and their LC_{50} values were < 200–300 ppm. This result may be due to several
| Oil name         | Conc. % | Mortality% (mean ± SD) | LC50 (lower-upper limit) RE (LC50) | LC90 (lower-upper limit) RE (LC90) | LC95 (lower-upper limit) RE (LC95) | Chi (Sig) | Equation     |
|------------------|---------|------------------------|-----------------------------------|-----------------------------------|-----------------------------------|----------|--------------|
| *Allium sativum* | 0       | 0 ± 0e                 | 15.57 (8.49–28.46) 2.4            | 38.86 (26.79–61.87) 1.9           | 45.47 (31.19–97.80) 1.9           | 24.40 (0.000a) | Y = 0.051 + 0.008*x |
|                  | 0.5     | 20.00 ± 6.67d          |                                   |                                   |                                   |          |              |
|                  | 2.0     | 24.44 ± 5.88d          |                                   |                                   |                                   |          |              |
|                  | 5.0     | 42.22 ± 2.22c          |                                   |                                   |                                   |          |              |
|                  | 10      | 48.89 ± 4.44c          |                                   |                                   |                                   |          |              |
|                  | 20      | 62.22 ± 8.01b          |                                   |                                   |                                   |          |              |
|                  | 40      | 86.67 ± 3.85a          |                                   |                                   |                                   |          |              |
| *Anethum graveolens* | 0  | 6.37 ± 18.75d          | 2.42 (0.08–4.22) 8.05             | 9.47 (4.66–17.80) 5.4             | 23.25 (7.17–129.13) 2.6           | 33.254 (.000a) | Y = 0.242 + 0.130*x |
|                  | 0.1    | 36.86 ± 15.46b         |                                   |                                   |                                   |          |              |
|                  | 0.5    | 41.66 ± 27.57b         |                                   |                                   |                                   |          |              |
|                  | 2      | 46.12 ± 11.77b         |                                   |                                   |                                   |          |              |
|                  | 5      | 75.96 ± 18.84a         |                                   |                                   |                                   |          |              |
|                  | 10     | 88.39 ± 7.27a          |                                   |                                   |                                   |          |              |
|                  | 20     | 91.85 ± 9.24a          |                                   |                                   |                                   |          |              |
|                  | 25     | 100.00 ± 0.00a         |                                   |                                   |                                   |          |              |
| *Camellia sinensis* | 0  | 3.57 ± 20.00c          | 9.01 (−17.75 to 23.09) 2.3        | 32.18 (19.96–170.57) 1.6           | 38.754 (24.052–218.98) 1.5       | 26.52 (0.000a) | Y = 0.644 + 0.106*x |
|                  | 0.1    | 51.51 ± 2.62b          |                                   |                                   |                                   |          |              |
|                  | 0.5    | 61.21 ± 6.30ab         |                                   |                                   |                                   |          |              |
|                  | 2      | 63.94 ± 10.22ab        |                                   |                                   |                                   |          |              |
|                  | 5      | 75.35 ± 29.22ab        |                                   |                                   |                                   |          |              |
|                  | 10     | 78.78 ± 16.87ab        |                                   |                                   |                                   |          |              |
|                  | 25     | 91.99 ± 0.45a          |                                   |                                   |                                   |          |              |
| *Foeniculum vulgare* | 0  | 10.50 ± 25.00d         | 15.07 (0.10–104.60) 1.4           | 33.34 (21.67–789.17) 1.5           | 38.53 (24.63–986.39) 1.5          | 22.19 (0.000a) | Y = 0.331 + 0.03*x |
|                  | 0.05   | 36.73 ± 16.93bc        |                                   |                                   |                                   |          |              |
|                  | 0.1    | 51.54 ± 11.47ab        |                                   |                                   |                                   |          |              |
|                  | 0.5    | 51.70 ± 2.27bc         |                                   |                                   |                                   |          |              |
|                  | 2      | 59.00 ± 16.87ab        |                                   |                                   |                                   |          |              |
|                  | 5      | 75.96 ± 1.36a          |                                   |                                   |                                   |          |              |
|                  | 10     | 36.73 ± 16.93bc        |                                   |                                   |                                   |          |              |
|                  | 15     | 51.54 ± 11.47ab        |                                   |                                   |                                   |          |              |
|                  | 20     | 59.00 ± 16.87ab        |                                   |                                   |                                   |          |              |
|                  | 25     | 75.96 ± 1.36a          |                                   |                                   |                                   |          |              |
| *Nigella sativa*  | 0      | 4.95 ± 20.61e          | 3.42 (−53.96 to 30.15) 6.0        | 5.44 (−14.41 to 84.13) 9.3        | 29.95 (15.87–1184.48) 3.2         | 57.88 (0.000a) | Y = 0.261 + 0.06*x |
|                  | 0.05   | 41.87 ± 12.75 cd       |                                   |                                   |                                   |          |              |
|                  | 0.1    | 60.68 ± 3.73bc         |                                   |                                   |                                   |          |              |
|                  | 0.5    | 72.91 ± 6.45ab         |                                   |                                   |                                   |          |              |
|                  | 1      | 74.54 ± 19.76ab        |                                   |                                   |                                   |          |              |
|                  | 2      | 78.09 ± 18.26ab        |                                   |                                   |                                   |          |              |
|                  | 10     | 92.96 ± 9.44ab         |                                   |                                   |                                   |          |              |
|                  | 25     | 91.99 ± 0.45a          |                                   |                                   |                                   |          |              |
| *Salvia officinalis* | 0  | 4 ± 0e                 | 20.46 (11.34–45.85) 1.0           | 50.76 (33.24–140.52) 1.0           | 59.35 (38.59–168.23) 1.0          | 25.35 (0.000a) | Y = 0.8022 + 0.091*x |
|                  | 0.5    | 17.78 ± 2.22d          |                                   |                                   |                                   |          |              |
|                  | 2.0    | 22.22 ± 2.22d          |                                   |                                   |                                   |          |              |
|                  | 5.0    | 37.78 ± 4.45c          |                                   |                                   |                                   |          |              |
|                  | 10     | 44.44 ± 4.44bc         |                                   |                                   |                                   |          |              |
|                  | 20     | 53.33 ± 3.85b          |                                   |                                   |                                   |          |              |
|                  | 40     | 73.33 ± 7.70a          |                                   |                                   |                                   |          |              |
| *Thymus vulgaris* | 0      | 3.57 ± 7.15c           | 3.08 (−3.29 to 7.48) 6.6           | 16.08 (10.43–41.60) 3.2           | 19.76 (12.83–52.76) 3.0           | 34.12 (0.000a) | Y = 0.350 + 0.091*x |
|                  | 0.1    | 38.74 ± 4.28b          |                                   |                                   |                                   |          |              |
|                  | 0.5    | 61.66 ± 7.26ab         |                                   |                                   |                                   |          |              |
|                  | 2      | 69.82 ± 9.85ab         |                                   |                                   |                                   |          |              |
|                  | 10     | 72.22 ± 14.69ab        |                                   |                                   |                                   |          |              |
|                  | 20     | 100.00 ± 0.00a         |                                   |                                   |                                   |          |              |

Continued
reasons, including the percentages of their principal components compositions that are manipulated according to the origin of plant oil, quality of oil, susceptibility of the strain used, oil storage conditions, and technical conditions.

Likewise our findings, *An. graveolens* and *F. vulgare* act as larvicidal, pupicidal, and oviposition deterrent agents against *M. domestica*. Moreover, *Ocimum basilicum* was the most effective extract tested on *Cx. pipiens* larvae and adults. *Argania spinosa* oil showed a low larvicidal effect in this study. A similar effect was recorded against *Cx. quinquefasciatus* larvae.

| Oil name          | Conc. % | Mortality% (mean ± SD) | LC50 (lower-upper limit) RE (LC50) | LC90 (lower-upper limit) RE (LC90) | LC95 (lower-upper limit) RE (LC95) | Chi (Sig) | Equation     |
|-------------------|---------|------------------------|------------------------------------|------------------------------------|------------------------------------|-----------|--------------|
| Viola odorata     | 0       | 3.57 ± 7.15d           | 10.8                               | 7.37 (4.46–29.82)                  | 6.9                                | 8.92 (5.43–37.58) | 6.6         | 21.99 (0.001a) | Y = 0.190 + 0.112*x |
|                   | 0.1     | 50.00 ± 10.00c         |                                    |                                    |                                    |           |              |              |              |
|                   | 0.5     | 54.95 ± 15.61c         |                                    |                                    |                                    |           |              |              |              |
|                   | 1       | 57.50 ± 19.20c         |                                    |                                    |                                    |           |              |              |              |
|                   | 2       | 65.83 ± 15.21bc        |                                    |                                    |                                    |           |              |              |              |
|                   | 6       | 85.05 ± 13.62ab        |                                    |                                    |                                    |           |              |              |              |
|                   | 10      | 100.00 ± 0.00a         |                                    |                                    |                                    |           |              |              |              |
| Reference oils    |         | Salvia officinalis     |                                    |                                    |                                    |           |              |              |              |

Table 6. The adulticidal effects of selected plant oils against *Culex pipiens* after 24 h post-treatments.

| Peak no. | R (min.) | MW | MF | Area % | Probabilities of the detected compounds |
|----------|----------|----|----|--------|----------------------------------------|
| 1        | 5.03     | 138 | C2H4 | 0.41 | 1-Pyrrolidine                           |
| 2        | 5.22     | 138 | C7H10N2O | 0.26 | 2,3,3a,4,7,7a-Hexahydro-1H-benzimidazol-2-one |
| 3        | 5.28     | 348 | C19H22ClN2O | 1.06 | 1-Chloro-3-(3-fluorobenzyl)-4-(2-(diethylamino)ethylamino)benzene |
| 4        | 6.38     | 136 | C10H16 | 0.41 | Saline                                |
| 5        | 6.49     | 262 | C12H23O4 | 1.01 | Dimethyl[2,2-dimethyl-3-(2′-methylpropyl)cylopropyl][methyl]phosphonate |
| 6        | 7.57     | 670 | C44H27D3N4I | 0.15 | 5,10,15,20-tetraphenyl[2-(2H)1]porphyrin-atozinc(II) |
| 7        | 9.17     | 136 | C10H16 | 8.96 | Limonene                              |
| 8        | 10.90    | 152 | C10H16O | 2.81 | 1,3,3-trimethyl Bicyclo[2.2.1]heptan-2-one |
| 9        | 14.26    | 148 | C10H12O | 70.36 | Estragole                             |
| 10       | 14.72    | 818 | C44H28Br2N4Ti | 0.11 | Tetraphenylporphyrinodibromotitanium IV |
| 11       | 16.70    | 166 | C11H18O | 0.47 | 3,7-Dimethyl-2,6-Nonadienal            |
| 12       | 17.28    | 152 | C10H16O | 1.41 | 2,4-Decadienal                        |
| 13       | 18.07    | 194 | C14H26 | 0.17 | 1′,1′-Bicycloheptyl                   |
| 14       | 21.00    | 152 | C18H32O2 | 0.20 | Tetradecanoic acid, trimethylsilyl ester |
| 15       | 21.69    | 160 | C10H21F | 0.15 | Fluoro decane                         |
| 16       | 23.36    | 244 | C13H24O4 | 0.11 | Oxalic acid isohexylpentyl ester      |
| 17       | 25.03    | 328 | C19H40O2Si | 1.74 | Hexadecanoic acid, trimethylsilyl ester |
| 18       | 25.78    | 282 | C18H34O2 | 0.15 | (Z)-9-Octadecenoic acid               |
| 19       | 30.03    | 138 | C10H18 | 0.25 | 7-Methyl-1-nonyne                     |
| 20       | 30.12    | 282 | C18H34O2 | 0.30 | (Z)-9-Octadecenoic acid               |
| 21       | 30.58    | 256 | C16H32O2 | 0.12 | Hexadecanoic acid                     |
| 22       | 31.57    | 280 | C18H32O2 | 1.44 | (Z,Z)-9,12-Octadecadienoic acid       |
| 23       | 31.64    | 280 | C18H32O2 | 1.03 | (Z,Z)-9,12-Octadecadienoic acid       |
| 24       | 31.70    | 356 | C21H40O4 | 0.53 | 2,3-Dihydroxypropyldiide              |
| 25       | 31.76    | 238 | C16H30O | 1.67 | Z-7-Octadecenial                      |
| 26       | 32.25    | 280 | C18H32O2 | 0.23 | (Z,Z)-9,12-Octadecadienoic acid       |
| 27       | 32.38    | 266 | C18H34O2 | 0.43 | 12-Octadecenal                        |
| 28       | 32.83    | 142 | C9H18O | 0.13 | Nonanal                               |
| 29       | 46.93    | 660 | C20C12I2 | 0.13 | Dodecachloroperylene                  |
| 30       | 48.70    | 295 | C20H25NO | 0.61 | (R)-1-[(N-1-cyclopentylpropionylamino-1-ethyl)naphthalene |
| 31       | 50.05    | 354 | C20H18O6 | 0.38 | Isoesasamin                           |

Table 7. GC/MS analysis of the *Foeniculum vulgare* Mill.
**Figure 1.** GC/MS analysis of the *Foeniculum vulgare* Mill.

Table 8. GC/MS analysis of the *Anethum graveolens* L.

| Peak no. | R_t (min.) | MW | MF | Area %  | Probabilities of the detected compounds                                                                 |
|---------|------------|----|----|---------|--------------------------------------------------------------------------------------------------------|
| 1       | 5.14       | 238| C13H18O4 | 0.49    | Diethyl 3,4-bis(methylene)cyclopentane-1,1-dicarboxylate                                                   |
| 2       | 5.21       | 600| C33H28O11 | 0.69    | (2S,3S,3′,5′)-hydroxyanhydrophlegmacin-9,10-quinone 8′-O-methylether                                     |
| 3       | 7.65       | 290| C19H19O2  | 0.06    | 2-(2′-Isopropenyldec-2′-enyl)methylcyclopentane-1,3-dione                                               |
| 4       | 9.18       | 136| C10H16    | 17.19   | 1,5-Dimethyl-1,5-Cyclooctadiene                                                                         |
| 5       | 9.35       | 136| C10H16    | 0.23    | α-s-Limonene                                                                                           |
| 6       | 14.05      | 152| C10H16O   | 5.98    | Dihydrocarvone                                                                                        |
| 7       | 14.25      | 150| C10H14O   | 14.62   | 2-Methyl-5-(1-methylethenyl)2-Cyclohexen-1-one                                                         |
| 8       | 15.80      | 733| C44H28Cl2N4V  | 0.07 | Dichloro(5,10,15,20-tetra phenylporphyrinato)vanadium                                                  |
| 9       | 16.71      | 692| C41H33FeO5P | 0.13    | Dicarbonyl(1,3-5-α-6-phenyl-2-(phenylethyl)cyclohept-4-ene-1,3-diyl)triphenoxyphosphaneiron             |
| 10      | 17.29      | 110| C8H14     | 0.47    | octahydro Pentalene                                                                                   |
| 11      | 18.89      | 675| C44H28CuN4 | 0.09    | (5,10,15,20-tetraphenyl[2-(2H1)]prophyrinato)copper(II)                                                |
| 12      | 20.82      | 204| C15H24    | 0.10    | α-Humulene                                                                                             |
| 13      | 21.36      | 686| C37H24Cl2N6O4 | 0.08 | 2,2-Bis[4-(4-chloro-6-(3-ethynylphenoxy)-1,3,5-triazin-2-yl]oxy]phenyl]propane                         |
| 14      | 21.92      | 134| C10H14    | 0.14    | 1,2,3,4-Tetramethyl-5-methylene cyclopenta-1,3-diene                                                    |
| 15      | 22.07      | 204| C15H24    | 0.38    | α-Bisabolene                                                                                            |
| 16      | 22.16      | 648| C35H38Cl2N4O4 | 0.11 | 2,4-bis(α-chloroethyl)-6,7-bis(α-methoxycarbonyylethyl)-1,3,5-trimethylporphyrin                       |
| 17      | 22.36      | 640| C32H26O5Ss | 0.23 | OTETRAKIS(TRIMETHYLSILYL)5,5-DIHYDROXY-2-(3-HYDROXY-1-OCTENYL)CYCLOPENTANEHEPTANOATE | |
| 18      | 23.34      | 208| C14H24O   | 0.18    | 3-Oxacycloc[3.3.1]non-6-ene                                                                          |
| 19      | 24.23      | 222| C15H26O   | 21.26   | 3a(1H)-Azulenol,3,3,4,5,6,8a-hexahydro-6,8-adimethyl-3-(1-methylethenyl)[3R-(3α,3a,8ai)]               |
| 20      | 24.57      | 572| C23H26Br2O2  | 0.10 | Dibromogomisin A                                                                                      |
| 21      | 25.05      | 222| C10H14N4S  | 32.13   | 4-Pyrindinecaldehyde-4-propyl-3-thiosemicarbazone                                                     |
| 22      | 25.28      | 238| C15H26O2  | 2.39    | Daucoel                                                                                                |
| 23      | 26.01      | 194| C12H18O2  | 0.06    | 3-(1-Hydroxyhexyl)phenol                                                                              |
| 24      | 27.54      | 220| C15H24O   | 0.06    | Trans-Z-α-Bisaboleneoxide                                                                             |
| 25      | 33.01      | 2598| N/A | 0.07    | YGRKKRRQRRRQPVKRRRD/5                                                                                |
| 26      | 34.16      | 691| C51H33NO2 | 0.07    | 2,6-Bis(2,3,5-triphenyl-4-oxocyclo pentadienyl)pyridine                                                |
| 27      | 35.47      | 733| C44H28Cl2N4V | 0.08 | Dichloro(5,10,15,20-tetraphenylporphyrinato)vanadium                                                  |
| 28      | 40.31      | 739| C39H81NO4Si4 | 0.13 | (3S,4R,1E,2″R,3″R)-1-tertButyldimethylsilyl4-(3″-tertbutyldimethylsiloxoy-2′-methylprop-1′-enyl)-3-(1″,3″ di(tertbutyldimethylsiloxoy)-2′-methylhex-5″-yl]-2-methylazetidin-2-one |
| 29      | 41.48      | 114| C6H10O2   | 0.13    | 3,4-Hexanedione                                                                                       |
| 30      | 50.56      | 680| C35H40OS6s5 | 0.06 | Pentamethylpentaphenylcyclopentasiloxane                                                               |
| 31      | 51.11      | 733| C44H28Cl2N4V | 0.09 | Dichloro(5,10,15,20-tetraphenylporphyrinato)vanadium                                                  |
Curcuma species was less effective in this study, but its 27 components as curcuminoids and monocarbonyl curcumin derivatives were effective larvicidal agents against Cx. Pipiens and Ae. albopictus.

Hexane extraction of Curcuma longa showed 100% larvicidal activity against Cx. pipiens and Aedes albopictus at 1000 ppm after being treated 24 h. Zingiber officinale and Syzygium aromaticum were less effective. In contrast, they were effective against Cx. pipiens (LC50 = as 71.85 and 30.75, respectively). Sesamum indicum is one of the L group in this study. In contrast, petroleum ether extract showed larvicidal, antifeedant and repellent action against Cx. pipiens. Furthermore, EOs of N. sativa, Allium cepa, and S. indicum, induced larvicidal effect and their LC50 values against both field and laboratory strains of Cx. pipiens were 247.99 and 108.63; 32.11 and 2.87; and finally, 673.22 and 143.87 ppm, respectively. They influenced the pupation and adult emergence rates besides developmental abnormalities at sublethal concentrations.

Boswellia serrata (M group) and Brassica carinata (L group) showed relative larvicide against Cx. pipiens in this study. A similar result was reported. The lethal concentration values of Fenugreek (Trigonella foenum-graecum), earth almond (Cyperus esculentus), mustard (Brassica compestris), olibanum (Boswellia serrata), rocket (Erba sativa), and parsley (Carum ptroselinum) were 32.42, 47.17, 71.37, and 83.36, 86.06, and 152.94 ppm.

Table 9. GC/MS analysis of the Viola odorata L.

| Peak no. | R_t (min.) | MW | MF | Area % | Probabilities of the detected compounds |
|----------|------------|----|----|--------|----------------------------------------|
| 1        | 23.923     | 170 | C12H10O 42.04 | Diphenyl ether |
| 2        | 24.735     | 192 | C13H20O 11.87 | alpha-Ionone |
| 3        | 26.485     | 192 | C13H20O 7.73 | 3-Buten-2-one, 4-(2,6,6-trimethyl-1-cyclohexen-1-yl) |
| 4        | 28.317     | 236 | C15H24O2 0.61 | Limonen-6-ol, pivalate |
| 5        | 28.58      | 226 | C13H22O3 0.9 | 2-Hydroxy-1,1,10-trimethyl-6,9-epidioxydecalin |
| 6        | 28.786     | 238 | C16H30O 1.26 | 7-Hexadecenal, (Z)- |
| 7        | 29.599     | 236 | C16H28O 0.83 | 7,11-Hexadecadienal |
| 8        | 29.713     | 296 | C20H40O 1.48 | Phytole |
| 9        | 29.959     | 242 | C16H34O 2.15 | 2-Hexyl-1-Decanol |
| 10       | 30.074     | 378 | C25H46O2 1.09 | Undec-10-yneic acid, tetradecyl ester |
| 11       | 30.211     | 296 | C20H40O 1.02 | PHYTOL ISOMER |
| 12       | 30.881     | 266 | C16H26O3 0.67 | 2-Dodecen-1-yl(-)-succinic anhydride |
| 13       | 31.338     | 242 | C16H34O 2.14 | 1-Decanol, 2-hexyl- |
| 14       | 31.939     | 218 | C16H26 2.79 | HexadecahydroPyrene |
| 15       | 32.054     | 240 | C17H36 0.7 | Tetradecane, 2,6,10-trimethyl |
| 16       | 34.245     | 250 | C16H26O2 7.22 | (Z)-5-(4-tert-Butyl-1-hydroxy-cyclohexyl)-3-methylpent-2-en-4-yl |
| 17       | 35.092     | 264 | C15H20O4 6.6 | 2,3,3a,4,5,6,7,9,9b-decahydro-3,5a,9-trimethyl-7,9a-peroxy Naphtho[1,2-b]furan-2-one |
| 18       | 35.269     | 264 | C15H20O4 4.73 | 2,3,3a,4,5,6,7,9,9b-decahydro-3,5a,9-trimethyl-7,9a-peroxy Naphtho[1,2-b]furan-2-one |
| 19       | 35.905     | 242 | C16H34O 2.19 | 2-hexyl-1-Decanol |
| 20       | 37.146     | 266 | C18H34O 1.89 | Z=E-2,13-Octadecadien-1-ol |
| 21       | 23.923     | 170 | C12H10O 0.78 | Diphenyl ether |
respectively. Against Cx. pipiens larvae. Furthermore, increasing concentrations were directly proportional to the reduction of both pupation and adult emergences rates48.

Some oil-resins as Commiphora molmol, Araucaria heterophylla, Eucalyptus camaldulensis, Pistacia lentiscus, and Boswellia sacra showed larvicidal activity against Cx pipiens larvae. The larvicidal effect 24 and 48 h PT, respectively, were for acetone extracts, 1500 ppm, of C. molmol (83.3% and 100% and LC50 = 623.52 and 300.63 ppm) and A. heterophylla (75% and 95% and LC50 = 826.03 and 384.71 ppm). On the other hand, the aqueous extract of A. heterophylla induced higher mortalities (LC50 = 2819.85 ppm and 1652.50 ppm), followed by C. molmol, (LC50 = 3178.22 and 2322.53 ppm)49.

A similar larvicidal effect was recorded for Rosmarinus officinalis, hexane extract (80 and 160 ppm), reduced 100% mortality against 3rd and 4th instars larvae of Cx. pipiens and the toxicity increased in the pupal and adult stages50.

Out of 36 essential oils, red moor besom leaf oil has strong fumigation activity against Cx. pipiens pallens adults51. Similar to the adulticidal effect of the applied oils in this work, some other oils have adulticidal activities against mosquitoes as Cedrus deodara, Eucalyptus citriodora, Cymbopogon flexuosus, Cymbopogon winterianus, Pinus roxburghii, S. aromaticum, and Tugtes minata52. The Leaf Oils of Cinnamonum species had adulticidal activities against Ae. aegypti and Aedes albopictus53. EOs have adulticidal effects against Musca domestica54 as A. sativum, S. aromaticum, and F. vulgare55. Essential oils of Melaleuca leucadendron (L.) and Callistemon citrinus (Curtil) showed 100% adult mortality against Aedes aegypti (L.) and Cx. quinquefasciatus (Say), 24 h exposure56.

The results showed that A. sativum, and S. officinalis oils were effective against mosquito larvae, maybe due to the presence of a number of active secondary compounds such as ISOCHIAPIN B<sub>2</sub> (&lt; sesquiterpene lactone) and 9-Octadecenamide, (Z)-that are anti-inflammatory activity57, also, Terpinen-4-ol and Camphor in Sage oil that these are excellent natural insecticide58, but these oils garlic and Sage did not show the required efficacy against adult mosquitoes.

The phytochemical analysis of this study revealed the major activated compounds of the analyzed oils. Green tea oil is a highly effective larvicide in this study contains a high amount of polyphenols that have antioxidant activity. A similar finding was reported59. Our data indicated that green tea oil also contains polyphenols as Gallic acid, Catechin, Methyl gallate, Coffeic acid, Coumaric acid, Naringenin, and Kaempferol which might aid in its insecticidal effect.

This study indicated that F. vulgare contains Estragole (70.36%) and Limonene (8.96%). Similarly, Limonene as a cyclic monoterpene has a viable insecticidal effect60. Besides, Estragole induced toxicity to adult fruit flies, Ceratitis capitata61. Moreover, An. graveolens contains thiosemicarbazone (32.13%) in this study. Likewise, thiosemicarbazide is a major component An. graveolens with insecticidal effect62. Also, Dauco and carotol are essential oils documented for An. graveolens in this work have repellent activity against adult Ae. aegypti, Ae. albopictus, and Anopheles quadrimaculatus Say63. Furthermore, V. odorata in the present analysis contains alphalone, which revealed anti-inflammatory and analgesic effects64. Thymus vulgaris showed good alpha-pinene and pyridine derivatives that play an important role as larvicidal and adulticidal effects against Ae. aegypti and growth regulator, respectively65,66. In addition, the combination of all constituents may promote their individual larvicidal and adulticidal effects.

The biochemical compositions showed that T. vulgaris oil affected the energy reserves with a marked effect on proteins and lipids67. The differences between our findings and those of the others could be attributed to the biological activities and the chemical composition for EOs, which could vary between plant age, tissues, geographical origin, the part used in the distillation process, distillation type, and the species. Therefore, types and levels of active constituents in each oil may be responsible for the variability in their potential against pests68.

**Conclusions**

Diseases transmitted by mosquitoes represent global concerns. Our findings demonstrate the potential of F. vulgare and C. sinensis as the most potent larvicides and N. sativa, V. odorata, and An. graveolens as the most effective adulticides as they contain good command of different essential oils. EOs could be used for integrated mosquito control programs as larvicides or synergists for enhancing the efficacy of current adulticides69.
| Peak no. | Rt (min.) | MW | MF          | Area % | Probabilities of the detected compounds |
|---------|-----------|----|-------------|--------|----------------------------------------|
| 1       | 5.1       | 208 | C13H20O2    | 0.86   | TRANS-á-IONON-5,6-EPOXIDE              |
| 2       | 5.23      | 122 | C8H15B      | 0.79   | 1-Borabicyclo[4.3.0]nonane             |
| 3       | 6.46      | 136 | C10H16      | 1.85   | Tricyclene                              |
| 4       | 6.86      | 136 | C10H16      | 0.69   | Camphene                                |
| 5       | 7.64      | 136 | C10H16      | 8.92   | 2-á-pinene                              |
| 6       | 9.07      | 119 | C7H5NO      | 12.37  | 2-Ethynyl-3-hydroxypropyridine         |
| 7       | 11.32     | 196 | C12H20O2    | 0.68   | Linallyl acetate                        |
| 8       | 12.50     | 152 | C10H16O     | 1.27   | (1S) Bicyclo[2.2.1]heptan-2-one, 1,7,7-trimethyl |
| 9       | 13.39     | 156 | C10H20O     | 0.78   | 1-Methyl-4-(1-methylthyl)cyclohexanol   |
| 10      | 13.51     | 154 | C10H18O     | 4.73   | 4-Methyl-1-(1-methylthyl)-3-cyclohexen-1-ol |
| 11      | 13.91     | 154 | C10H18O     | 1.13   | 4,4,4-trimethyl (S) 3-Cyclobexene-1-methanol |
| 12      | 15.67     | 182 | C11H18O2    | 0.63   | linallyl formmate                       |
| 13      | 16.48     | 196 | C12H20O2    | 1.76   | EXOBORNYL ACETATE                      |
| 14      | 18.17     | 196 | C12H20O2    | 5.00   | á-terpinyl acetate                     |
| 15      | 20.52     | 142 | C9H18O      | 0.56   | 3-Ethylheptanal                         |
| 16      | 21.94     | 268 | C19H40      | 0.58   | Nonadecane                              |
| 17      | 22.84     | 199 | C9H13NO4    | 1.87   | 2S,7S Methyl-2-Hydroxy-3-oxotetrahydro-1-Hpyrrolizine-7a-(5H)-carboxylate |
| 18      | 22.97     | 226 | C16H34      | 0.92   | Pentadecane-5-methyl                   |
| 19      | 23.10     | 212 | C15H32      | 0.75   | 3-ethyl Tridecane                       |
| 20      | 23.22     | 348 | C19H40O3S   | 0.84   | hexyltridecyl ester Sulfurous acid     |
| 21      | 23.39     | 226 | C16H34      | 1.09   | 3-methyl Pentadecane                   |
| 22      | 24.06     | 168 | C11H20O2    | 1.52   | 1,6-diisocyanato Hexane                 |
| 23      | 24.24     | 298 | C20H42O     | 3.26   | 1,1’-oxybis Decane,                     |
| 24      | 24.40     | 282 | C20H42      | 0.81   | Eicosane                                |
| 25      | 24.65     | 234 | C18H38O3S   | 0.57   | Sulfurous acid, butyltridecyl ester    |
| 26      | 25.10     | 282 | C20H42      | 4.12   | 10-Methylnonadecane                    |
| 27      | 25.24     | 268 | C19H40      | 1.00   | 7-heptyl Tridecane                     |
| 28      | 25.37     | 334 | C18H38O3S   | 1.10   | 6-Tetradecanolsulfonic acid, butyl ester|
| 29      | 25.49     | 334 | C18H38O3S   | 1.44   | 6-Tetradecanolsulfonic acid, butyl ester|
| 30      | 25.68     | 250 | C16H26O2    | 4.54   | 5-(6,6-Dimethyl-5-oxohept-2-enyl)-cycloheptanone |
| 31      | 25.98     | 222 | C13H18O     | 7.70   | 2,5-Dipropoxybenzaldehyde              |
| 32      | 26.30     | 352 | C25H52      | 1.33   | Pentacosane                             |
| 33      | 26.44     | 282 | C20H42      | 3.55   | 9-methyl Nonadecane                     |
| 34      | 26.62     | 224 | C16H32      | 1.08   | 1-Hexadecene                           |
| 35      | 26.84     | 236 | C16H28O     | 2.14   | 7,11-Hexadecadien                      |
| 36      | 27.25     | 232 | C11H12N4O2  | 5.05   | 5-Amino-8-cyano-7-methoxy-3,4-dihydro-3-methyl-1,1,6-naphthyridin-2(1H)-one |
| 37      | 27.32     | 232 | C15H20O2    | 2.01   | (2R,3R)-3-(2-Methoxy-4-methylphenyl)-2,3-dimethylocyclopentanone |
| 38      | 27.42     | 282 | C20H42      | 0.87   | 2,6-dimethyl Octadecane                |
| 39      | 27.54     | 310 | C22H46      | 0.77   | 8-heptyl Pentadecane                   |
| 40      | 27.65     | 376 | C21H44O3S   | 0.61   | Sulfurous acid, hexyl pentadecyl ester |
| 41      | 27.82     | 226 | C16H34      | 0.88   | Hexadecane                             |
| 42      | 28.42     | 348 | C5H9BO      | 0.62   | 1-Bromo-2-methyl-3-Buten-2-ol          |
| 43      | 28.54     | 242 | C16H34O     | 1.25   | 2-Hexyl-1-decanol                      |
| 44      | 28.69     | 111 | C7H13N      | 1.08   | 1-isocyano Hexane                      |
| 45      | 29.32     | 116 | C7H16O      | 1.94   | 2-ethyl 1-Pentanol                     |
| 46      | 30.70     | 200 | C13H28O     | 0.82   | 2-Propyldecan-1-ol                     |
| 47      | 31.33     | 197 | C11H19NO2   | 0.98   | 2-Ethylhexyl cyanoacetate              |
| 48      | 33.27     | 592 | C41H84O     | 0.70   | 1-Hentetacontanol                      |
| 49      | 36.28     | 324 | C23H48      | 0.57   | 9-hexyl Heptadecane                    |
| 50      | 37.92     | 366 | C26H54      | 0.58   | 5,14-dibutyl Octadecane                |

Table 10. GC/MS analysis of *Thymus vulgaris* L.
Figure 4. GC/MS analysis of *Thymus vulgaris* L.

| Peak no. | Rt (min.) | MW | MF | Area % | Probabilities of the detected compounds |
|----------|-----------|----|----|--------|----------------------------------------|
| 1        | 6.27      | 146 | C6H10S2 | 4.54 | Diallyl disulphide                     |
| 2        | 7.49      | 152 | C4H8S3 | 9.68 | Trisulphide, methyl 2-propenyl         |
| 3        | 9.35      | 178 | C6H10S3 | 14.86 | Trisulphide, di-2-propenyl            |
| 4        | 12.22     | 350 | C19H26O6 | 8.63 | ISOCHIAPIN B %2<                       |
| 5        | 14.97     | 334 | C20H30O4 | 3.54 | 1,2-Benzenedicarboxylic acid, butyl octyl ester |
| 6        | 16.05     | 346 | C19H22O6 | 3.11 | ISOCHIAPIN B                           |
| 7        | 17.67     | 387 | C17H37N7O3 | 7.84 | 9-OCTADECENAMIDE                       |
| 8        | 19.61     | 281 | C18H35NO | 29.07 | 9-Octadecanamide, (Z)-                |
| 9        | 21.40     | 208 | C11H12O2S | 4.25 | 3-(Benzylthio)acrylic acid, methyl ester |
| 10       | 23.27     | 436 | C26H44O5 | 1.82 | 3 Ethyl iso-allocholate               |
| 11       | 23.54     | 490 | C34H50O2 | 6.81 | CHOLEST-5-EN-3-YL BENZOATE            |

Table 11. GC/MS analysis of the *Allium sativum*. 9-Octadecanamide, (Z)- (29.07), Trisulfide, di-2-propenyl (14.86), and ISOCHIAPIN B %2< (8.63).

Figure 5. GC/MS analysis of *Allium sativum*. 
Table 12. GC/MS analysis of the *Salvia officinalis*.

| Peak no. | R<sub>t</sub> (min.) | MW  | MF    | Area % | Probabilities of the detected compounds                        |
|----------|---------------------|-----|-------|--------|---------------------------------------------------------------|
| 1        | 10.22               | 152 | C10H16O | 16.08  | Camphor                                                      |
| 2        | 10.90               | 156 | C10H20O | 5.24   | Cyclohexanol, 1-methyl-4-(1-methylethyl)                      |
| 3        | 11.47               | 154 | C10H18O | 17.35  | Terpinen-4-ol                                                |
| 4        | 13.86               | 254 | C13H24O2| 2.47   | Tridecanedioi                                               |
| 5        | 14.50               | 280 | C18H32O2| 3.43   | 17-Octadecynoic acid                                        |
| 6        | 15.70               | 400 | C28H48O | 0.90   | Cholestane-3-ol, 2-methylene-, (3α,5α)-                     |
| 7        | 16.68               | 268 | C17H32O2| 1.80   | 7-Methyl-Z-tetradec-1-ol acetate                             |
| 8        | 17.50               | 280 | C19H36O | 1.63   | 12-Methyl-E,E-2,13-octadecadien-1-ol                         |
| 9        | 17.99               | 288 | C21H36  | 2.03   | 14-á-H-PREGNA                                               |
| 10       | 19.18               | 288 | C18H37Cl| 5.13   | 1-CHLOROOCTADECANE                                          |
| 11       | 19.51               | 288 | C21H36  | 1.77   | 14-á-H-PREGNA                                               |
| 12       | 19.86               | 450 | C32H66  | 4.33   | DOTRIACONTANE                                               |
| 13       | 20.18               | 536 | C37H76O | 1.41   | 1-Heptatriacotanol                                           |
| 14       | 20.32               | 268 | C16H20O3| 1.15   | 7-(13,14-Epoxy)tetradec-11-en-1-ol acetate                   |
| 15       | 20.55               | 258 | C16H14S | 1.58   | tert-Hexadecanethiol                                        |
| 16       | 20.80               | 312 | C20H40O | 3.17   | Ethanol, 2-(9-octadecenyloxy)-, (Z)-                         |
| 17       | 20.90               | 288 | C21H36  | 2.18   | 14-á-H-PREGNA                                               |
| 18       | 21.26               | 350 | C19H26O6| 0.73   | ISOCHIAPIN B %2<                                             |
| 19       | 21.61               | 288 | C18H37Cl| 6.82   | 1-CHLOROOCTADECANE                                          |
| 20       | 21.84               | 294 | C21H36  | 3.7    | 14-á-H-PREGNA                                               |
| 21       | 22.39               | 288 | C21H36  | 0.82   | 1-Heptatriacotanol                                           |
| 22       | 22.47               | 346 | C19H22O3| 2.74   | ISOCHIAPIN B                                               |
| 23       | 22.73               | 288 | C21H36  | 9.25   | 14-á-H-PREGNA                                               |
| 24       | 23.09               | 280 | C19H36O | 2.20   | 12-Methyl-E,E-2,13-octadecadien-1-ol                         |
| 25       | 23.23               | 350 | C19H26O6| 2.05   | ISOCHIAPIN B %2<                                             |

Figure 6. GC/MS analysis of *Salvia officinalis*. 
studies are needed to develop nanoformulations that improve the efficacy and minimize applications after revealing their ecotoxicological side views.

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Conceptualization, A.A., A.M. and M.B.; methodology, H.K., M.B., I.R.; validation, M.B., I.R. and A.A.; formal analysis, A.A. and H.K.; resources, A.A.; writing—original draft preparation, M.B., I.R., H.K. and A.A.; writing—review and editing, H.K., A.A., A.M. and A.S.; supervision, H.K.; project administration, A.S.; funding acquisition, A.S. All authors have read and agreed to the published version of the manuscript.

Competing interests
The authors declare no competing interests.

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