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RESEARCH ARTICLE



# Efficacy of mosquito repellent finishes on polyester fabrics using four essential oils against the vector of the West Nile virus, *Culex pipiens* (Diptera: Culicidae)

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## ABSTRACT

One of the most common ways to prevent mosquito bites is to use treated textiles, which can be used to repel mosquitoes in the form of nets, clothing, uniforms, and other items. Four eco-friendly essential oils: lemongrass (*Cymbopogon citratus*), eucalyptus (*Eucalyptus camaldulensis*), clove (*Dianthus caryophyllus*), and marjoram (*Origanum vulgare*), were evaluated and used to finish polyester fabrics (PE) to give them a pleasant odor and antibacterial properties, as well as mosquito repellent as medical textiles. A low-temperature drying process and thermal stabilization technique were used to bond the polyester to the fragrance-containing compounds. The effect of essential oil finishes on polyester textile properties has been studied using various techniques such as mechanical measurements, contact angle, ultraviolet tests, infrared spectroscopy, Fourier transform infrared spectroscopy, scanning electron microscopy, GC-MS analysis, etc. Scanning electron microscopy showed the physical adsorption of oil on PE surfaces and the penetration of oil droplets between PE fibers. The ability of polyester textiles treated with essential oils of lemongrass, eucalyptus, clove, and marjoram to kill *Staphylococcus aureus*, *Escherichia coli*, *Candida albicans*, and *Aspergillus niger* was also tested. Polyester fabrics treated with clove showed the strongest antibacterial effect, surpassing that of eucalyptus oil. 12 essential oils were screened to determine their superiority in repelling mosquitoes at a concentration of 1% through the CDC bottle and WHO cone tests. *Cymbopogon citratus* (LT<sub>50</sub> = 60.03 min) oil was the most effective against *Cx. pipiens*, followed by *Eucalyptus camaldulensis* (LT<sub>50</sub> = 61.65 min) and *Dianthus caryophyllus* (LT<sub>50</sub> = 64.73 min) with CDC tests. It was found that finishing PE with clove and eucalyptus oils provided 76 and 68% protection from *Culex pipiens* mosquitoes, even after 6 cycles of washing. This approach is innovative because it meets the goal of having antibacterial, mosquito-repellent, and pleasant-smelling fabrics as requirements for economically viable medical textiles. There is an urgent need to engineer textiles, with or without insecticides, to effectively repel mosquitoes. Our data showed the efficiency of polyester fabric treated with clove and eucalyptus oils in repelling mosquitoes after 6 washings.

## ARTICLE HISTORY

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## KEYWORDS

Eco-friendly; essential oils; finishing polyester; medical textiles; *Culex pipiens*; washing

## 1. Introduction

Over the past few years, increased global trade, urbanization, and climate change have all contributed to global outbreaks of mosquito-borne diseases. Malaria, West Nile, yellow fever, dengue, chikungunya, and zika are the main mosquito-borne illnesses (Mosquito-Borne Diseases, 2022). More than 700,000 people die from these illnesses each year around the world, with malaria accounting for 400,000 of those deaths. In addition, the prevalence of dengue has increased 30-fold globally in the past 30 years, with an estimated 96 million cases per year and more than 3.9 billion people at high risk of developing dengue in more than 128 countries (Norris & Coats, 2017).

In general, there are two primary strategies for reducing the spread of mosquito-borne diseases. One method used is to target the disease agent. Some of these tactics include

preventive measures, vaccines, large-scale drug distribution campaigns, and antibiotic and antiviral treatments that specifically target the parasite or prevent the virus from replicating in the host (Tolle, 2009). The second method of control entails stopping the carrier from spreading the pathogen. Several methods are used to achieve this, including reducing vector numbers and using deterrents (Peter et al., 2005). Vector control has been a remarkably effective tool in preventing worldwide transmission of the mosquito-borne disease (Norris & Coats, 2017).

One of the most common strategies for preventing mosquitoes and other bloodsucking pests from biting is to expel or kill them in a safe and natural way by using essential oils. Insect repellents can be applied directly to the skin, clothing, or other items, such as mosquito bed nets and anti-mosquito screens. Commercially available mosquito

## Nomenclature

<i>C. citratus</i>	<i>Cymbopogon citratus</i>	<i>D. caryophyllus</i>	<i>Dianthus caryophyllus</i>
<i>Cx. pipiens</i>	<i>Culex pipiens</i>	GC-MS	Gas chromatography–mass spectrometry
<i>E. camaldulensis</i>	<i>Eucalyptus camaldulensis</i>	<i>O. vulgare</i>	<i>Origanum vulgare</i>
EDX	Energy dispersive X-ray spectroscopy	PE	Polyester fibers

coils, mats, and fumigators that contain synthetic pyrethroids such as pyrethrin, allethrin, and permethrin, as well as chemicals such as N,N-diethyl-m-toluamide (DEET), may have negative side effects when used for an extended period of time (Maguranyi et al., 2009). Mosquitoes may also become resistant to these chemicals. The synthetic and chemical insecticides used in commercial mosquito repellents are dissolved in deodorizing kerosene, which is also harmful to the user. The development of natural products for disease vector control has long attracted attention due to some negative aspects of synthetic (chemical) insecticides, such as lack of selectivity, environmental pollution, and the emergence and spread of vector resistance. Among the plants that possess pest control or repellent properties, approximately 300 plant species contain bioactive substances with mosquito repelling or lethal potential (Gunasekaran & Kalyanasundaram, 2008).

Products made from essential oils are gaining popularity as efficient, safe, and reasonably priced insect repellants as people look for alternate and safe ways to avoid mosquito bites (Mittal & Subbarao, 2003). Due to several factors, the use of repellents such as lotions, coils, and liquidators is restricted. The creation of materials that repel mosquitoes has become necessary as a result.

The effect of the natural (alcoholic) extract of mint leaves, stems, and garlic cloves as final mosquito repellent substances on woven fabric was examined. The study showed that concentrations of 5%, 15%, 25%, and 35% of the PGE solution (garlic and mint extract) affected the developed fabric, leading to the expulsion of *Aedes aegypti* mosquitoes. The results also showed that the tissue samples treated with PGE C (25% PGE) and D (35% PGE) had the highest mosquito mortality rates (50.00% and 76.67%, respectively) and repellency rates (78.6% and 85.6%, respectively) (Parvez et al., 2023).

In order to create mosquito repellent fabric, scientists work with natural substances such as mint leaves, castor oil, and fresh moringa leaf, neem and tulsi, sweet basil and eucalyptus, and marigold petals (Elsayed & Hassabo, 2021; Mia et al., 2020; Pavan et al., 2023). *Allium sativum*, or mint, has recently piqued the curiosity of researchers as a potential tick-repellent source, particularly in light of a study that found eating garlic shielded soldiers exposed to ticks from getting tick bites while on the front lines (Stjernberg & Berglund, 2000). According to the previous analysis of the literature, the majority of studies on mosquito-repellent fabrics substitute synthetic repellents with plant-based essential oils (Boominathan et al., 2021; Ho et al., 2019; Karthigeyan & Premalatha, 2019; Soroh et al., 2021).

Textile items with the ability to provide protection from something in some ways are referred to as protective textiles. Protective textiles, which aid in protection from species that are likely to cause damage, include finished fabrics that are mosquito repellent. One of the most innovative ways to enhance the textile industry is to use materials that repel mosquitoes, a trait that is desperately needed (Prabha & Vasugi, 2012).

The goal of this study was to find out if lemongrass (*Cymbopogon citratus*), river redgum (*Eucalyptus camaldulensis*), carnation flower (*Dianthus caryophyllus*), and oregano (*Origanum vulgare*) essential oils could keep mosquitoes away after being applied to polyester fabrics as textile finishes with essential oils. There hasn't been much research on these essential oils' ability to do this when applied to textile materials. The flow chart of the article summarizes the steps for our study, as shown in Figure 1.

## 2. Materials and methods

### 2.1. Chemicals and plant oils

Twelve natural plant essential oils were purchased from the Nefertari Company for Natural Plant Oils and Cosmetics (Cairo, Egypt). By using 100% pure and natural hydrodistillation, the oils were extracted from plants, as shown in Table 1. The de-ionized water, acetone, polysorbate 20, spam 80, and tween 80 were purchased from Alfa Esar (Thermo Fisher GmbH, Kandel, Germany) and used without further purification.

### 2.2. Textile measurements and analysis

#### 2.2.1. Mechanical measurements

The mechanical properties of untreated and treated silk fabrics, such as tensile strength and elongation, were tested using a Shimadzu Universal Tester of type S500-Japan. The estimations are carried out according to ASTM 2000, D 3822-96 standard test strategy for tensile properties of a single fabric (Indrie et al., 2022).

#### 2.2.2. Contact angle

The contact angle was measured using a dynamic contact angle tensiometer (SEO) model Phoenix 300 (Kromtech Alliance Corp., London, UK), with three phases (air/water/sample). The static contact angles were determined according to the Young-Laplace equation by observing sessile water droplets of 4.00  $\mu$ L deposited on the cotton fabric's surface. The contact angle values were collected on the drop

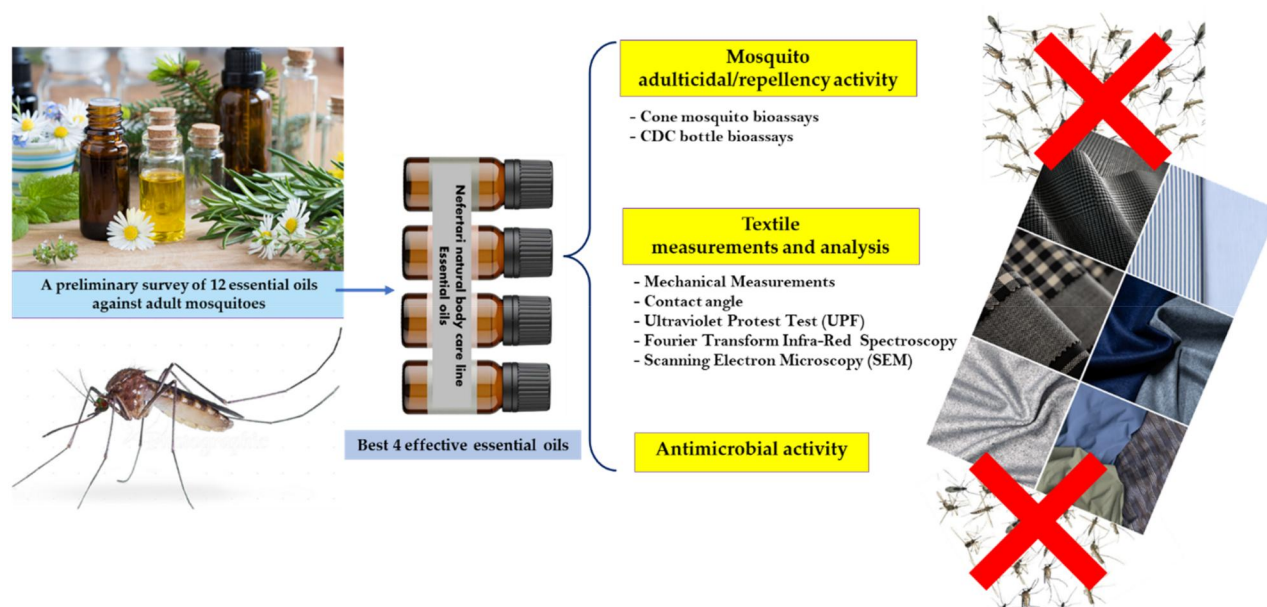


Figure 1. The flow chart of the article.

Table 1. List of plant oils tested against *Culex pipiens* adults.

No.	Oil name	Plant essential oils		
		Order	Family	Common name
1	<i>Rosa canina</i> L.	Rosales	Rosaceae	dog rose
2	<i>Origanum vulgare</i> L.	Lamiales	Lamiaceae	oregano/ marjoram
3	<i>Dianthus caryophyllus</i> L.	Caryophyllales	Caryophyllaceae	clove /carnation
4	<i>Cymbopogon citratus</i> D.	Poales	Poaceae	lemongrass
5	<i>Ruta chalepensis</i> L.	Sapindales	Rutaceae	fringed rue
6	<i>Cupressus sempervirens</i> L.	Pinales	Cupressaceae	italian cypress
7	<i>Camellia sinensis</i> L.	Ericales	Theaceae	tea
8	<i>Azadirachta indica</i> A.	Sapindales	Meliaceae	neem
9	<i>Melissa indicum</i> L.	Lamiales	Lamiaceae	common balm
10	<i>Pimpinella anisum</i> L.	Apiales	Apiaceae	anise burnet saxifrage
11	<i>Eucalyptus camaldulensis</i> D.	Myrtales	Myrtaceae	river redgum
12	<i>Pelargonium graveolens</i> L.	Geraniales	Geraniaceae	scented geranium

after 30 s of contact with the fabric's surface (Rohaeti & Rakhmawati, 2017).

### 2.2.3 Ultraviolet protest test (UPF)

A 2 cm<sup>2</sup> fabric was mounted under moderate tension at the transmittance port of the integrating sphere. The spectra of fabric samples were collected from 280–400 nm using Jasco software. The Jasco software automates the determination of UPF, average UVA transmittance, and average UVB transmittance according to AS/NZS 4399:1996, EN 13758-1:2001, and AATCC 183:2004. Fabrics can be ranked as providing good, very good, or excellent UV protection if their UPF values range from 12–24, 25–39, and above 40, respectively.

### 2.2.4. Fourier transform infra-red spectroscopy (FTIR)

Alterations happening within the chemically useful bunches of maturely treated silk fabrics were observed by Bruker's Vertex 70—Fourier Transform Infra-Red Spectroscopy with Weakening Add-up to Reflection (FTIR-ATR) with a resolution of 4 cm<sup>-1</sup>.

### 2.2.5. Scanning electron microscopy (SEM)

SEM microscopy was carried out to characterize the morphology and dispersion of AgNPs in the polymer matrix. SEM is a surface imaging routine used to determine the different particle sizes, size distributions, nanomaterial shapes, and surface morphology of the produced particles at the nano-scale. Energy dispersive X-ray (EDX) spectroscopy was used to determine the composition and elemental analysis of the nanomaterial. The internal structure of the textile samples was analyzed using a scanning electron microscope. The fabric and treated fabrics were dehydrated and scanned. Scanning electron microscopy was achieved by JEOL (JXA-840A), an electron probe microanalyzer from Edwards, England.

### 2.3. The antimicrobial activity

The anti-microbial activity of PE and PE-treated oils was studied by the disc agar diffusion method. The four representative test microbes used were *Staphylococcus aureus* ATCC 6538-P (G+ve) and *Escherichia coli* ATCC 25933 (G-ve), *Candida albicans* ATCC 10231 (yeast), and *Aspergillus niger* NRRL-A326 (fungus). Nutrient agar plates

were heavily inoculated regularly with 0.1 ml of 105–106 cells/ml in the case of bacteria and yeast. Potato dextrose agar plates seeded with 0.1 ml (106 cells/ml) of the fungal inoculum were used to evaluate the antifungal activities. Textile-treated discs (15 mm in diameter) were placed over the inoculated plates. Then plates were kept at a low temperature (4 °C) for 2–4 h to allow maximum diffusion. The plates were then incubated at 37 °C for 24 h for bacteria and at 30 °C for 48 h in an upright position to allow maximum growth of the organisms. The antimicrobial activity of the test agent was determined by measuring the diameter of the zone of inhibition, expressed in millimeters (mm). The experiment was carried out more than once, and the mean reading was recorded.

## 2.4. Mosquito adulticidal repellency bioassay

### 2.4.1. Mosquito colony

The adult stage of the *Culex pipiens* colony was used in all tests, as its larvae were raised and maintained in the Mosquito Breeding Laboratory, Department of Entomology, Faculty of Science, for several generations (F10). Mosquito larvae were reared in enamel dishes (30 × 25 × 15 cm), filled with 3 L of dechlorinated tap water, and given mixed powdered dog biscuits and Tetramin® fish food (w/w) every 2 days. At 27 ± 2 °C, 75–80% RH, and a photoperiod of 12/12 h (L/D), the colony remained healthy. The pupae were transferred to a mosquito cage (30 × 25 × 25 cm) for molting and the emergence of the adult used in the experiments. Adult mosquitoes use an 8% sucrose solution as food. Both larvae and adults were kept in identical laboratory conditions and were continuously available for upcoming tests (Baz, 2013).

### 2.4.2. Adulticidal repellency efficacy in vitro

**2.4.2.1. Cone mosquito bioassays.** The effectiveness of cotton fabrics treated with selected essential oils and the untreated was evaluated using the WHO cone test method. Five replicates of the experiments were carried out. The test mosquito was a female adult *Aedes aegypti* that was raised in a lab environment with a temperature and humidity of around 27 ± 2 °C and 75 ± 5%. In the experiment, batches of mated but not blood-fed mosquitoes aged between 3 and 5 days were used. The cone test was carried out in a lab setting that replicated mosquito rearing conditions. The cone in use had a base diameter of 12 cm and a height of 6 cm, and it was manufactured of glass. At the cone's base, the treated textiles were positioned. There is a gap between the lower edge or base of the cone and the upper opening of the cone, from which the mosquitoes emerge into a large cup with a capacity of 2 liters that is completely covering the cone and the piece of cloth. Inside the cone, ten (10) vulnerable, mature adult female mosquitoes were released. For 60 min, adult mosquito repellency was viewed, noted, and recorded at 10-min intervals. Also, the untreated textiles were positioned at the cone's base to test. The term “repellency” in this study refers to the ability or quality to

deter. The number of adult mosquitoes that landed or rested on the textiles was used to quantify repellency, and the percentage repellency value was determined for each time, as shown in the formula, to calculate the adult mosquitoes' percent repellency.

$$\% \text{repellency} = 100 - \{X/N \times 100\}$$

where X is the number of mosquitoes that landed or rested on the filter paper and N is the overall number of mosquitoes. The mean % repellency ± standard deviation (SD) was reported for the untreated and treated fabric with selected essential oil (Schreck, 1977).

**2.4.2.2. CDC bottle bioassays.** According to Vatandoost et al. (2019), adult mosquito susceptibility testing for the promising oils was done using the CDC bottle bioassays. Three bottles, one for each concentration of oils, were used. Different concentrations of each oil (0.5, 1, 2, 4, and 5%) were produced using pure ethanol as the solvent. The bottles were coated with the necessary concentrations, and they were then allowed to evaporate for an hour at 27 ± 2 °C. A hand aspirator was used to collect 15 adult mosquitoes (aged 3–4 days) to fill each bottle. After an hour of exposure, the mosquitoes were removed from the bottles and placed in a small cage (25 × 25 × 15 cm) with a 10% sucrose solution. After 24 h, mortality was determined using three duplicates.

## 2.5. Phytochemical analysis

### 2.5.1. Identification of volatile contents using the GC/MS

For the GC/MS, which was used for the biochemical analyses, Thermo Scientific Trace GC Ultra/ISQ Single Quadrupole MS and TG-5MS fused silica capillary columns, 0.1 mm, 0.251 mm, and 30 m thick, were used. It was accomplished with the aid of an electronic ionizer with an ionization energy of 70 eV. Helium was used as a carrier gas (flow rate: 1 ml/min). Both the MS transmission line and the injector were adjusted to a temperature of 280 °C. The oven was preheated to 50 degrees, increased to 150 degrees at a pace of 7 degrees per min, paused for two min, then increased to 270 degrees at a rate of 5 degrees per min, and lastly increased to 310 degrees at a rate of 3.5 degrees per min (continued for 10 min). To investigate the quantification of all components found, a relative peak area was used. It was possible to figure out what chemicals they were by comparing their retention times and mass spectra to those of NIST and Willy Library data from the GC/MS instrument. The total spectrum of user-generated reference libraries was used for identification. Single-ion chromatographic reconstructions were carried out to assess peak uniformity. Whenever possible, co-chromatographic analysis of reference chemicals was employed to confirm GC retention times (Shaker et al., 2022).

**Table 2.** Knock-down effect of 12 essential oils against adult *Culex pipiens* mosquitoes through the CDC bottle technique at 1%, 60 min exposure.

No.	Oil name	Mortality	LC <sub>50</sub> (Low-Up.)	RE (LT <sub>50</sub> )	LC <sub>90</sub> (Low-Up.)	Slope ± SE	Chi (Sig.)
1	<i>Rosa canina</i>	28.89 ± 2.22 <sup>ea</sup>	133.35 (88.40–282.73)	1.1	765.82 (341.04–3593.53)	1.688 ±0.274	2.185 (0.534)
2	<i>Origanum vulgare</i>	51.11 ± 2.22 <sup>aa</sup>	68.14 (53.53–97.44)	2.1	338.47 (201.53–779.52)	1.841 ±0.227	4.669 (0.197)
3	<i>Dianthus caryophyllus</i>	53.33 ± 3.85 <sup>aa</sup>	64.73 (50.50–93.42)	2.2	369.07 (212.94–896.19)	1.695 ±0.210	4.156 (0.245)
4	<i>Cymbopogon citratus</i>	55.55 ± 2.22 <sup>aa</sup>	60.03 (46.63–87.11)	2.4	397.16 (221.93–1021.57)	1.561 ±0.195	3.927 (0.269)
5	<i>Ruta chalepensis</i>	33.33 ± 0.00 <sup>da</sup>	117.33 (79.34–232.50)	1.2	791.43 (353.90–3473.22)	1.545 ±0.237	0.269 (0.965)
6	<i>Cupressus sempervirens</i>	37.78 ± 2.22 <sup>ca</sup>	103.59 (71.40–193.05)	1.4	778.44 (351.22–3258.46)	1.463 ±0.217	1.119 (0.772)
7	<i>Camellia sinensis</i>	40.00 ± 0.00 <sup>bca</sup>	105.11 (70.79–208.37)	1.4	940.07 (394.94–4608.65)	1.346 ±0.206	1.154 (0.764)
8	<i>Azadirachta indica</i>	26.67 ± 3.85 <sup>ea</sup>	143.81 (92.42–328.08)	1.0	906.04 (379.40–4926.33)	1.603 ±0.27	1.390 (0.707)
9	<i>Melissa indicum</i>	40.00 ± 3.85 <sup>bca</sup>	106.88 (71.90–212.52)	1.3	932.27 (393.13–4542.18)	1.362 ±0.208	1.797 (0.615)
10	<i>Pimpinella anisum</i>	37.78 ± 2.22 <sup>ca</sup>	100.76 (69.47–189.28)	1.4	799.36 (356.99–3404.71)	1.424 ±0.211	1.195 (0.754)
11	<i>Eucalyptus camaldulensis</i>	53.33 ± 3.85 <sup>aa</sup>	61.65 (48.22–88.40)	2.3	368.30 (211.85–896.79)	1.651 ±0.203	2.379 (0.497)
12	<i>Pelargonium graveolens</i>	42.22 ± 2.22 <sup>ba</sup>	93.00 (64.41–172.51)	1.5	830.52 (364.44–3637.68)	1.347 ±0.199	0.956 (0.811)

a, b & c: There is no significant difference ( $p > 0.05$ ) between any two means, within the same column have the same superscript letter. A, B & C: There is no significant difference ( $p > 0.05$ ) between any two means, within the same row have the same superscript letter.

**Table 3.** Knock-down effect of 12 essential oils against adult *Culex pipiens* mosquitoes through the mosquito cone technique at 1%, 60 min exposure.

No.	Oil type	Total No. exposed	No. of specimen escaped	No. of specimen dead	Mosquito repellency %
1	<i>Rosa canina</i>	15	5	1	40.0
2	<i>Origanum vulgare</i>	15	6	3	60.0
3	<i>Dianthus caryophyllus</i>	15	7	4	73.3
4	<i>Cymbopogon citratus</i>	15	8	3	73.3
5	<i>Ruta chalepensis</i>	15	4	3	46.7
6	<i>Cupressus sempervirens</i>	15	5	3	53.3
7	<i>Camellia sinensis</i>	15	5	2	46.7
8	<i>Azadirachta indica</i>	15	4	2	40.0
9	<i>Melissa indicum</i>	15	7	2	60.0
10	<i>Pimpinella anisum</i>	15	6	1	46.7
11	<i>Eucalyptus camaldulensis</i>	15	7	3	66.7
12	<i>Pelargonium graveolens</i>	15	6	2	53.3

## 2.6. Data analyses

The data were analyzed by the software, SPSS V23 (IBM, USA), for doing the probit analyses to calculate the lethal concentration (LC) values and the one-way analysis of variance (ANOVA) (Turkey's HSD test). The significant levels were set at  $p < 0.05$ .

## 3. Results

### 3.1. Adulticidal repellency activity

#### 3.1.1. Screening of adulticidal activity for 12 oils

To measure the efficiency of the selected oils, they were tested through two methods: the CDC bottle and the mosquito cone methods for the suppression of mosquitoes, to determine their efficiency in repelling mosquitoes.

**3.1.1.1. CDC Bottle test.** The adulticidal activity of 12 essential oils against female *Cx. pipiens* is presented in Table 2. In terms of lethal concentrations, LC<sub>50</sub> (50%, median lethal concentration) for *Cymbopogon citratus* oil (55.5% mortality) appeared to be most effective against *Cx. pipiens*

(LT<sub>50</sub> = 60.03 min), followed by *Eucalyptus camaldulensis* (53.3 MO%) (LT<sub>50</sub> = 61.65 min), *Dianthus caryophyllus* (53.3 MO%) (LT<sub>50</sub> = 64.73 min), and *Origanum vulgare* (51.1 MO%) (LT<sub>50</sub> = 68.14 min) at low concentration (1%), 60 min PT (Table 2). According to the relative effects (RE) of oils: *Rosa canina*, *Origanum vulgare*, *Dianthus caryophyllus*, *Cymbopogon citratus*, *Ruta chalepensis*, *Cupressus sempervirens*, *Camellia sinensis*, *Melissa indicum*, *Pimpinella anisum*, *Eucalyptus camaldulensis*, and *Pelargonium graveolens* and based on the LT<sub>50</sub> values, they were 1.1, 2.1, 2.2, 2.4, 1.2, 1.4, 1.4, 1.3, 1.4, 2.3, and 1.5 times, respectively, than *Azadirachta indica* (Table 2).

**3.1.1.2. WHO Cone test.** Results showed that the mosquito repellency rate (MR%) of the textile (polyester fabrics) treated with *Origanum vulgare*, *Dianthus caryophyllus*, *Cymbopogon citratus*, *Cupressus sempervirens*, *Melissa indicum*, *Eucalyptus camaldulensis*, and *Pelargonium graveolens* oils was more than 50% for adults of *Culex pipiens*, with MR% = 60, 73.3, 73.3, 53.3, 60.0, 66.7, and 53.3%, respectively. While for the rest of the oils, the MR% was less than 50% at 1% concentration after 60 min of exposure (Table 3).

**Table 4.** Knock-down effect of four essential oils against adult *Culex pipiens* mosquitoes through the CDC bottle technique at different concentration, 60 min exposure.

Oil type	Conc. (%)	Mortality	LC <sub>50</sub> (Low-Up.)	LC <sub>90</sub> (Low-Up.)	Slope ± SE	Chi (Sig.)
<i>Cymbopogon citratus</i>	1	53.33 ± 3.85 <sup>d</sup>	0.93	8.65	18.22	1.273 ± 0.165
	5	75.55 ± 2.22 <sup>c</sup>				
	10	97.78 ± 2.22 <sup>b</sup>				
	15	100 ± 0.00 <sup>a</sup>				
<i>Eucalyptus camaldulensis</i>	1	51.11 ± 5.88 <sup>d</sup>	1.16	9.90	18.18	1.375 ± 0.159
	5	71.11 ± 4.44 <sup>c</sup>				
	10	88.89 ± 4.44 <sup>b</sup>				
	15	100 ± 0.00 <sup>a</sup>				
<i>Dianthus caryophyllus</i>	1	55.55 ± 2.22 <sup>d</sup>	0.98	7.22	12.72	1.479 ± 0.173
	5	73.33 ± 3.85 <sup>c</sup>				
	10	95.55 ± 2.22 <sup>b</sup>				
	15	100 ± 3.85 <sup>a</sup>				
<i>Origanum vulgare</i>	1	51.11 ± 2.22 <sup>d</sup>	1.18	18.79	41.16	1.066 ± 0.152
	5	66.67 ± 3.85 <sup>c</sup>				
	10	80.00 ± 3.85 <sup>b</sup>				
	15	95.55 ± 2.22 <sup>a</sup>				

a, b & c: There is no significant difference ( $p > 0.05$ ) between any two means, within the same column have the same superscript letter.

**Table 5.** Knock-down effect of four essential oils against adult *Culex pipiens* mosquitoes through the mosquito cone technique at different concentration, 60 min exposure.

Oil type	Conc. (%)	Total No. exposed	No. of specimen escaped	No. of specimen dead	Mosquito repellency %
<i>Cymbopogon citratus</i>	1	15	7	3	66.7
	5	15	9	4	86.7
	10	15	15	0	100.0
	15	15	15	0	100.0
<i>Eucalyptus camaldulensis</i>	1	15	7	4	73.3
	5	15	9	5	93.3
	10	15	12	3	100.0
	15	15	15	0	100.0
<i>Dianthus caryophyllus</i>	1	15	7	5	80.0
	5	15	9	5	93.3
	10	15	14	1	100.0
	15	15	15	0	100.0
<i>Origanum vulgare</i>	1	15	6	3	60.0
	5	15	8	5	86.7
	10	15	11	3	93.3
	15	15	14	1	100.0

### 3.1.2. Adulticidal activity for four effective oils

*Cymbopogon citratus*, *Eucalyptus camaldulensis*, *Dianthus caryophyllus*, and *Origanum vulgare* provide 100% adult mortality post-treatment, with 15% through the CDC bottle method. The mortality percentages of the adults subjected to 10% oil were 97.7, 88.8, 95.5, and 80.0%, respectively. Their adulticidal LC<sub>50</sub> values at 60 min PT were 0.93, 1.16, 0.98, and 1.18, respectively. *D. caryophyllus*, followed by *C. citratus*, were the more effective oils against adults based on LC<sub>90</sub> values (7.22 and 8.65 min, respectively) (Table 4). Also, results showed that the rate of mosquito repellency (%MR) was high through the CDC bottle method, reaching 100% for polyester fabrics treated with four oils at a concentration of 15%. While the %MR values were 100, 100, 100, and 93.3% at a 10% concentration for *Cymbopogon citratus*, *Eucalyptus camaldulensis*, *Dianthus caryophyllus*, and *Origanum vulgare*, respectively (Table 5).

### 3.1.3. Efficacy of polyester fabrics before and after cycles washing

The stability of polyester textile fabrics treated with 5 and 15% concentrations of *Cymbopogon citratus*, *Eucalyptus camaldulensis*, *Dianthus caryophyllus*, and *Origanum vulgare* on the repulsion efficiency of adult mosquitoes (*Culex pipiens*) by the cone

(funnel) technique before and after washing is shown in Table 6 and Figure 2. The data showed that polyester textile fabrics treated with essential oils had a long-term effect on expelled female mosquitoes for up to 6 washings, with a mortality rate of 24, 50, 55, and 40 at 5% concentration, whereas 42, 68, 76, and 56 after washing with 15% (Table 6).

## 3.2. Textile measurements and analysis

### 3.2.1. FT-ATR

Polyester fibers (PE) or condensed phthalic acids are usually characterized by specific peaks as follows: Peaks in 1709, 1407, and 1339 cm<sup>-1</sup> belong to PE function carboxylic groups, aromatic rings, and alkane chains (Figure 3). While treating PE fabrics with different oils doesn't affect the ATR peaks of PE except for *Dianthus caryophyllus* oil, it may be attributed to the penetration of all oils and the high volatilization of *D. caryophyllus* oil. All treated samples don't affect the PE peaks, which confirm the physical adsorption of oils on the PE surface.

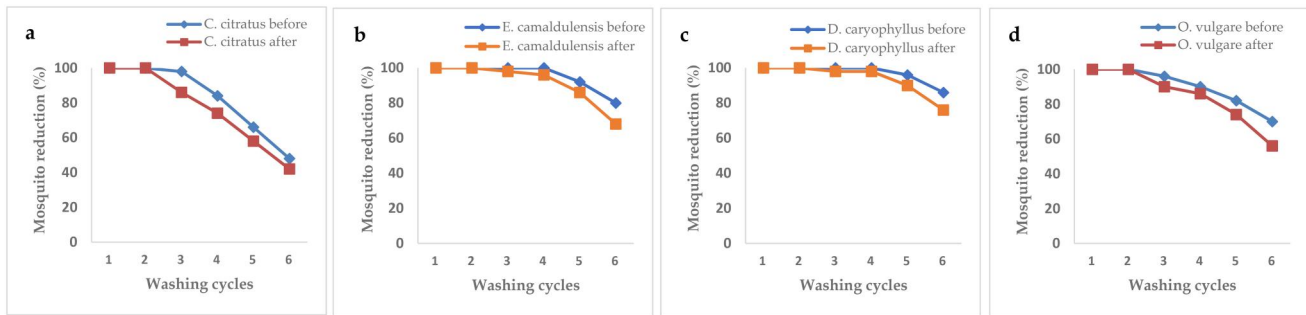
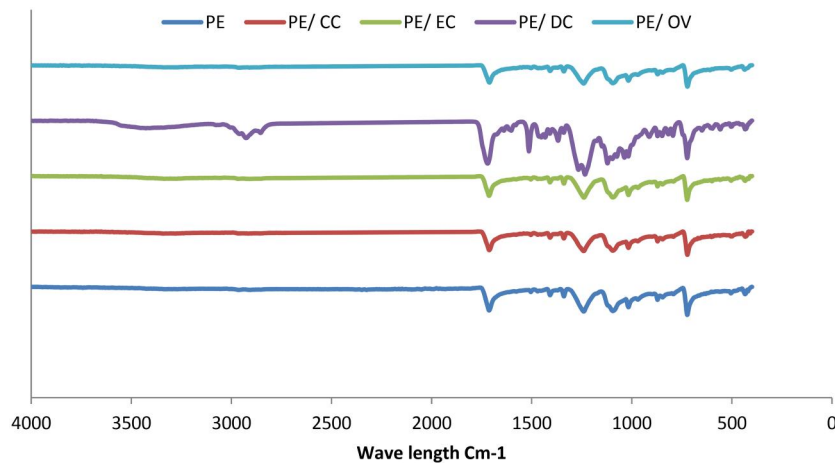
### 3.2.2. Mechanical properties

The mechanical properties of PE and PE-treated oil samples are represented in Table 7. There is no significant change at

**Table 6.** Stability of treated polyester fabrics before and after washing cycles on the efficacy of repelling mosquitoes.

Conc.	Oil name	Mosquito repellency (%)											
		Before						After					
		Wash 1	Wash 2	Wash 3	Wash 4	Wash 5	Wash 6	Wash 1	Wash 2	Wash 3	Wash 4	Wash 5	Wash 6
5%	<i>C. citratus</i>	92 ± 2.0 <sup>aA</sup>	90 ± 4.4 <sup>bA</sup>	76 ± 6.0 <sup>bB</sup>	60 ± 4.4 <sup>cC</sup>	48 ± 3.7 <sup>cD</sup>	36 ± 4.0 <sup>dE</sup>	88 ± 3.7 <sup>bA</sup>	86 ± 5.1 <sup>bA</sup>	62 ± 5.8 <sup>bB</sup>	50 ± 8.3 <sup>dC</sup>	36 ± 6.0 <sup>dD</sup>	24 ± 2.4 <sup>dE</sup>
	<i>E. camaldulensis</i>	94 ± 2.4 <sup>aA</sup>	94 ± 2.4 <sup>aA</sup>	90 ± 3.1 <sup>aB</sup>	82 ± 4.9 <sup>bC</sup>	74 ± 7.4 <sup>bD</sup>	70 ± 4.4 <sup>bE</sup>	92 ± 2.0 <sup>aA</sup>	90 ± 3.1 <sup>aA</sup>	90 ± 3.1 <sup>aA</sup>	76 ± 2.4 <sup>bB</sup>	64 ± 2.4 <sup>bC</sup>	50 ± 6.0 <sup>bD</sup>
	<i>D. caryophyllus</i>	94 ± 2.4 <sup>aA</sup>	94 ± 2.4 <sup>aA</sup>	92 ± 3.7 <sup>aA</sup>	86 ± 2.4 <sup>aB</sup>	76 ± 4.0 <sup>aC</sup>	74 ± 6.0 <sup>aD</sup>	94 ± 2.4 <sup>aA</sup>	92 ± 3.7 <sup>aA</sup>	92 ± 3.7 <sup>aA</sup>	80 ± 4.4 <sup>aB</sup>	70 ± 6.3 <sup>aC</sup>	55 ± 4.4 <sup>aD</sup>
	<i>O. majorana</i>	80 ± 4.4 <sup>bA</sup>	76 ± 2.4 <sup>bB</sup>	68 ± 3.7 <sup>cC</sup>	60 ± 6.3 <sup>cD</sup>	54 ± 5.1 <sup>bE</sup>	48 ± 8.6 <sup>cF</sup>	76 ± 8.7 <sup>cA</sup>	70 ± 4.4 <sup>cB</sup>	62 ± 5.8 <sup>bC</sup>	56 ± 6.0 <sup>cD</sup>	44 ± 7.4 <sup>cE</sup>	40 ± 4.4 <sup>cF</sup>
15%	<i>C. citratus</i>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	98 ± 2.0 <sup>aA</sup>	84 ± 4.0 <sup>bB</sup>	66 ± 5.1 <sup>dC</sup>	50 ± 7.0 <sup>dD</sup>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	86 ± 5.1 <sup>cB</sup>	74 ± 2.4 <sup>cC</sup>	58 ± 5.8 <sup>dD</sup>	42 ± 3.7 <sup>dE</sup>
	<i>E. camaldulensis</i>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	92 ± 3.7 <sup>bB</sup>	80 ± 3.1 <sup>bC</sup>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	98 ± 2.0 <sup>aB</sup>	96 ± 4.0 <sup>aB</sup>	86 ± 2.4 <sup>bC</sup>	68 ± 3.7 <sup>bD</sup>
	<i>D. caryophyllus</i>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	96 ± 4.0 <sup>aB</sup>	86 ± 4.0 <sup>aC</sup>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	98 ± 2.0 <sup>aA</sup>	98 ± 2.0 <sup>aA</sup>	90 ± 3.1 <sup>aB</sup>	76 ± 4.0 <sup>aC</sup>
	<i>O. majorana</i>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	96 ± 2.4 <sup>bB</sup>	90 ± 4.4 <sup>bC</sup>	82 ± 3.7 <sup>cD</sup>	70 ± 4.4 <sup>cE</sup>	100 ± 0.0 <sup>aA</sup>	100 ± 0.0 <sup>aA</sup>	90 ± 3.1 <sup>bB</sup>	86 ± 2.4 <sup>bC</sup>	74 ± 5.1 <sup>cD</sup>	56 ± 5.1 <sup>cE</sup>

a, b & c: There is no significant difference ( $p > 0.05$ ) between any two means, within the same column have the same superscript letter. A & B: There is no significant difference ( $p > 0.05$ ) between any two means, within the same row have the same superscript letter.

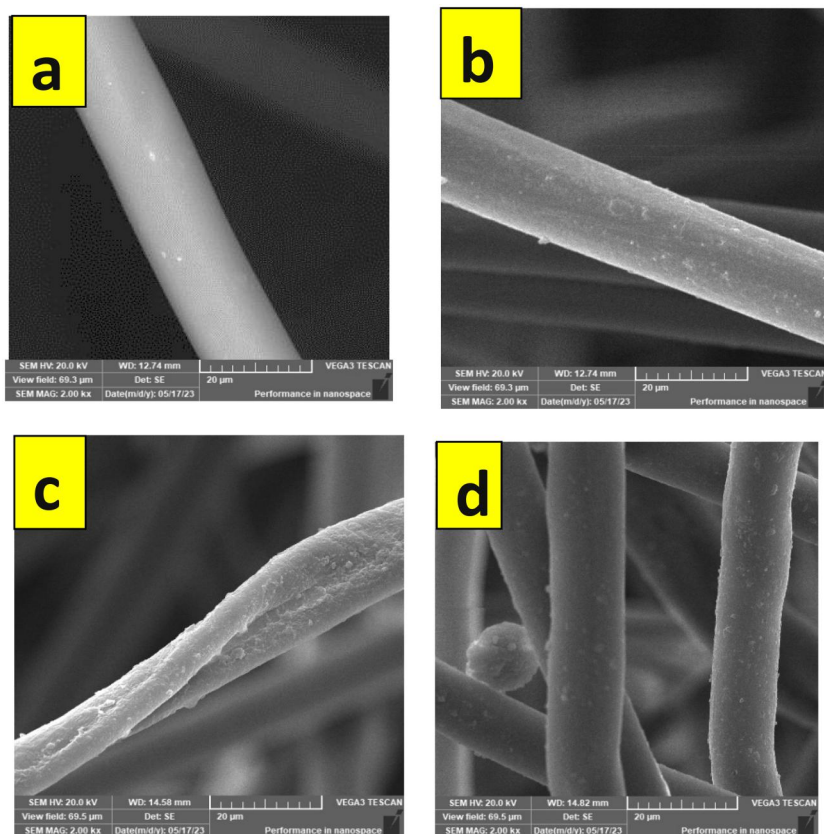
**Figure 2.** Stability of treated polyester fabrics with *Cymbopogon citratus* (a), *Eucalyptus camaldulensis* (b), *Dianthus caryophyllus* (c), and *Origanum vulgare* (d) essential oils before and after washing cycles.**Figure 3.** ATR of prepared samples, where polyester (PE), polyester/*Cymbopogon citratus* (PE/CC), polyester/*Eucalyptus camaldulensis* (PE/EC), polyester/*Dianthus caryophyllus* (PE/DC), and polyester/*Origanum vulgare* (PE/OV).**Table 7.** Mechanical properties of PE and PE treated oil samples.

Sample	Max load (Kgf)	Max load (Kgf) 5 L	Max strain Kgf/MM2	Max strain Kgf/MM2 5 L	Max strain %	Max strain % 5 L
PE	54.77	53.3	1.095	1.022	33.75	33.5
PE/CC	60.20	55.6	1.204	1.781	30.67	29.6
PE/EC	49.07	48.9	0.9813	0.887	28.67	27.55
PE/DC	55.82	52.2	1.116	1.012	30.58	28.43
PE/OV	55.96	54.2	1.119	1.065	33.75	31.34

all in mechanical parameters such as tensile strength and elongation of PE before and after treatment with oils. The tensile and elongation properties of polyester (PE) were as follows: polyester/*Cymbopogon citratus* (PE/CC), polyester/*Eucalyptus camaldulensis* (PE/EC), polyester/*Dianthus caryophyllus* (PE/DC), and polyester/*Origanum vulgare* (PE/OV).

### 3.2.3. Scanning electron microscope (SEM)

A scanning electron microscope is used for monitoring the external morphology of fibers before and after treatments. The obtained images showed the physical adsorption of oil on PE surfaces and the penetration of oil droplets between PE fibers. Figure 4(a) shows a smooth cylindrical PE fiber



**Figure 4.** SEM of PE and treated samples, where (a) polyester (PE), (b) polyester/*Cymbopogon citratus* (PE/CC), (c) polyester/*Eucalyptus camaldulensis* (PE/EC), and (d) polyester/*Dianthus caryophyllus* (PE/DC).

**Table 8.** Color Strength of PE and PE treated oils.

Wavelength (525 nm)	k/S	k/s after 5L
PE	24.43	24.43
PE/CC	28.06	27.27
PE/EC	28.51	31.09
PE/DC	29.87	30.34
PE/OV	27.62	30.72

Where Polyester (PE), Polyester/*Cymbopogon citratus* (PE/CC), Polyester/*Eucalyptus camaldulensis* (PE/EC), Polyester/*Dianthus caryophyllus* (PE/DC), and Polyester/*Origanum vulgare* (PE/OV).

**Table 9.** Color Strength of PE and PE treated oils.

Wavelength (525 nm)	UPF	UPF after 5L
PE	8947	8947
PE/CC	5118	6425
PE/EC	12659	21822
PE/DC	17538	4608
PE/OV	18429	2765

Where Polyester (PE), Polyester/*Cymbopogon citratus* (PE/CC), Polyester/*Eucalyptus camaldulensis* (PE/EC), Polyester/*Dianthus caryophyllus* (PE/DC), and Polyester/*Origanum vulgare* (PE/OV).

shape. As is known, PE fibers are produced through the melting process of PE ships, causing continuous cylindrical fibers to cool. Figure 4(b–d) show selected images for PE treated with oil.

### 3.2.4. Color strength K/S

Table 8 shows the color strength of PE and PE-treated oils. The color strength was measured at 525 nm, which belongs to the red PE-dyed fabrics under investigation. Color

strength increased with the oil treatment. There is no significant change after 5 laundries. Dying PE fabrics is usually characterized by high stability, which results from disperse-dyes inside semi-melted PE fiber in a high-temperature dying bath. The increment in color strength value may be attributed to physical light reflection that resulted from oil droplets occurring on PE fibers.

### 3.2.5. UV protection factor (UPF)

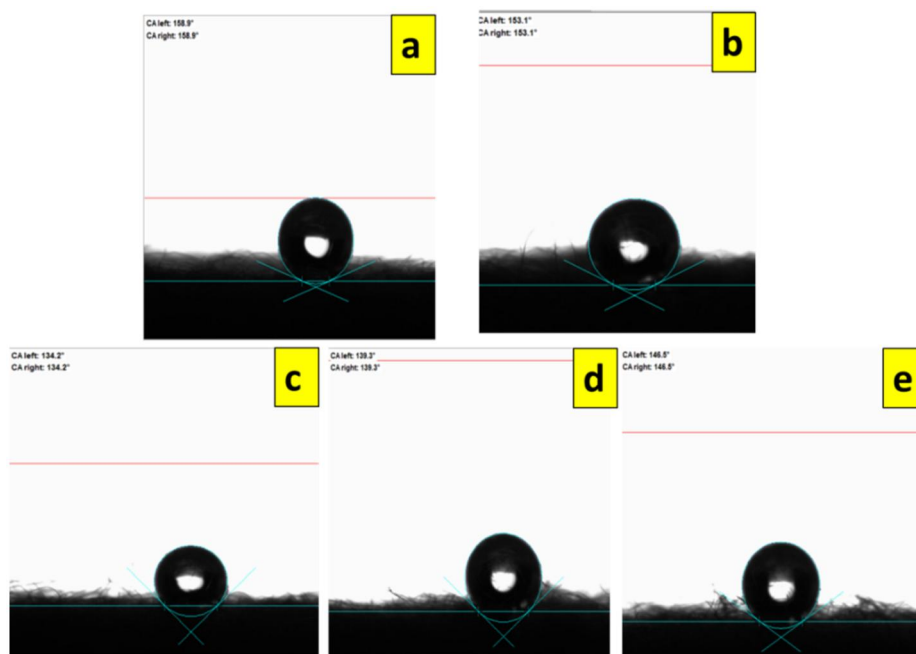
Table 9 shows the UV protection factor (UPF) of PE and PE-treated oils. UPF data for either PE or PE-treated oils showed (+%50) UPF results. It may be attributed to red dye resonance, which can scavenge UV rays and prevent their transfer. The durability after 5 L showed also (+50) results.

### 3.2.6. Contact angel

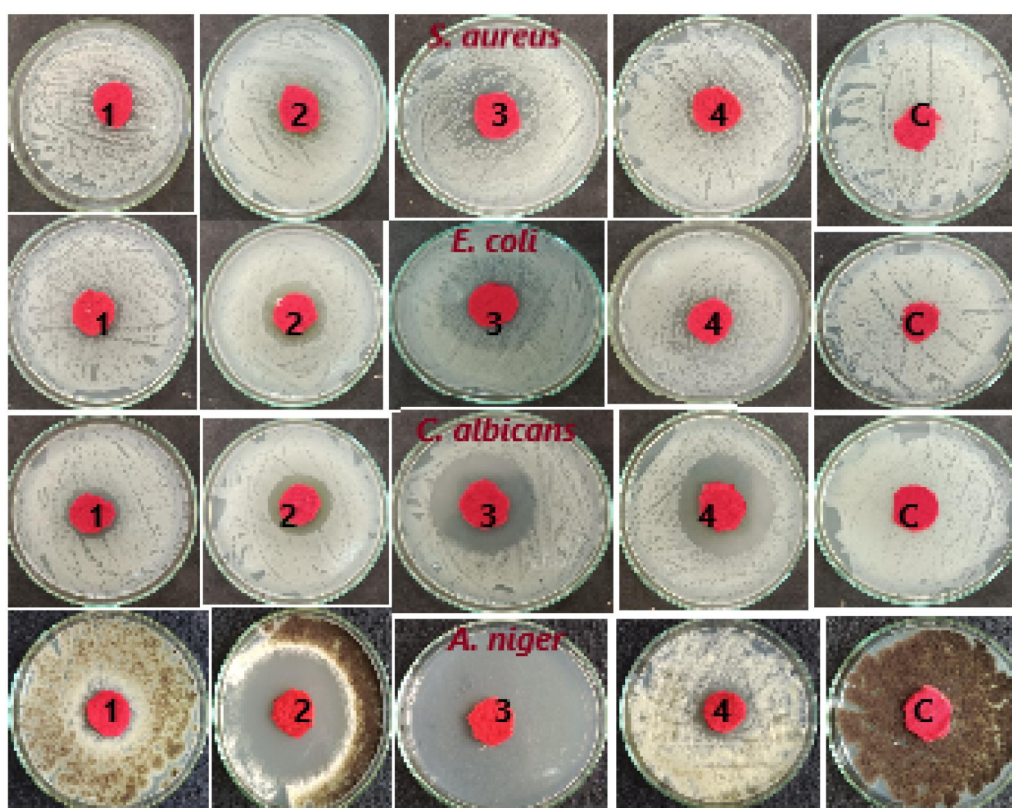
The contact angles of PE fabrics and PE fabrics treated with oils are presented in Figure 4(a), where the superhydrophobic surface with a CA was 153° (Figure 5). The data showed that PE, as an aromatic-alkylated polymer chain, has hydrophobic properties.

### 3.2.7. Antibacterial properties

Antimicrobial properties of PE and PE-treated oils were screened through an inhibition zone experiment. Figure 6 showed anti-microbial properties against *Staphylococcus*



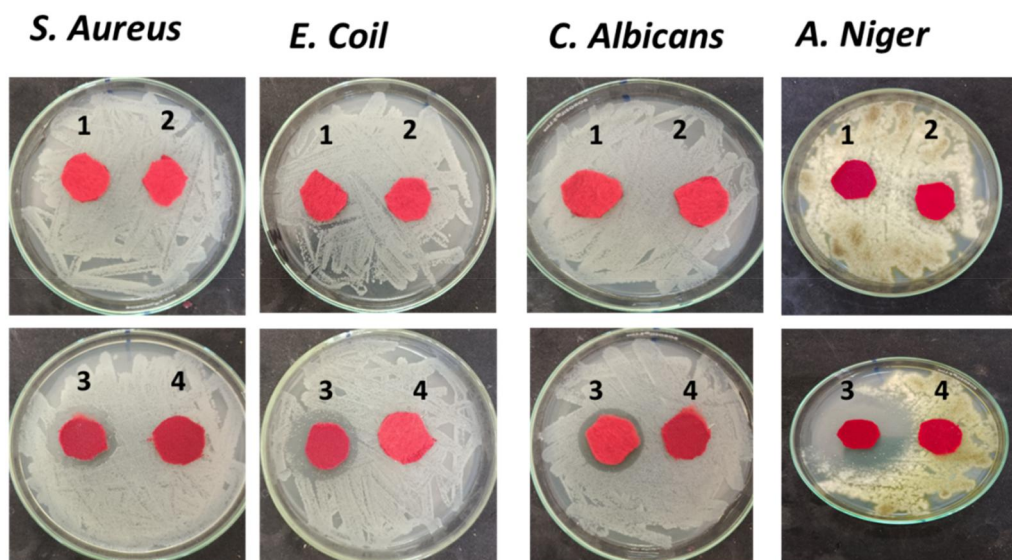
**Figure 5.** Contact angle of PE and PE-treated oils where: (a) polyester (PE), (b) polyester/*Cymbopogon citratus* (PE/CC), (c) polyester/*Eucalyptus camaldulensis* (PE/EC), (d) polyester/*Dianthus caryophyllus* (PE/DC), (e) polyester/*Origanum vulgare* (PE/OV).



**Figure 6.** Antimicrobial Properties of PE and PE treated oils where: (1) polyester/*Cymbopogon citratus* (PE/CC), (2) polyester/*Eucalyptus camaldulensis* (PE/EC), (3) polyester/*Dianthus caryophyllus* (PE/DC), (4) polyester/*Origanum vulgare* (PE/OV).

*aureus* (G+), *E. coli* (G), *C. albicans* as yeast, and *A. niger* as a fungus. All PE-treated samples showed anti-microbial activity compared with PE blank fabric. The obtained results showed *Eucalyptus camaldulensis* and *Dianthus caryophyllus* to have the best antimicrobial results.

PE-treated oils were examined for their durability for five washing cycles. The obtained antimicrobial result is shown in Figure 7. However, all PE fiber-treated oils previously showed antimicrobial activity, but their activity declined after 5 laundries, except for the sample treated with clove



**Figure 7.** Antimicrobial activity of PE and PE treated oils after 5 laundry cycles are: (1) polyester/*Cymbopogon citratus* (PE/CC), (2) polyester/*Eucalyptus camaldulensis* (PE/EC), (3) polyester/*Dianthus caryophyllus* (PE/DC), and (4) polyester/*Origanum vulgare* (PE/OV).

**Table 10.** The major chemical constituents of *Cymbopogon citratus* oil.

No.	RT	Compound name	Area (%)	M. F.	M.W
<b>Terpenes (monoterpene and sesquiterpene)</b>					
1	2.02	1-Octanol, 2,7-dimethyl-	2.55	C <sub>10</sub> H <sub>22</sub> O	158
2	6.26	à-Myrcene	6.27	C <sub>10</sub> H <sub>16</sub>	136
3	9.58	1,6-Octadien-3-ol, 3,7-dimethyl-	5.23	C <sub>10</sub> H <sub>18</sub> O	154
4	11.54	3,6-Octadienal, 3,7-dimethyl-	15.53	C <sub>10</sub> H <sub>16</sub> O	152
5	15.05	2,6-Octadienal, 3,7-dimethyl-, I-	46.89	C <sub>10</sub> H <sub>16</sub> O	152
6	18.54	2,6-Octadien-1-ol, 3,7-dimethyl-, acetate	8.38	C <sub>12</sub> H <sub>20</sub> O <sub>2</sub>	196
7	20.16	Trans-à-bergamotene	5.27	C <sub>15</sub> H <sub>24</sub>	204
<b>Phenylpropenes</b>					
8	12.75	Estragole	9.88	C <sub>10</sub> H <sub>12</sub> O	148

**Table 11.** The major chemical constituents of *Eucalyptus camaldulensis* oil.

No.	RT	Compound name	Area (%)	M. F.	M.W
<b>Terpenes (monoterpene and sesquiterpene)</b>					
1	4.91	à-Phellandrene	3.64	C <sub>10</sub> H <sub>16</sub>	136
2	8.94	Terpinen-4-ol	2.34	C <sub>10</sub> H <sub>18</sub> O	154
3	12.55	Bicyclo[3.1.1]heptane-2,3-diol, 2,6,6-trimethyl-	7.89	C <sub>10</sub> H <sub>18</sub> O <sub>2</sub>	170
4	14.26	2-Octen-1-ol, 3,7-dimethyl-, isobutyrate, (Z)-	0.59	C <sub>14</sub> H <sub>26</sub> O <sub>2</sub>	226
5	16.77	6-Nonenal, 3,7-dimethyl-	8.64	C <sub>11</sub> H <sub>20</sub> O	168
6	18.73	Caryophyllene oxide	25.48	C <sub>15</sub> H <sub>24</sub> O	220
7	24.43	1-Heptatriacotanol	3.12	C <sub>37</sub> H <sub>76</sub> O	536
<b>Phenylpropene and Phenols</b>					
8	5.27	Benzene, 1-methyl-3-(1-methylethyl)-	6.55	C <sub>10</sub> H <sub>14</sub>	134
9	10.52	Benzaldehyde, 4-(1-methylethyl)-	1.78	C <sub>10</sub> H <sub>12</sub> O	148
<b>Fatty acid and Esters</b>					
10	25.46	4,7-Octadecadiynoic acid, methyl ester	12.84	C <sub>19</sub> H <sub>30</sub> O <sub>2</sub>	290
11	26.96	hexadecanoic acid	5.96	C <sub>16</sub> H <sub>32</sub> O <sub>2</sub>	256
12	42.81	17-Pentatriacontene	17.51	C <sub>35</sub> H <sub>70</sub>	490
<b>Alkane</b>					
13	38.93	heptacosane	3.66	C <sub>27</sub> H <sub>56</sub>	380

\*Fatty acid methyl ester (FAME); monounsaturated fatty acid (MUSFA); phthalic acid monoester (FAMS).

oil (sample No. 3) which showed superior antibacterial activity compared with the other three oils.

### 3.3. Phytochemical analysis of four essential oils

GC-MS analysis led to the identification of various compounds such as terpenes, fatty acids, esters, alkanes, phenylpropenes, and phenols in the oils of *Cymbopogon citratus*,

**Table 12.** The major chemical constituents of *Dianthus caryophyllus* oil.

No.	RT	Compound name	Area (%)	M. F.	M.W
<b>Terpenes (monoterpene and sesquiterpene)</b>					
1	6.56	d-limonene	3.05	C <sub>10</sub> H <sub>16</sub>	136
2	12.04	1,5,5-trimethyl-6-methylene-cyclohexene	0.34	C <sub>10</sub> H <sub>16</sub>	136
3	12.23	3,7-dimethyl-2,6-octadien-1-ol	3.55	C <sub>10</sub> H <sub>18</sub> O	154
4	15.47	2,6-octadien-1-ol, 3,7-dimethyl-, acetate	20.10	C <sub>12</sub> H <sub>20</sub> O <sub>2</sub>	196
5	16.54	Caryophyllene	11.20	C <sub>15</sub> H <sub>24</sub>	204
6	17.89	2,6-Dimethyl-3,5,7-octatriene-2-ol, E,E-	0.14	C <sub>10</sub> H <sub>16</sub> O	152
<b>Phenylpropene and Phenols</b>					
7	6.41	benzyl alcohol	0.94	C <sub>7</sub> H <sub>8</sub> O	108
8	8.24	phenylethyl alcohol	5.95	C <sub>8</sub> H <sub>10</sub> O	122
9	13.37	2,6-octadien-1-ol, 3,7-dimethyl-, formate, (z)-	0.21	C <sub>11</sub> H <sub>18</sub> O <sub>2</sub>	182
10	14.67	phenol, 2-methoxy-3-(2-propenyl)-	23.53	C <sub>10</sub> H <sub>12</sub> O <sub>2</sub>	164
<b>Fatty acid and Esters</b>					
11	20.21	1,2-benzenedicarboxylic acid, diethyl ester	30.62	C <sub>12</sub> H <sub>14</sub> O <sub>4</sub>	222
<b>Polyacetylenes</b>					
12	36.91	8,8'-diapo-20-methoxycarotene-8,8'-dial	0.37	C <sub>21</sub> H <sub>26</sub> O <sub>3</sub>	326

*Eucalyptus camaldulensis*, *Dianthus caryophyllus*, and *Origanum vulgare* oils (Table 10–13). The most prevalent bioorganic substance was terpene (monoterpene and sesquiterpene).

The eight chemicals found in *Cymbopogon citratus* oil are shown in Table 10. The most common ones are terpene and phenylpropene: 2,6-Octadienal, 3,7-dimethyl-, I- (46.89%), and Estragole (9.88%), respectively. Table 11 shows that most of *E. camaldulensis* is made up of terpene and fatty acid chemicals called caryophyllene oxide (25.48%) and 17-Pentatriacontene (17.51%).

The 12 compounds found in *Dianthus caryophyllus* oil are shown in Table 12 and have an abundance of terpenes (2,6-octadien-1-ol, 3,7-dimethyl-acetate (20.10%) and caryophyllene (10.20%), phenylpropene and phenol (phenol, 2-methoxy-3-(2-propenyl)-23.53%), and fatty acid (1,2-benzenedicarboxylic acid, diethyl ester (30.62%). The chemical compounds that are most common in *Origanum vulgare* are listed in Table 13: 3-Cyclohexen-1-ol,4-methyl-1-(1-methylethyl) (29.46%), Bicyclo[3.1.0]hexan-3-ol,4-methyl-1-(1-methylethyl) (16.44%), and à-Phellandrene (12.67%) that were most common in terpenes in *O. vulgare* oil.

**Table 13.** The major chemical constituents of *Origanum vulgare* oil.

No.	RT	Compound name	Area (%)	M. F.	M.W
<b>Terpenes (monoterpene and sesquiterpene)</b>					
1	2.02	Cyclobutane, 1,1-dimethyl-2-octyl-	1.60	C <sub>14</sub> H <sub>28</sub>	196
2	6.70	α-Phellandrene	12.67	C <sub>10</sub> H <sub>16</sub>	136
3	7.37	Cyclohexanol, 1-methyl-4-(1-methylethenyl)-, acetate	7.63	C <sub>12</sub> H <sub>20</sub> O <sub>2</sub>	196
4	8.23	γ-Terpinene	12.69	C <sub>10</sub> H <sub>16</sub>	136
5	9.59	Bicyclo[3.1.0]hexan-3-ol, 4-methyl-1-(1-methylethyl)	16.44	C <sub>10</sub> H <sub>18</sub> O	154
6	12.16	3-Cyclohexen-1-ol,4-methyl-1-(1-methylethyl)-	29.46	C <sub>10</sub> H <sub>18</sub> O	154
7	14.42	1,6-Octadien-3-ol, 3,7-dimethyl-, acetate	6.84	C <sub>12</sub> H <sub>20</sub> O <sub>2</sub>	196
8	19.68	Caryophyllene	5.92	C <sub>15</sub> H <sub>24</sub>	204
9	24.40	(-)-Sathulenol	3.22	C <sub>15</sub> H <sub>24</sub> O	220
<b>Phenylpropene and Phenols</b>					
10	7.23	Benzene, 1-methyl-3-(1-methylethyl)-	3.53	C <sub>10</sub> H <sub>14</sub>	134

## 4. Discussions

Only female mosquitoes have the mouthparts necessary to suck blood for nutrition and complete the life cycle. So, they are responsible for transmitting diseases like malaria, dengue, yellow fever, chikungunya, etc. (Onen et al., 2023). Mosquitoes can be found up to 200 miles from where they were born (Lutambi et al., 2013). As a result, many scientists, companies, and medical organizations are researching a mosquito repellent, a finishing material or process applied to skin, clothing, and other surfaces that repel mosquitoes. In today's world, smart or functional textiles are currently one of the fastest-growing areas in the global textile industry. Protective textiles are also among these smart technology applications (Chavan et al., 2022; Mia et al., 2019). Protective textiles are those that have the function of providing protection from mosquitoes, insects, bacteria, fungi, heat, cold, etc. Hence, medical textiles are also an area that requires great attention because they are directly related to humans (Teli & Chavan, 2018). The domestic medical textile market and export market have great potential. Fabrics treated with natural materials to provide protection against insect bites and bacteria also fall into the category of medical textiles and are gaining increasing importance for consumers (Desai, 2013; Saber & Abd El-Aziz, 2022). Within this general framework, our research seeks to find natural alternatives.

### 4.1. Adulticidal repellency activity

#### 4.1.1. Screening of 12 oils

We tested 12 essential oils on polyester fabric to see which ones work best as natural mosquito repellents against the *Culex pipiens* mosquito vector of the West Nile virus. This was the first step in finding out which oils are good at keeping mosquitoes away or killing them (Table 2). All the tested essential oils in this study showed insecticidal activity against female mosquito adults (*Culex pipiens*) at 1% concentrations and intervals of exposure (60 min). To measure the efficiency of the selected oils, they were tested through two methods: the CDC bottle and the mosquito cone methods for the suppression of mosquitoes, to determine their efficiency in repelling mosquitoes. Initially, 12 essential oils were tested (surveyed) to determine the best oils to complete the rest of the study on. Due to their availability, accessibility, and relative safety, essential oils may be a good

substitute for synthetic insecticides against mosquito larvae and adults (M. M. Baz, A. M. Selim et al., 2022; Khater, 2013).

Many studies have been conducted to evaluate several essential oils against adult mosquitoes for the purpose of finding natural pesticides to repel or kill *Culex pipiens*, *Aedes aegypti*, *Culex quinquefasciatus* and *Anopheles gambiae* mosquitoes using the CDC technique, as was done in (M. M. Baz, A. Selim et al., 2022; El Zayyat et al., 2017; Faustino et al., 2021; Leyva et al., 2021; Martianasari & Hamid, 2019; Sritabutra & Soonwera, 2013). Bosly's Bosly, (2022) study evaluated the components of three essential oils: *Lavandula angustifolia*, *Mentha piperita* and *Rosmarinus officinalis*, by GC-MS analysis against adults of *Culex mosquitoes*. According to the study, lavender oil had the highest knockdown rate at 5% oil concentration at one hour (95.6%), then peppermint oil (88.9%), and finally rosemary oil (84.4%).

From his findings, it is evident that the essential oils of *Cymbopogon citratus*, *Dianthus caryophyllus*, *Eucalyptus camaldulensis*, and *Origanum vulgare* have a clear effect on female *Cx. pipiens* mosquitoes' replants, and those four oils are the most efficient or clearest of the two previous methods (CDC bottle and WHO cone). Similar to our study, the knockdown and adulticidal effects of *Cymbopogon citratus* and *Eucalyptus globulus* essential oils (Eos) and their mixtures against three medical insect pests (*Aedes aegypti*, *Aedes albopictus*, and *Musca domestica*) were very effective (Soonwera & Sittichok, 2020).

Many essential oils have been evaluated against adult mosquitoes through the WHO Cone test for the purpose of repelling and killing them to reduce the spread of diseases (Bohounton et al., 2021; Li et al., 2021; Nonviho et al., 2010; Yang et al., 2005). Narayanaswamy et al. (2014) tested natural coumarins and synthetic coumarin analogues that are known for their many medical uses, one of which is killing insects. They showed that all coumarins and halogenated coumarins kill adult *Anopheles arabiensis* mosquitoes, but not those that have been exposed to them for 24 h.

In a study parallel to our work, of all the essential oils tested, *Eucalyptus cloeziana* showed the longest effective protection duration (465 min, at 50.0% w/w) for human beings. At concentrations of 12.5% (w/w) and 25.0% (w/w), the effective protection times were 30 and 300 min, respectively. To test how well essential oils from three different *Eucalyptus* species killed insects, airtight fumigation in a

conical flask was used. It was found that the oils had a strong and quick effect on *Culex pipiens* (Tian et al., 2020).

The author showed that the combinations of *C. citratus* and *E. globulus* oils had greater knockdown and adulticidal effects against the three medicinal insect pests, with 100% knockdown and mortality rates at mixtures of 7.5% *C. citratus* + 7.5% *E. globulus* and 10% *C. citratus* + 10% *E. globulus* Eos showing the best efficiency against females of the three species. (Soonwera & Sittichok, 2020).

Another study agreed with ours that the oils of *Citrus aurantium*, *Cymbopogon citratus*, and *Cinnamomum verum* were very good at killing *Aedes aegypti* and *Aedes albopictus* adults and eggs, with an inhibition rate of 91.0–93.0% for *C. verum* oil (Moungthipmalai et al., 2023). From the above, it became clear to us to choose these four oils and complete the rest of the points of work on them in terms of the physical and chemical properties of the treated tissue and the strength of those treated tissues in repelling mosquitoes.

Sharififard et al. (2019) showed that the efficacy of clove oil in olive oil and coconut oil gave the longest lasting periods of 76.50 and 96.00 min, respectively, against *Aedes aegypti*. At 165.00, 105.00, and 112.50 min, respectively, citronella grass oil in coconut oil, citronella grass oil in olive oil, and lemongrass oil in coconut oil all demonstrated protection against *Culex quinquefasciatus*. Their research made it very evident that lemongrass, citronella, and clove oils held the greatest promise for mosquito repellent.

#### 4.1.2. Screening of four selected effective oils

Our results showed that the rate of mosquito repellency was high through the CDC bottle method, reaching 100% for polyester fabrics treated with four oils at a high concentration of 15%. Untreated textiles are used to calculate the natural mortality rate of female mosquitoes. It has been observed that mosquitoes, in the first days of the experiment, chose to rest on the inner surface of the cone instead of the treated cloth surface repeatedly due to the strength of the selected oils, especially lemon grass (*C. citratus*) and carnation (*D. caryophyllus*).

*Allium sativum*, *Anethum graveolens*, *Camellia sinensis*, *Foeniculum vulgare*, *Nigella sativa*, *Salvia officinalis*, and *Thymus vulgaris* were some of the essential oils that were studied. *Viola odorata* and *Anethum graveolens* oils worked well on adult *Cx. pipiens* (Desai, 2013). Some other oils, like *Cedrus deodara*, *Eucalyptus citriodora*, *Cymbopogon flexuosus*, *Cymbopogon winterianus*, *Pinus roxburghii*, *S. omaticum*, and *Tagetes minuta* (Makhaik et al., 2005), also kill mosquitoes, just like the oils used in this study.

According to Peng et al. (2022), menthol and essential oils—such as citronella oil and lemon eucalyptus—were the primary active ingredients in botanical preparations, working together to provide a repelling effect. In another study, citronella oil—an essential oil that is frequently used in botanical products—and a combination of citronellal, citronellol, and geraniol showed good activity against mosquitoes when applied at 100% concentration (Demirak & Canpolat, 2022). The foliar oils of cinnamon species had lethal

activities against *Aedes aegypti* and *Aedes albopictus* (Jantan et al., 2005). Essential oils have lethal effects against *Musca domestica* (Khater & Geden, 2019), such as *A. sativum*, *S. aromaticum*, and *F. vulgare* (Levchenko et al., 2021). The essential oils of *Melaleuca eucadendron* and *Callistemon citrinus* killed 100% of *Ae. aegypti* and *Cx. quinquefasciatus* adults after 24 h (Pushpalatha & Viswan, 2013). Also, many essential oils are effective repellents for other pests, such as bugs and weevils, and *Ocimum basilicum* and *Eucalyptus globulus* have the most potent fumigating activity against weevils in stores (Abd El-Salam et al., 2019).

Wu et al. (2022) evaluated a concentration of 10 µg/cm<sup>2</sup>, and eight essential oils—cinnamon, marjoram, lemongrass, bay, chamomile, jasmine, peppermint, and thyme—showed an acceptable repellent rate (>40%). Demirak and Canpolat (2022) referred to the main active components of botanical products as menthol and essential oils (i.e. citronella oil and lemon eucalyptus), which created a synergistic repellent effect. Citronella oil, which is made up of citronellal, citronellol, and geraniol and is an essential oil that is often used in botanical products, worked well against mosquitoes when it was diluted to 100%.

Strong antimicrobial action against skin-associated bacteria such as *E. coli*, *S. aureus*, *S. epidermidis*, and *T. rubrum* with mosquito-repelling qualities were demonstrated by textiles treated with the litsea and lemon essential oil microemulsion (Soroh et al., 2021). Many textiles and similar fabrics have been used to repel mosquitoes, protect humans from mosquito bites, and ensure their safety from diseases such as filaria, malaria, and types of fever such as dengue, Rift Valley fever, and West Nile fever (Anuar & Yusof, 2016; Luan et al., 2021; Mia et al., 2020; Raja et al., 2015).

#### 4.1.3. Efficacy of polyester fabrics before and after cycles washing

Our data showed the efficiency of polyester fabric treated with clove oil and eucalyptus in repelling mosquitoes, especially at the highest concentration of 15% after 6 washings. Dhillon et al. (2020) tested the efficacy of *Eucalyptus globulus* and *Rosemary officinalis* (10% concentration), as the effect of application with this concentration on the physical properties of the microcoated fabric was less and within permissible limits. Prepared finishes were applied to the fabric by the dry curing method. It was found that the eucalyptus oil-coated finish provided 100% protection against *Aedes* mosquitoes even after 15 washes, while protection against *Anopheles* and *Culex* species was lower and further reduced after successive washes. On the other hand, rosemary essential oil showed less protection, between 40 and 49% for all three mosquito species.

Moreover, we find that when several essential oils were tested in the laboratory to be ineffective, as was done in Peng et al. (2022) laboratory study, three well-known traditional Chinese repellents—cooling ointment, essential balm, and toilet water—showed reduced preventive effectiveness. In detail, there was no disgusting effect of the cooling cream or Liushen (which removes prickly heat).

Pušić et al. (2023) showed that after ten washing cycles, the antibacterial activity of polyester materials treated with chitosan against *Staphylococcus aureus* was 20% lower, even though chitosan is still present on the surface of the polyester fabrics. In another study, thyme-treated polyester fabrics exhibited the strongest antibacterial action after 50 days with a strong odor, and after 60 days or five cycle washings, a medium odor was found (El-Molla & El-Ghorab, 2022). The factors that affect the stability of essential oils can be due to several factors, including the degree of purity of the oil, molecular weight, polydispersity index, temperature, and humidity, sterilization, heat treatments, and physical methods (Turek & Stintzing, 2013).

## 4.2. Textile analysis

FT-ATR analysis showed that treating PE fabrics with different oils doesn't affect the ATR peaks of PE except for *Dianthus caryophyllus* oil. This might be because all the oils got through and the *D. caryophyllus* oil burned off quickly, which proves that the oils physically stuck to the PE surface. There is no significant change at all in mechanical parameters such as tensile strength and elongation of PE before and after treatment with oils. The obtained data showed 10% of PE were affected. This is attributed to the absence of chemical treatment on PE fabrics. Occasionally, mechanical destruction does not follow the physical treatment of fabrics. In addition, polyester fabrics are characterized by higher strength due to aromatic rings and alkane chains. The durability of washed and laundered PE fabrics has slightly changed due to these reasons.

Scanning electron microscope revealed the smooth cylindrical PE fiber shape before and after treatments. This adsorption and penetration may be due to the interaction between hydrophobic PE fiber and hydrophobic oils. Additionally, the penetration of oil particles into PE fibers may be attributed to small, sprayed oil particles, which will improve durability properties. Contact Angel experiments express the hydrophilicity and hydrophobicity properties of textile fabrics. PE fabrics treated with oils showed a slight decline in CA with 5%. It may be due to the hydrophilic extraction of oil under investigation. As known, water increases surface hydrophilicity, which reflects on CA data.

### 4.2.1. Antibacterial properties

Antimicrobial properties of PE and PE-treated oils were screened through an inhibition zone experiment. Figure 6 showed anti-microbial properties against *Staphylococcus aureus* (G+), *E. coli* (G), *C. albicans* as yeast, and *A. niger* as a fungus. All PE-treated samples showed anti-microbial activity compared with PE blank fabric. It was attributed to essential oil constituents. The composition is complex and consists mostly of terpenes (mostly monoterpenes and sesquiterpenes), terpenoids (oxygenated compounds such as phenols, alcohols, aldehydes, ketones, or ethers), and aromatic compounds. Parts of these compounds are hydrophobic, but some are hydrosoluble. Even though terpenes are

considered hydrophobic, they may present some water solubility depending on their structure and mixing temperature. Terpenoids have better solubility in water than terpenes.

However, G-bacteria usually showed more resistance than G+ because of their lipophilic double membrane. Herein, all samples showed nice antibacterial activity towards G- rather than G+, which may be attributed to the effects of essential oil on bacteria. The gram-positive peptidoglycan cell wall allows hydrophobic molecules to penetrate and reach the internal environment. The lipopolysaccharide, which is part of the external layer of Gram-negative bacteria, allows mainly small hydrophilic molecules to pass and is only partly permissive for hydrophobic molecules. The hydrophobicity of essential oils is responsible for the disruption of bacterial structures. The way essential oil works on bacteria is by breaking down the cell wall and cytoplasmic membrane, causing cytoplasmic coagulation, and allowing molecules to move through the membrane's double lipid layer. It also changes how permeable and functional the membrane is.

The obtained results showed *Eucalyptus camaldulensis* and *Dianthus caryophyllus* to have the best antimicrobial results. *E. camaldulensis* leaves contain 5–11% tannin. The kino (a class of wood exudates) contains 45% kinotannic acid as well as kino red, a glucoside, catechol, and pyrocatechol. Leaves and fruits test positive for flavonoids and sterols. The bark contains 2.5–16% tannin, the wood 2–14%, and the kino 46.2–76.7% (Watt & Breyer-Brandwijk, 1962). Some of the reported phytoconstituents of the tree included essential oils, sterols, alkaloids, glycosides, flavonoids, tannins, and phenols. While *D. caryophyllus* oil is composed of, a phytochemical analysis of *D. caryophyllus* showed that it contained triterpenes, alkaloids, coumarins, cyanogenic glycosides, cyanidin, pelargonidin, the yellow isosalipurposide, essential oils, volatile oils, and many other chemical contents. Pharmacological studies revealed that the plant possessed anticancer, antiviral, antibacterial, antifungal, insecticidal, repellent, antioxidant, renoprotective, anesthetic, and analgesic effects.

Antibacterial finishing is usually done on textiles to make them more resistant to microorganisms so that fibers don't get damaged or discolored and the fabrics last longer. This is an important part of keeping things clean in clinical and sensitive settings because it reduces the number of microorganisms that can live on textiles and spread from fabric surfaces.

Herein, PE-treated oils were examined for their durability for five washing cycles. The obtained antimicrobial result is shown in Figure 7. However, all PE fiber-treated oils previously showed antimicrobial activity, but their activity declined after 5 laundries, except the sample treated with clove oil (sample No. 3) showed superior antibacterial activity compared with the other three oils. As mentioned before, *Dianthus caryophyllus* is composed of phytochemicals. The phytochemical analysis of *D. caryophyllus* revealed that it had many different chemicals in it, such as triterpenes, alkaloids, coumarins, cyanogenic glycosides, cyanidin, pelargonidin, the yellow isosalipurposide, volatile oils, essential oils,

and more. Pharmacological studies revealed that the plant possessed anticancer, antiviral, antibacterial, antifungal, insecticidal, repellent, antioxidant, renoprotective, anesthetic, and analgesic effects.

#### 4.2.2. Phytochemical analysis of four essential oils

In this study, the organic compounds of the oils were looked at, with *D. caryophyllus* clove oil having a higher chemical percentage and a greater number of different organic compounds. Phenylpropene, phenols, terpenes, fatty acids, esters, and compounds were abundant, while polyacetylene compounds were the least common. *E. camaldulensis* contains similar compounds as clove oil, in addition to containing a high percentage of terpenes.

A lot of different chemicals were found in *D. caryophyllus*. These included triterpenes, alkaloids, coumarins, cyanogenic glycosides, cyanidin, pelargonidin, yellow isosalipurposide, and volatile compounds (Al-Snafi, 2017). Eltayeb (2016) tested the phytochemicals of *Dianthus caryophyllus*, which have triterpenes, alkaloids, coumarins, and cyanogenic glycosides. The findings demonstrated the abundance of phytochemicals, such as flavonoids, phenols, saponins, terpenes, and tannins, in *E. camaldulensis* extracts (Anigboro et al., 2020).

Every plant produces a remarkable variety of secondary metabolites. Among these metabolites, phenolic chemicals represent one of the most significant classes. Numerous phenolic compounds could repel mosquitoes (Mayeku et al., 2013; Vachon et al., 2020). According to Wu et al. (2022), cinnamaldehyde, citral, and terpinene-4-ol were the main components that GC-MS analysis revealed to be effective mosquito repellents in cinnamon, marjoram, lemongrass, bay, chamomile, jasmine, peppermint, and thyme essential oils.

Mosquito-repelling fabric is another type of bio-functional textile. Plant-based essential oils are a traditional source of insect repellents; at present, 144 patents are pending in this field (Pohlit et al., 2011). Cinnamon, citronella, eucalyptus, lavender, peppermint, clove, lemongrass, germanium, camphor, lemon, chamomile, jasmine, juniper, verbena, and wild soybean are among the essential oils that have been shown to support the mosquito-repelling effect. Not only are textiles infused with essential oils effective in their intended role as mosquito repellents, but they also offer a sustainable method of doing so (Asadollahi et al., 2019; Mehta & MacGillivray, 2022).

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## 5. Conclusion

The purpose of this study is to determine which textile finishes are best suited for creating mosquito repellent fabric and other insect repellent fabrics. The results of both qualitative and quantitative investigations point to the immense power of natural elements as well as their varying impacts on various types of fabric. When essential oils were applied to polyester textiles, *E. camaldulensis* and *D. caryophyllus* oils effectively repelled insects. The effectiveness of the created textiles as mosquito repellents was assessed. This study showed that textile fabrics can be finished with insect repellent agents to offer external protection against mosquito bites in the form of door curtains, military clothing, bed linens, tablecloths, and sofa covers. Scanning electron microscopy showed the physical adsorption of oil on PE surfaces and the penetration of oil droplets between PE fibers, where the clove (*D. caryophyllus*) essential oil was the best in biological activity and in textile measurements and analysis. Whereas the growing number of diseases spread by mosquitoes in the modern world necessitates the development of novel insect repellents, both synthetic and natural.

## Disclosure statement

The authors declare that they have no conflict of interest.

## Author contributions

Conceptualization, MMB, RMA, and ASM; methodology, MMB, RMA, and ASM; validation, MMB, RMA, and ASM; formal analysis, MMB, RMA, and ASM; investigation, MMB, RMA, and ASM; resources, MMB, RMA, and ASM; data curation, MMB, and ASM; writing—original draft preparation, MMB, RMA, and ASM; writing—review and editing, MMB, RMA, and ASM; supervision, MMB, RMA, and ASM; All authors have read and agreed to the published version of the manuscript.

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The study was conducted according to the guidelines of the Declaration of Benha University and approved by the Ethics Committee of the Faculty of Science, Benha University (Code No. BUFS 2023-17Ent).

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