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Potential Applications of Microencapsulated Essential Oil Components in Mosquito Repellent Textile Finishes

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Abstract: This study aimed to develop a cost-effective and eco-friendly mosquito-repellent textile finish based on microcapsules incorporated with limonene, camphor, linalool, menthol, and 1-octanol individually. Essential oil-based microcapsules were prepared by the emulsion extrusion microencapsulation method. The concentration of active components was determined by the gas chromatography-mass spectrometry (GC-MS) technique at different time intervals. The prepared microcapsules were incorporated into the textile finish to prepare an insect-repellent finish and applied to polyester: cotton (40:60) fabric using a conventional pad-dry cure method. Fourier transform infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM) analyses were performed to ensure the presence and stability of essential oil components on the fabric. FT-IR spectra showed that peaks observed in the range of (3400–3200 cm^{-1}) and (1720–1600 cm^{-1}) correspond to –OH stretching and bending vibrations in both untreated and microencapsulated essential oil-treated fabric. Mosquito-repellent activity was assessed by exposing treated and untreated fabric to mosquitoes. To study the long-lasting impact of microencapsulation of essential oil components on fabric, mosquito repellency was repeated every 10 to 50 days. Fabrics treated with microencapsulated essential oil components presented higher and longer-lasting protection from mosquitoes than untreated fabrics. Menthol (97%), linalool (93%), and limonene (93%) encapsulated finishes showed significantly higher repellency (>90%) as compared with octanol finishes. The studied mosquito repellent finishes could be ideal candidates for textile finishing industries.

Keywords: microencapsulation; essential oil; menthol; linalool; camphor; 1-octanol; limonene; mosquito; textile; GC-MS

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1. Introduction

Everything is created by nature with a specific principle and intention in this world, but not everything is valuable for human beings; some are dangerous and have even deadly effects. Mosquitoes are one of these harmful creations. In English, the word “mosquito” was introduced many years ago, around 1572. In some languages, they were also called little flies or biting flies. The word “mosquito” was introduced because the term “house fly” was often confused with “biting fly” [1]. As people’s living standards have advanced, the smartness and comfort of textiles have become very important in our daily lives [1], and the production of modified textiles to add high-value characters is a current progression in textile chemistry to meet consumers' needs for better living [2]. Nowadays, textiles with new high-added-value functions could be much more than conventional textiles, with new wrinkle-free and stain-resistant functions to give a pleasant fragrance, antibacterial properties, and many more [3]. Furthermore, new textile products are being developed through the use of various natural products, for example, insect-repellent textiles, fragrant textiles, and textiles for medical uses with antibiotics, hormones, and other drugs [4–7].

Diseases transmitted by mosquitoes have been a major source of various infections and complications, and the risk of such diseases is increased by intense globalization and weather changes [8,9]. A large number of harmful diseases spread by mosquitoes, like Dengue, Chikungunya, Japanese encephalitis, Filariasis, and Malaria, are responsible for the deaths of millions of people around the world every year [10,11]. Annually, 700,000,000 people are affected by diseases caused by mosquitoes, and these diseases are the cause of the deaths of 1 out of every 17 people presently alive. An infection causes malaria in mosquitoes that carry protozoa, and according to World Health Organization information, malaria is responsible for the deaths of 3,000,000 people. Mosquitoes cause dengue, epidemic polyarthritis, yellow fever, dengue, malaria, several forms of encephalitis, and hemorrhagic fever. *Anopheles mosquitoes* mainly cause malaria, and it has always been a major alarm in spite of much hard work undertaken for various strategies and policies of vector control. In 2015, approximately 90% of deaths due to malaria were registered in African countries, which also included the deaths of children after every 2 min who were five years old or under five years [5,9]. Urban yellow fever is mainly caused worldwide by the genus *Aedes* mosquitoes. Mosquito bites have infected approximately 120 million people globally, and 4 million people are experiencing common chronic symptoms [9].

According to the World Health Organization (WHO), malaria due to mosquito bites can be prevented by recent prevention methods, such as indoor spraying of pesticides and durable insecticide-treated clothing that acts as repellents when people are not inside their homes [1].

Therefore, it is necessary to introduce new formulations for preventing mosquitoes in an environmentally safer manner, using environment-friendly, target-specific, and biodegradable repellents. Plant essential oils and their components have been recognized to have excellent properties as natural mosquito repellents because they are selective, biodegrade into non-toxic products, and have some effects on non-targeted organisms and the atmosphere [12–15]. However, due to the volatile nature of essential oils, special techniques, including microencapsulation, can be used to apply essential oils to textiles [16].

A suitable encapsulation method could be used to apply the volatile components to textiles. Encapsulation is chosen based on the following factors: microcapsule use and application; the required size of the microcapsules; the nature of the shell and core material; the properties of the core and shell material, either chemical properties or physical properties; the system needed for core release; and the manufacturing cost and production scale. As reported in the literature [16], the main methods for encapsulation are spray drying, spray cooling, extrusion, coacervation (simple and complex), and emulsification. The emulsion extrusion method was used in the present study. In the extrusion method of microencapsulation, a polysaccharide gel is immobilized on the core material when it is in contact with ions of multiple valencies. The extrusion method involves the emulsion of core material and a solution of sodium alginate, and then the mixture is extruded drop by drop into a hardening solution such as calcium chloride through a syringe or a pipette. The very long shelf life of microcapsules because of their almost-resistant barrier against oxygen is the main advantage of this encapsulation method. The formation of larger particles by extrusion is one of the flaws of this microencapsulation method (typically 500–1000 mm), which limits its applications [16].

Although considerable work has been conducted on the insect repellency of essential oils, to the best of our knowledge, no such comparative study has been conducted on the mosquito repellent efficacy of selected essential oil components using microencapsulation finishes on fabric. The current study deals with encapsulating essential oil components, i.e., limonene, camphor, linalool, menthol, and 1-octanol, for durable mosquito-repellent textiles via the extrusion method. The presence and stability of essential oil components in the finish and on the treated fabric were characterized by gas chromatography-mass spectrometry (GC-MS), scanning electron microscopy (SEM), and Fourier transform-infrared spectroscopy (FT-IR) techniques. The conventional pad dry cure method was applied to treat the fabric with these essential oil-loaded microcapsules. The mosquito repellency of the essential oil-treated fabric and the untreated fabric was measured and compared.

2. Material and Methods

2.1. Materials

Limonene, camphor, linalool, menthol, and 1-octanol were purchased from Sigma-Aldrich GmbH, Schnellfrof, Germany. Sodium alginate ($C_6H_9O_7Na$), calcium chloride ($CaCl_2$), and other chemicals were purchased from Alfa Aesar Co., Waltham, MA, USA. *Anopheles stephensi* mosquitoes were procured from the Department of Zoology, Government College University, Faisalabad. For mosquito repellency tests, *Anopheles stephensi* were held under insectary conditions (26 ± 2 °C and 75 ± 10% RH) and given access to a sugar solution. The polyester:cotton (PC, 40:60) fabric (76 × 68/30 × 30) was supplied by Ibrahim Textiles, Faisalabad, Pakistan.

2.2. Encapsulation

Microencapsulation of essential oil components was performed using the emulsion extrusion method, as reported by [17]. Briefly, a 2% sodium alginate solution (2 g/100 mL distilled water) was prepared and left standing for 24 h to remove bubbles. For homogenization of essential oil components and sodium alginate solution, 3 mL of each of the 5 essential oil components was added to 30 g of 2% sodium alginate solution at 1000 rpm for about 45 min in a homogenizer. An essential oil component was steadily added during mixing to the alginate solution until the desired oil loading was obtained. A 0.5% calcium chloride solution (0.5 g/100 mL distilled water) was prepared as a hardening agent. The above-homogenized mixture was loaded into a syringe and extruded into a 0.5% calcium chloride solution for encapsulation. The mixture fell into the calcium chloride solution in very small droplets through the syringe. The resulting droplets (microcapsules) were allowed to harden in a calcium chloride solution for 20 min, separated from the solution by filtration, washed twice with distilled water, and dried and weighed.

2.3. Analysis of Microcapsules

2.3.1. GC-MS Analysis

Qualitative and quantitative analysis was performed on GC-MS to determine the exact concentrations of essential oil components in the microcapsules as reported [18,19].

2.3.2. FTIR Analysis

To ensure the presence of essential oil compounds on the fabric, Attenuated Total Reflectance Fourier Transform Infrared (FTIR) spectrophotometric (ATR-FTIR, Tensor-II, Ettingen, Germany) analysis was performed at wavelengths 400 to 4000 cm^{-1} [20]. Forty scans from each of the five samples were collected and superimposed at a resolution of 4 cm^{-1}.

2.3.3. Scanning Electron Microscopy (SEM) Analysis

Morphological analysis of microencapsulated essential oil components (1-octanol, linalool, menthol, camphor, and limonene) was performed using SEM (Emcrafts cube series 3000, EmCrafts Co. Ltd, Kwangju, South Korea) [17].

2.4. Fabric Treatment

The fabric analysis of polyester-cotton blended fabric was determined using the ASTM method (ASTM D629-15). Fabric was treated with the resulting microcapsules using the pad dry cure method in order to develop mosquito-repellent fabric [9]. Briefly, the fabrics were treated with a citric acid solution in water (8 g/100 mL) and then padded with encapsulated essential oil components after 30 min. The fabrics were dried at 80 °C for 5 min and cured at 150 °C for 2 min (Figure 1).

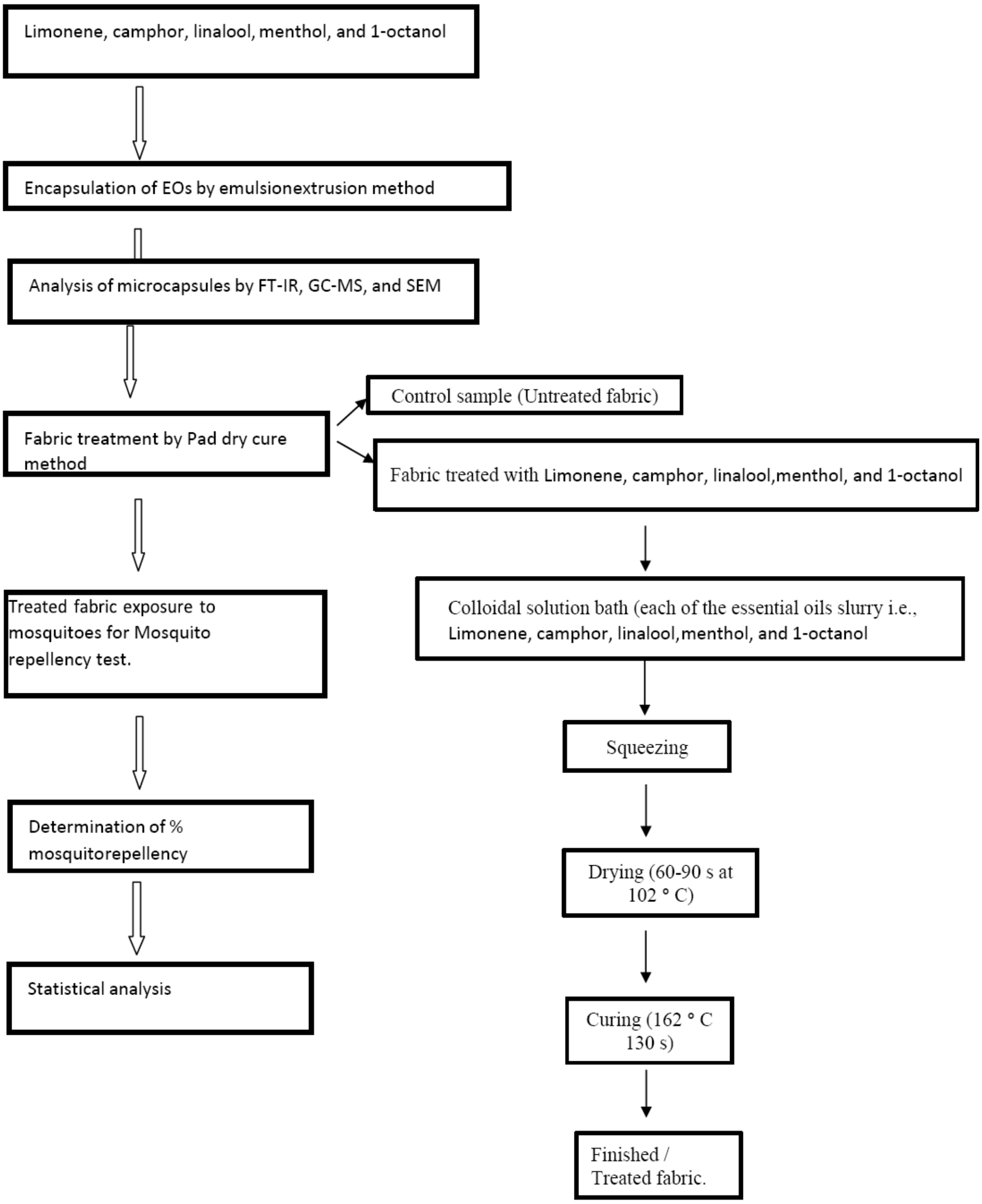

Figure 1. Schematic representation of the application of essential oil components on the fabric.

2.4.1. Mosquito Repellency Test

Two specially created excito-repellency test chambers were utilized to measure the effectiveness of repellent activity, as reported earlier [21,22]. Tests for repellency were conducted as soon as the essential oil components were applied and then every 10 days for a total of 50 days. The excito-repellency testing device's wooden outer chamber, which faces the front panel with the single escape port, has dimensions of 34 cm × 32 cm × 32 cm. The mosquito escape zone's top and bottom exit funnels have a 14-centimeter-long, outward-projecting funnel with a 1.5-centimeter-wide horizontal slit that allows mosquitoes to flee the chamber. Plexiglas makes it easier to see within the chamber's mosquito population. This experiment uses four groups of 25 *Anophese* mosquitoes. This test involves introducing four groups of 25 *Anopheses stephensi* mosquitoes into the room for one minute. Mosquitoes were starved of all food and water for at least 4 h prior to exposure in the trials. Over the course of five minutes of observation, a large number of mosquitoes that fled the chamber were manually counted at 1-minute intervals.

Estimating the percentage of mosquitoes that are repellent and do not escape from chambers was carried out using a survival analysis method. In the excito-repellency test, a specimen can only have one of two potential results.

It will either exit the exposure room alive or not (mosquitoes that escape are considered dead).

$$\%\ Mosquito\ Repellency = \lfloor \frac{Number\ of\ Mosquitoes\ escaped + Number\ of\ Mosquitoes\ dead}{Number\ of\ Mosquitoes\ Exposed} \rfloor \times 100$$

2.4.2. Statistical Analysis

Three emulsions of each essential oil were prepared and analyzed individually in triplicate. The data is thus reported as the mean ± standard deviation (SD). Ten observations were recorded and reported as mean ± SD for mosquito repellent activity. STATISTICA-5.5 (Stat Soft Inc., Tulsa, OK, USA) software was used to run the analysis of variance (ANOVA), and a probability value of $p \leq 0.05$ was reflected to show a statistically significant difference.

3. Results and Discussion

3.1. Analysis of Microcapsules

3.1.1. GC-MS Analysis

The microcapsules that were prepared by the extrusion method were qualitatively and quantitatively analyzed by GC-MS for the presence of essential oil components, as shown in Figure 2. Qualitative analysis was performed by matching retention time with authentic standards and matching spectra with the built-in standard library (NIST-02), while quantitative analysis was performed by using the standard addition method. GCMS analyses were performed for the pure essential oil components as well as the essential oil-loaded microcapsules. The most important fragmentations of different essential oil components are the following; limonene; retention time 3.750 min ($C_7H_{10}^+$ m/z = 95, $C_5H_8^+$ m/z = 68 by retro-Diels Alder allylic cleavage and base peak $C_3H_4^+$ m/z = 39 by McLafferty rearrangement), linalool; retention time 9.934 min ($C_4H_7O^+$ m/z = 71, $[M\text{-}H_2O]^+$ m/z = 136 by water cleavage, $[M\text{-}H_2O\text{-}C_3H_7]^+$ m/z = 93 and base peak at m/z = 39 which was the result of McLafferty rearrangement), camphor; retention time 3.809 min (m/z = 108, 95, 81, 69 for different molecular ions and base peak at 39 and 55 by McLafferty rearrangement), menthol; retention time 8.337 min ($[M^+]$ ion peak m/z = 150, $C_7H_7O^+$ m/z = 107, $C_7H_7^+$ m/z = 91, $C_5H_5^+$ m/z = 65 and base peak is $C_3H_3^+$ m/z = 39), 1-octanol; retention time 3.575 min ($[M^+]$ ion peak m/z = 130, $C_8H_{17}O^+$ m/z = 129, $C_7H_{14}O^+$ m/z = 114 by methyl cleavage and base peak observed at 39 and 55 via McLafferty rearrangement. The area under the peak shows the concentration of the component in microcapsules [18,19].

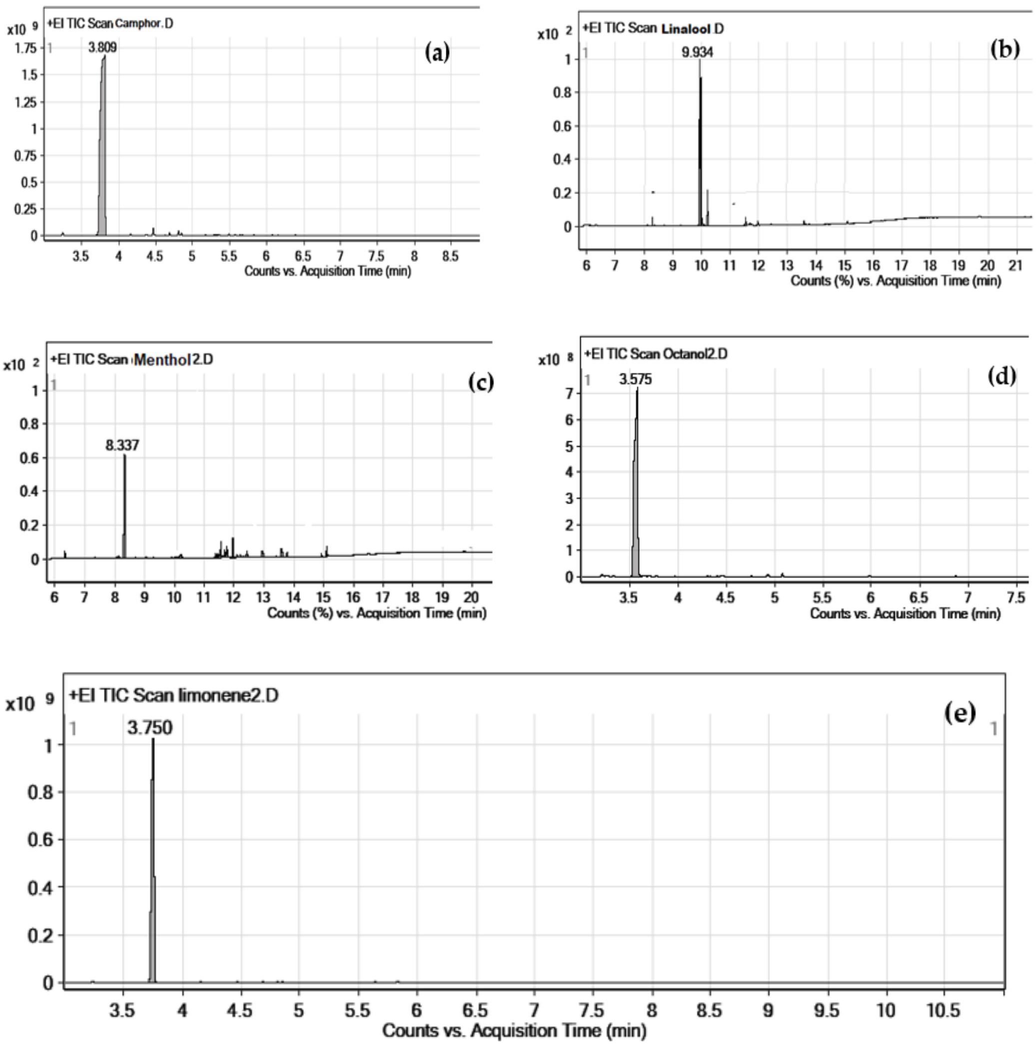

Figure 2. GC-MS of microcapsules of each essential oil component; (**a**) camphor, (**b**) linalool, (**c**) menthol, (**d**) 1-octanol, and (**e**) limonene.

3.1.2. SEM Analysis

Morphological analysis of microencapsulated essential oil components (1-octanol, linalool, menthol, camphor, and limonene) was performed via SEM, which exhibits that SEM micrographs of microcapsules evidently confirm the two types of surface, cubical surface (<10 μm) and agglomerated surface (25–100 μm), as shown in Figure 3. These microcapsules exhibit plane floating up with several visible cracks, pinholes, and apertures, and a sponge-like structure that contains micron-sized apertures arbitrarily spread on the peripheral surface [17].

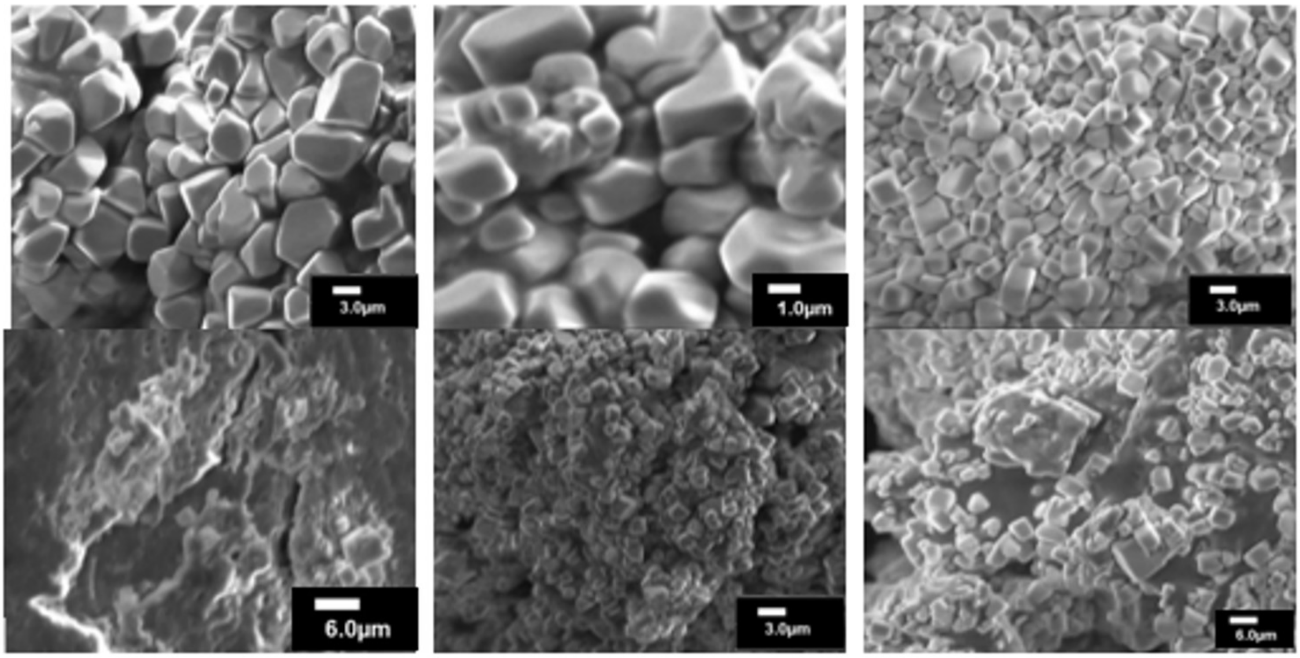

Figure 3. SEM micrographs of microencapsulated essential oil components.

3.2. Analysis of Fabric

The fabric used in the study contained 60% polyester and 40% cotton. The warp and weft were 76 and 68, respectively, and the thread count was 30.

3.2.1. FT-IR Analysis

The fabric treated with microcapsule solution by the pad dry cure method and the control sample (untreated fabric) were subjected to Fourier transform infrared spectroscopy (FT-IR) analysis, as shown in Figure 4. Regarding the FT-IR study, the peak observed in the range of (3400–3200 cm^{-1}) and (1720–1600 cm^{-1}) corresponds to –OH stretching and bending vibrations in both untreated and microencapsulated essential oil-treated fabric. The foremost constituent in fabric is cellulose, which exhibits the following vibrational bands.

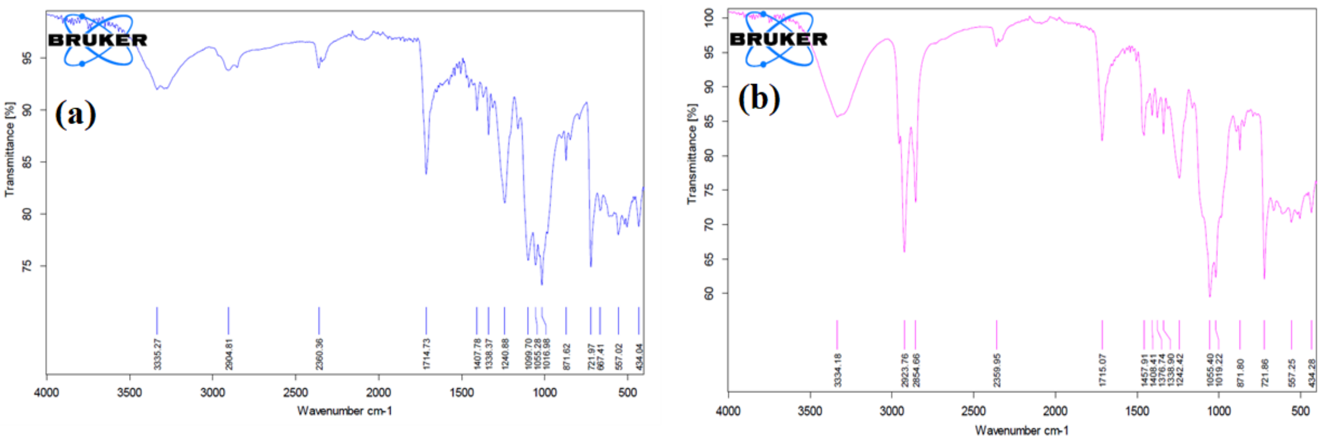

Figure 4. *Cont.*

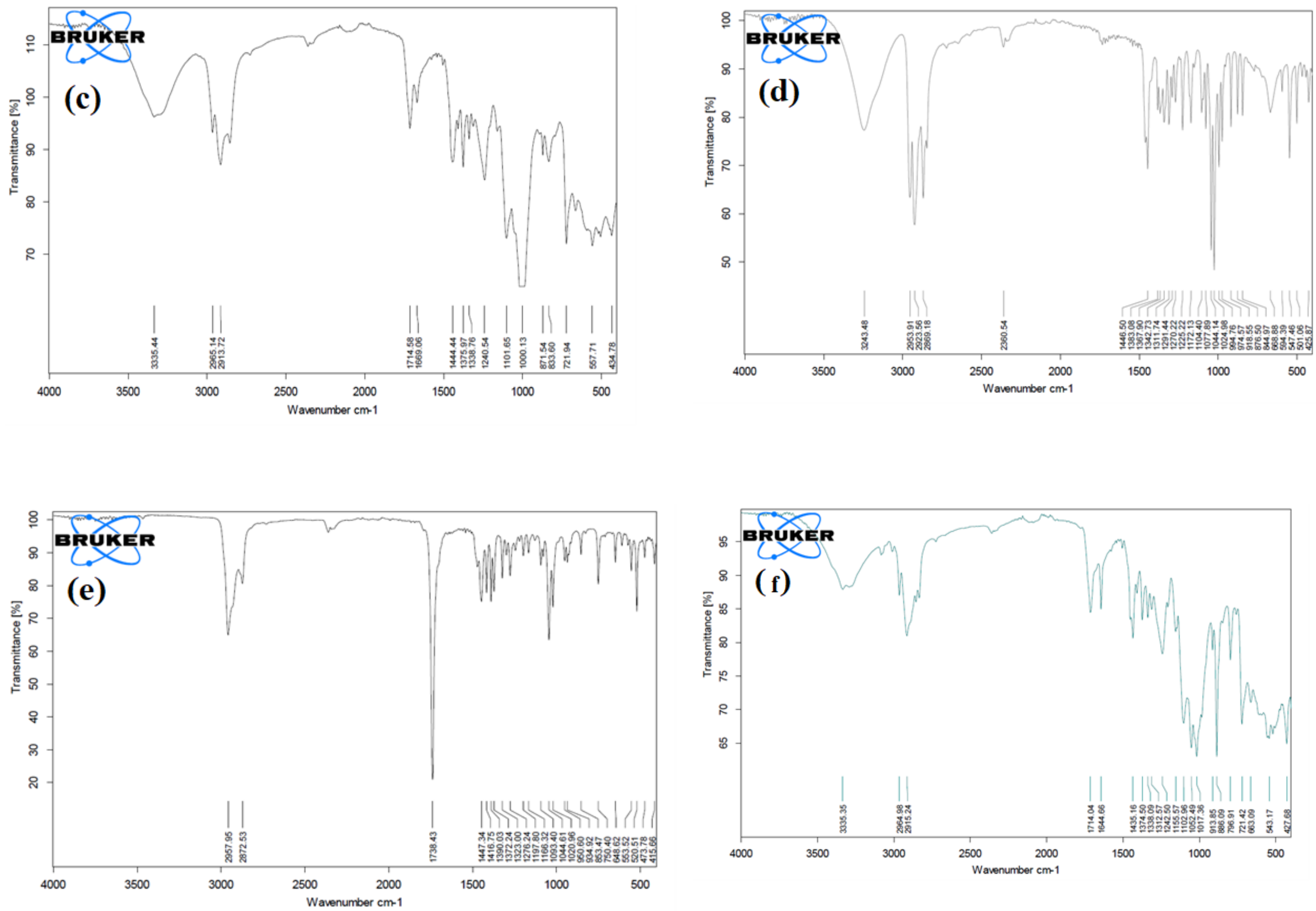

Figure 4. FT-IR spectra of (**a**) a control sample (untreated fabric) and treated fabrics with microencapsulated essential oil components; (**b**) 1-octanol, (**c**) linalool, (**d**) menthol, (**e**) camphor, and (**f**) limonene.

C–C (1100–1000 cm^{-1}), C–O (1300–1100 cm^{-1}), asymmetric in-plane ring stretch (1000–9000 cm^{-1}), –OH in-plane bend (1250–1200 cm^{-1}), and –CH wagging at (1350–1300) in both treated and untreated fabric spectra. The characteristic peaks appear in the range of 2900–2800 cm^{-1} (sp^3 C–H stretch), 3100–3000 cm^{-1} (sp^2 C–H stretch), 1370–1350 cm^{-1} (CH_3 bending), and 1470–1400 cm^{-1} (CH_2 bending) in microencapsulated essential oil treated fabric that are absent in untreated fabric [20].

3.2.2. Mosquito Repellent Activity

It has been identified that microencapsulated essential oil components have remarkable mosquito repellent properties, as shown in Figure 5. limonene: repellency percentage (93%), after 10 days (93%), 20 days (91%), 30 days (89%), 40 days (88%) and even after the interval of 50 days (87%); camphor: repellency percentage (87%), after 10 days (85%), 20 days (83%), 30 days (83%), 40 days (81%) and after 50 days (79%); linalool: repellency percentage (93%), after 10 days (91%), 20 days (90%), 30 days (89%), 40 days (85%) and after 50 days (87%); menthol: repellency percentage (97%), after 10 days (96%), 20 days (93%), 30 days (93%), 40 days (91%), and after 50 days (89%); 1-octanol: repellency percentage (83%), after 10 days (80%), 20 days (69%), 30 days (67%), 40 days (60%), and after 50 days (50%). With the exception of the octanol component, statistical analysis revealed that no significant ($p > 0.05$) alterations in the repellency of essential oil components were seen after days 30, 40, and 50. However, significant ($p \leq 0.05$) variations were observed in the case of the octanol component after days 30, 40, and 50.

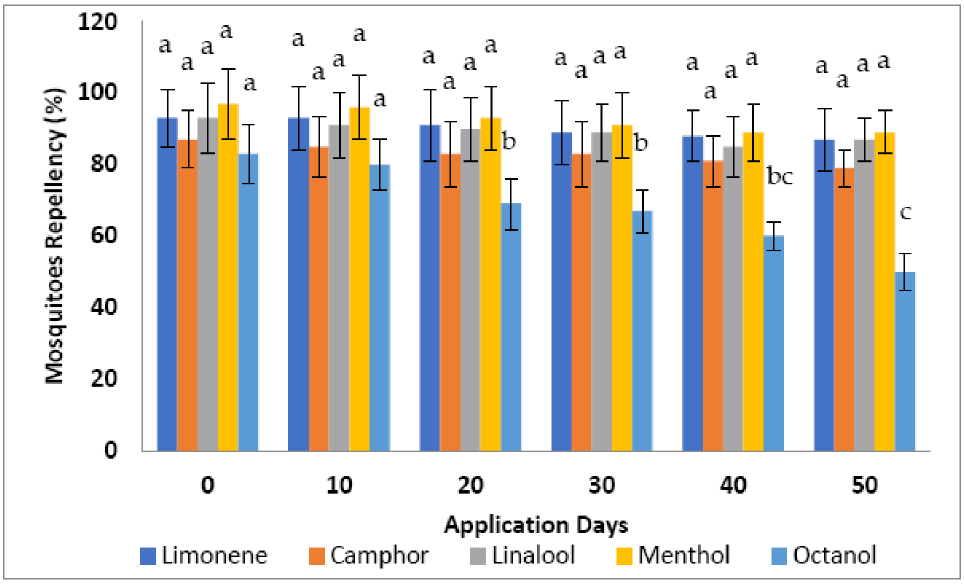

Figure 5. Effect of application days on fabric on the mosquito (*Anopheles stephensi*) repellency (%) of the essential oil components. Different letters above each bar represent significant ($p \leq 0.05$) differences among the application days.

In addition, Table 1 presents the mosquito repellency (%) of fabric treated with microencapsulated essential oil components (freshly treated fabric). The values are the mean ± standard deviation of ten independent observations. Untreated fabric (the control sample) was also tested for mosquito repellency. The average mosquito repellency activity of these five microencapsulated essential oil components showed that menthol showed the highest percentage for mosquito repellency, which was 96.6%; linalool was 93.3%; limonene was 93.3%; camphor was 86.6%; and octanol showed the highest percentage for mosquito repellency, which was 83.3%. Regarding these obtained results, it was confirmed from a comparative study that the selected microcapsules of essential oils have better efficiency than previously reported.

Table 1. Mosquito repellency (%) of fabric treated with microencapsulated essential oil components. The mosquito species used in testing was *Anopheles stephensi*.

Components	Mosquitoes Exposed (Count)	Mosquitoes Escaped (Count)	Mosquitoes Remained (Count)	Mosquitoes Dead (Count)	Repellency (%)
Control	30	1 ± 0	29 ± 1	0	3.33 ^{a}
Limonene	30	3 ± 0	2 ± 0	25 ± 1	93.3 ^{bc}
Camphor	30	4 ± 0	4 ± 0	22 ± 2	86.6 ^{b}
Linalool	30	1 ± 0	2 ± 0	27 ± 1	93.3 ^{bc}
Menthol	30	2 ± 0	1 ± 0	27 ± 2	96.6 ^{c}
Octanol	30	4 ± 1	5 ± 0	21 ± 2	83.3 ^{b}

The values are the mean ± standard deviation of ten independent observations. Different alphabet letters in the same column represent significant ($p \leq 0.05$) differences among different samples.

3.2.3. Fragrance and Mosquito Repellency Test

Fragrance was present in all treated fabrics with linalool, limonene, 1-octanol, menthol, and camphor (separately) and was checked by washing every 10 days' interval. Fabrics treated with menthol, camphor, and linalool gave a stronger fragrance as compared to 1-octanol and limonene. The intensity of fragrance starts decreasing after 35 washes, but fragrance was present even after 50 washes.

4. Comparison with Previously Reported Literature

In Table 2, these are the essential oils reported for their applications in mosquito repellent textile finishes. Bleached 100% cotton plain weaves treated with citronella oil by the conventional padding method showed higher repellency, i.e., 90%, but then it vanished after 21 days [9]. By the pad-dry cure method, 100% polyester synthetic curtain fabric treated with lemon grass showed mosquito repellent properties, and the treated fabric showed 70% mortality up to 15 industrial washes [20]. Thyme oil, cypress oil, and grapefruit oil (2:1:1) were applied to Bamboo/tencel (50:50) fiber blended fabric by the method of immersion, and the treated fabric's durability was up to 60% after 30 washes [22]. Citriodiol and citronella oil were applied to bleached 100% cotton plain weaves by the padding method, and this oil showed high repellency on day 1 but remained 70% up to 28 days [23]. Eucalyptus oil was applied to cotton and trevira knitted fabrics by the conventional padding method, and the resultant mosquito repellency efficiency had a mean protection time of just 6 hours [24]. Lemon grass-treated bleached plan-knitted single jersey cotton fabric treated by the pad dry cure method showed mosquito repellency, but it remained just about 28.5% after 30 washes [25]. *Cymbopogon flexuosus* (CF) oil was applied on nylon net fabric by a method called the layer-by-layer (LBL) method, and the activity against mosquito repellency was investigated by comparing the number of layers and the mortality rate of the mosquitoes. This work reported the activity by number of washes of that treated fabric, and the result was that up to 15 washes of material were intact on the fabric and dropped after 25 washes [26]. While in this present work, five important essential oils are discussed and their mosquito repellency power is reported, they showed a higher repellency percentage than the reported literature, and their durability check also shows that these five oils were present on the treated fabric even after 50 washes. Moreover, we can determine that among these five essential components, which one is a more suitable option for manufacturing materials like fabric, wall paints, etc. against mosquito protection. That is, menthol > limonen = linalool > camphor > 1-octanol.

Table 2. Application of essential oils in mosquito repellent textile finishes (reported data).

Essential Oil/Essential Oil Component	Essential Oil Finishing Method	Textile Application	Durability	Ref.
Cymbopogon flexuosus (CF) oil	Layer-by-layer (LBL) method	nylon net fabric	Up to 15 washes of material were intact on the fabric, which dropped after 25 washes	[26]
Lemon grass	Pad-dry curemethod	100% polyester synthetic curtain fabric	15 industrial washes, up to 70% mortality	[20]
Citriodiol and citronella oil	Padding	Bleached 100% cotton plain weaves	remained around 70% up to 28 days	[23]
Thyme oil, cypress oil, grapefruit oil (2:1:1)	Immersion	Bamboo/tencel (50:50) fiber blended fabric	Up to 60% after 30 washes	[22]
Citronella oil	Padding	Bleached 100% cotton plain weaves	Higher than 90% in the first 21 days	[9]
Lemon grass	Pad-dry cure method	Scoured, bleached plan-knitted single jersey cotton fabric of 240 GSM	28.5% after 30 washes	[25]
Eucalyptus oil	Padding	Cotton and trevira knitted fabrics	Mean protection time of 6.0 h	[24]

5. Conclusions

This research work emphasizes the production of microencapsulation of essential oil components (1-octanol, menthol, camphor, limonene, and linalool) for mosquito repellent textiles. As the essential oils are volatile, to enhance their effect and durability, the microencapsulation technique was performed by emulsion extrusion. The produced microcapsules were characterized by GC-MS, FT-IR, and SEM analysis. The microcapsule slurry was applied to polyester cotton fabric, and then the fabric was tested for mosquito repellency percentage. It can be concluded from the results that the application of selected essential oil components on fabric can give tremendous results (limonene 93.3%, linalool 93.3%, camphor 86.6%, menthol 96.6%, and 1-octanol 83.3%), and these mosquito repellent textiles can be the best alternatives to synthetic repellent to prevent mosquitoes and diseases caused by mosquitoes. They are also a safer alternative to lotions and ointments, which, when applied to the skin, cause various skin problems. Moreover, microencapsulation techniques of essential oils and other compounds can be used in various other ways, and insecticidal properties can be encapsulated for developing insect-repellent wall paints and many other tremendous applications that are more sustainable and environmentally friendly.

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