Extraction of eco-friendly essential oils and their utilization in finishing polyester fabrics for fragrant and medical textiles

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Abstract
Eco-friendly fragrances essential oils of Lavender, thyme, and clove were prepared and extracted from natural sources and utilized for finishing polyester fabrics, in order to give them good smell and antibacterial characteristics, that is, medical textile. We developed medical textiles using the essential oils (lavender, thyme, and clove) with β-cyclodextrin inclusion compounds. The fragrance inclusion compounds were fixed onto polyester by drying with a low temperature and then thermo fixed method. The essential oils were studied to prove their structure using various techniques such as IR spectra, GC-MS, etc. Moreover, the antibacterial activity of treated polyester fabrics with lavender, thyme, and clove essential oils against Staphylococcus aureus and Escherichia coli was evaluated. The results show that the finishing polyester fabrics with essential oils have antibacterial properties. The antibacterial activity of treated polyester fabrics with thyme was the highest. A strong odor was detected after 50 days, while a medium odor was detected after 60 days or five pieces of washing. The novelty of this work has arisen from its ability to fit the requirements of economic feasibility of medical textile, achieve the goal of fabrics has at smell good and has antibacterial.

Keywords
Eco-friendly, essential oils, fragrances, finishing polyester, medical textiles

Introduction
Essential oils (EOs) can be found in a variety of aromatic plants that are commonly grown in tropical and subtropical regions. Essential oils are extracted from fragrant plants’ leaves, flowers, fruits, seeds, buds, rhizomes, roots, and barks, among other parts. Essential oils have been extracted from the plant using a variety of methods. Hydro distillation, solvent extraction, cold pressing, and supercritical fluid extraction are the processes involved.1–3 Steam distillation is the most popular process for obtaining essential oils; Hippocrates, an ancient Greek physician, is said to have referred to fragrant herbs as the “father of medicine.” Aromatic plants, which produce essential oil, have been utilized for a variety of reasons since ancient times, including medical treatments, food preservatives, and food flavoring.5 Alternatively, β-cyclodextrin (β-CD), a cyclic oligosaccharide with seven D-glucopyranose units, has been identified as a human-safe component. Due to the unique configuration of the hydrophilic outside edge and the hydrophobic interior cavity, β-CD can encapsulate hydrophobic guest molecules to form a host–guest complex.6 β-CD is frequently employed because of its

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ideal cavity size, which may selectively encapsulate diverse guest molecules, particularly aromatic compounds. As a result, β-CD has the potential to be used as an encapsulate in cotton garments to extend the life of their scent without posing any health or environmental risks. There are over 3000 essential oils recognized today, with about 300 of them being commercially marketed. Terpenes/terpenoids, aromatic and aliphatic molecules, which are low-molecular-weight aroma chemicals, are the main ingredients of essential oils. In general, the biological qualities of essential oils are determined by these primary components, which are detailed in two comprehensive studies on essential oil biological activities. Textile fragrance finishing is one of the processes that adds a variety of fragrances to a product to increase its value. The global marketplace is always evolving, and as a result, customer demand is increasing. Everyone wants something fresh and special to happen on a regular basis. In order for the change to be effective, it must be applied in the market. According to this comprehensive analysis, fragrance finishing is an emerging field that has flooded the textile industry with vibrant value added finish by incorporating various aromas into fabrics, resulting in the creation of fragranced fabrics. Fragrance full textile materials were used in a variety of ways. This cost-effective technology is utilized in the field of textiles to apply various functional agents to fabrics in order to obtain higher functionality durability, for example, finding a means to extend the durability of scents on textiles has been a long-term goal of textile chemists. There have been numerous attempts to add perfumes directly to fibers and fabrics; but, because all fragrances are volatile, their permanence is low. Furthermore, after one or two wash cycles, the desired scent is completely gone. The purpose of this study was to extract eco-friendly fragrances essential oils of lavender, thyme, and clove from natural sources, then used to finish polyester fabrics in order to give them a good smell and antibacterial properties (medical textiles) and increase their durability.

**Materials and methods**

**Materials**

*Polyester fabric.* 100% polyester knitted fabric of 150 g/m², supplied by Economic and Overseas Company was used.

**Chemicals**

β-Cyclodextrin hydrate 99% was provided by Merck, Germany (m.w. = 1135 g/mol), citric acid anhydrous was provided by Merck, Germany (m.w. = 192.12 g/mol), absolute ethyl alcohol ≥99.8%, hexadecane, egyptol, were of laboratory grade chemicals were used. Lavender plant, thyme plant and cloves plant were purchased from the local market (Table 1).

**Methods**

**Essential oils extraction.** Hydro-distillation was used to separate the essential oils of lavender, thyme, and clove. In a Clevenger-type device, samples of dried flowering plants and buds were hydro distilled for 4 h. Using a liquid–liquid continuous extractor, the distillate (900 mL) was extracted for 6 h with 100 mL of dichloromethane. A rotary flash evaporator was used to remove the solvent from the resultant extract, which was dried over anhydrous sodium sulfate. When the volume of extract was decreased to around 1 mL, the distillation was stopped. The solvent was then extracted using a pure nitrogen stream until the volume was reduced to 0.5 mL (1.04 g) of volatile extract, which was then stored in the dark in the freezer until the essential oils were evaluated using gas-liquid chromatography and mass spectrometry (GC-MS). Three times the experiment was carried out.

**Preparation of microencapsulation of essential oils**

The essential oils of lavender, thyme, and clove (10%), as well as β-Cyclodextrin hydrate (5%) and citric acid as catalyst (5%), were created using a combination solution including absolute alcohol and distilled water, as well as ratio (1:3) hexadecane as a stabilizer and Egyptol as an emulsifier. A high-speed mixer was used to emulsify the solution for 20 min at a speed of roughly 10,000 rpm and a temperature of 40°C. The emulsified mixture was poured into a flask to cool.

**Fabrics finishing process**

The exhaustion approach was used to treat polyester fabrics with various essential oils namely lavender, thyme,
and cloves, using 10% Egyptol as an emulsifier. For 20 min at 40°C in a shaking water bath, polyester cloth samples 10 cm long and 5 cm wide were immersed in a solution containing the essential oil emulsified product (liquor: 1:15 ratio). The treated polyester fabrics were removed after completing and dried in the oven at 100°C for 4 min before being cured at 130°C for 3 min.

Measurements and analysis

Gas chromatography–Mass Spectrometry (GC–MS) analysis. About 200 mL of each pure essential oil were utilized to identify volatile components. A Hewlett-Packard model 5890 (Hewlett-Packard, PerkinElmer Co., USA) equipped with a flame ionization detector was used for GC analysis. A fused silica capillary column DB-5 (Zebron Co., USA) (internal diameter: 60 m 0.32 mm) was used.20

Infra-red analysis

Using a Perkin Elmer system 2000 FT-IR infrared spectrometer, the infrared of either essential oils Lavender, Thyme, and Clove and/or polyester fabrics after treatment and finishing with the essential oils were measured (Fourier transform IR spectrometer).

Scanning electron microscope (SEM)

After drying and fixation, polyester fabrics are finished with a solution containing the emulsified product of essential oils (lavender, thyme, and clove essential oil particles), mounted on aluminum stubs, and sputter-coated with gold in a 150 sputter (Coated Edwards), and examined by Jeol (JXA-840A) Electron Probe Microanalysis (Japan), magnification range 35–10,000, accelerating volts in order to confirm the presence of fragrance particles.

Antibacterial activity testing

Antimicrobial activity of the tested finishing polyester fabrics with essential oils against Gram-positive bacteria (Staphylococcus aureus, SA) and Gram-negative bacteria (Escherichia coli, EC) was tested according to AATCC Test Method 100-1999.

Free radical scavenging activity (DPPH assay)

Using the stable DPPH radical method,20 the free radical scavenging activity of the three essential oils was measured in terms of hydrogen donating or radical scavenging ability.

Washing

Some cured samples were laundered for 30 min at 40°C using a soap solution (5 g/L of Cibaboon R from Ciba) with a pH of seven and a liquor-to-fabric ratio of 1:50 according to the ISO 105-C01:1989 (E) standard.21 Some samples were washed five times for fragrance evaluation. They were air-dried after washing.

Results and discussion

GC-MS analysis of essential oils

One or two major components were identified in the essential oils, which are listed according to their retention time and % contribution. By comparing the relative retention times and mass spectra of oil components with mass spectra from the data library, the chemical compositions of essential oils from lavender, thyme, and clove were determined. The essential oils were identified by one or two prominent components, which are presented in order of retention time and contribution % (Table 2). Linalool (29.40%), cineole (23.45%), dihydro carveol (15.90%), thujanol (8.46%), thuj-3-en-10-al (4.34%), and camphor (2.41%) were the primary components of lavender essential oils detected by GC-MS (Table 2 and Figure 1).

The 16 components of thyme essential oil were identified via GC-MS analysis (Table 2). Thymol (37.13%), p-cymene (20.46%), terpinyl acetate (15.90%), thujanol (8.46%), thuj-3-en-10-al (4.34%) and camphor (2.41%) were the primary components of lavender essential oils detected by GC-MS (Table 2 and Figure 1).

The 16 components of thyme essential oil were identified via GC-MS analysis (Table 2). Thymol (37.13%), p-cymene (20.46%), terpinyl acetate (15.58%), carvacrol (10.07%), and bisabolene (5.91%) were the primary components of thyme essential oil detected by GC-MS (5.91%). Thujene, caryophyllene, and geraniol are among the others (Table 3 and Figure 2).

Because of a variety of factors, the chemical composition of Thymus species varies greatly. Increased percentages of thymol, carvacrol, borneol, linalool, or -terpineol are found in some thymus oils. Clove essential oils contain 85.6% eugenol, 9.27% heptyl acetate, 1.81% linalool, 0.8% eugenyl acetate, and 0.97% caryophyllene oxide (Table 4 and Figure 3).

### Table 2. The main chemical composition of Lavender essential oil analyzed by GC/MS.

| No | Compound                  | %    |
|----|---------------------------|------|
| 1  | Tricyclene                | 2.42 |
| 2  | Artemisia triene          | 0.035|
| 3  | α-pinene                  | 3.35 |
| 4  | β-pinene                  | 1.2  |
| 5  | Cineole                   | 23.45|
| 6  | Ocimene                   | 1.26 |
| 7  | 4-Thujanol                | 8.46 |
| 8  | Linalol                   | 29.40|
| 9  | Isophorone                | 0.05 |
| 10 | Isophorone (4-Keto)       | 5.43 |
| 11 | Camphor                   | 2.41 |
| 12 | Terpinoel (cisDihyro)     | 0.56 |
| 13 | Thuj-3-en-10-al           | 4.34 |
| 14 | DihydroCarveol (ISO)      | 15.90|
| 15 | Pulegol (trans)           | 1.71 |

Table 3. The main chemical composition of Thyme essential oil analyzed by GC/MS.

| No | Compound                  | %    |
|----|---------------------------|------|
| 1  | Thymol                    | 37.13|
| 2  | p-cymene                  | 20.46|
| 3  | terpinyl acetate          | 15.90|
| 4  | thujanol                  | 8.46 |
| 5  | thuj-3-en-10-al           | 4.34 |
| 6  | camphor                   | 2.41 |
| 7  | carvacrol                 | 10.07|
| 8  | bisabolene                | 5.91 |

Table 4. The main chemical composition of Clove essential oil analyzed by GC/MS.

| No | Compound                  | %    |
|----|---------------------------|------|
| 1  | eugenol                   | 85.6 |
| 2  | heptyl acetate            | 9.27 |
| 3  | linalool                  | 1.81 |
| 4  | eugenyl acetate           | 0.80 |
| 5  | caryophyllene oxide       | 0.97 |
According to our findings, eugenol appears to contain the largest amount of clove essential oils studied. The content of essential oils might vary due to ecological and regional factors, as well as meteorological and soil conditions.

**Transforms infrared (FTIR) spectra**

Figure 4 shows the Fourier transforms infrared (FTIR) spectra analysis for Lavender, Thyme, and Clove. The (-OH) group and another methoxy group (-O-CH3) in the clove molecule can be assumed from the C-H stretching region (3200–2800 cm⁻¹). At 3100–3000 cm⁻¹, C-H spans from aromatic to vinyl hydrocarbons. The comparable C-H stretches for methylene groups are found near 2925 (asymmetric) and 2850 (symmetric) cm⁻¹ with the corresponding C-H stretches for methyl groups near 2962 and 2872 cm⁻¹.

Figures 5 to 7 show the Fourier transforms infrared (FTIR) spectra analysis of polyester fabrics treated and finished with 10% beta-cyclodextrin, 10% citric acid, 1% hexadecane, and 5% synthetic scent compounds (Lavender, Thyme, and Clove), respectively. Figures 5 to 7 show absorption bands at 3423, 3426, and 3428 cm⁻¹, which suggest (-OH) groups in mixed fragrance compounds and finished polyester fabric. The (-OH) group of synthetic scent is thought to link with the (-OH) group of beta-cyclodextrin at this point. Carbonyl bands in ketones are frequently seen at 1720–1690 cm⁻¹, while carbonyl bands in esters are usually found at 1750–1730 cm⁻¹. Between 1400 and 1000 cm⁻¹, neighboring C-O stretching vibrations occur in esters and aldehydes.22 When using Lavender, Thyme, and Clove to finish polyester fabrics, a new absorption band appeared at 1726, 1723, and 1723 cm⁻¹, which is due to C=O stretching vibration, and the adjacent C-O stretching vibrations and absorption bands from 1400 to 1000 cm⁻¹, which is possibly due to the ester bond (-COO-) of the polyester. Because of the improvement of a macromolecular chain, the (C=O) and C-H stretching vibrations 2940, 2958,

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**Table 3.** The main chemical composition of Thyme essential oil analyzed by GC/MS.

| No | Compound           | %     |
|----|--------------------|-------|
| 1  | Thujene            | 1.3   |
| 2  | p-cymene           | 20.46 |
| 3  | Caren-7ol          | 0.07  |
| 4  | Thymol             | 37.13 |
| 5  | Methyl thymol ether| 1.06  |
| 6  | α-agarofuran       | 1.02  |
| 7  | Spathulenol        | 1.08  |
| 8  | Carvacrol          | 10.07 |
| 9  | Terpinyl acetate   | 15.58 |
| 10 | Caryophyllene oxide| 1.08 |
| 13 | Caryophyline       | 2.11  |
| 14 | Geraniol           | 0.5   |
| 15 | Cadinene           | 2.68  |
| 16 | Bisabolene         | 5.91  |
and 2913 cm\(^{-1}\) were marginally improved. The new strong absorption peaks for finished polyester with Lavender, Thyme, and Clove are 1243–1088, 1245–1090, and 1250–1097 cm\(^{-1}\), respectively. This demonstrates that the aroma oils of Lavender, Thyme, and Clove were first microencapsulated before reacting with polyester, and that -cyclodextrin molecules can form inclusion compounds with essential oils that fit into the cone-shaped hydrophobic cavity.

**Antioxidant activity.** Using the (1,1-diphenyl-2-picrylhydrazyl) DPPH free radical scavenging assay, the antioxidant capabilities of microencapsulated essential oils of lavender, thyme, and clove for use in finishing polyester fabrics were assessed for probable free radical pathways. The standards TBHQ (TertButylhydroquinone) (IC50 value 5.251.45 g/mL), the results showed that clove essential oil was superior antioxidant in the DPPH scavenging activity (IC50 value 0.5040.04 g/mL), followed by thyme (IC50 value 34.0160.06 g/mL) and lavender (IC50 value 250.0590.02 g/mL) essential oils.\(^{23}\) Free radical scavenging assays employing synthetic radicals such as DPPH (1,1-diphenyl-2-picrylhydrazyl), ABTS+ (2,2′-azinobis-3-ethyl benzothiozoline-6-sulfonic acid), or biological radicals such as superoxide radical anions to screen herbal drugs, food, and beverages for in vitro antioxidant activity. Antioxidants, such as phenolic substances, can defend against dangerous reactive oxygen species by inactivating them. The antioxidant activity of phenolic substances such as eugenol (clove), thymol, and carvacrol (thyme), as well as camphor in lavender essential oils,\(^{24}\) might be related to their redox characteristics, which allow them to operate as reducing agents, hydrogen donors, and singlet oxygen quenchers.

**Scanning electron microscope (SEM)**

Figure 8 shows a SEM image of polyester fabric after being treated with a solution containing the emulsified product of essential oils (particles of lavender, thyme, and clove essential oils). The surface of the treated polyester fabrics is uniformly covered by particles of the finishing agent, which contains the essential oils of lavender (a), thyme (b), and clove (c) with cyclodextrin, citric acid, and alcohol, as shown in the figures. When comparing the three figures, we notice that the surface of treated polyester with clove essential oil (Figure 8(c) is more homogeneous, whereas the surface of treated polyester with lavender and thyme essential oils (Figure 8(a) and (b) is covered by a large particle of the finishing fragrance used, possibly due to a reaction between the fragrances.

**Table 4.** The main chemical composition of clove essential oils analyzed by GC/MS.

| No. | Compound          | %     |
|-----|------------------|-------|
| 1   | Heptanone        | 0.02  |
| 2   | a-pinene         | 0.02  |
| 3   | p-cymene         | 0.02  |
| 4   | Limonene         | 0.05  |
| 5   | Heptyl acetate   | 9.27  |
| 6   | Linalool         | 1.81  |
| 7   | Methyl salicylate| 0.04  |
| 8   | p-allyl phenol   | 0.21  |
| 9   | Eugenyl acetate  | 0.8   |
| 10  | Eugenol          | 85.6  |
| 11  | Caryophyllene    | 0.49  |
| 12  | Caryophyllene oxide | 0.97 |
Figure 3. GC/MS of extracted Clove.

Figure 4. IR spectra of extracted essential oils. Lavender (red line), Thyme (black line) and Clove (blue line).
Antibacterial activity

Essential oils were found to have antibacterial activities against a variety of bacteria, including *Listeria monocytogenes*, *Listeria innocua*, *Salmonella typhimurium*, *Escherichia coli*, *Shigella dysenteriae*, *Bacillus cereus*, *Staphylococcus aureus*, and *S. typhimurium*.25 According to another study, 30 out of 60 essential oils had considerable inhibitory effect against *Helicobacter pylori*, bacteria linked to severe gastritis and a higher risk of peptic ulcers.26

Essential oils are said to have the following antibacterial properties in decreasing order: oregano > clove > coriander > cinnamon > thyme > mint > rosemary > mustard > cilantro/sage.27,28 After a 24-h incubation period, the antibacterial activity was measured by measuring the clear zone inhibition around the test sample. Figure 9 and Table 5 show the antibacterial capabilities of treated polyester fabric after finishing with essential oils of lavender, thyme, and clove, then drying at 100°C in the oven for 4 min and curing at 130°C for 3 min. On the examined microorganism, all of the substances tested had antibacterial action; however, none of them had antifungal activity. The differences in the diameter of the zone of inhibition reported between the polyester fabrics treated with lavender, thyme, and clove, then drying at 100°C in the oven for 4 min and curing at 130°C for 3 min. On the examined microorganism, all of the substances tested had antibacterial action; however, none of them had antifungal activity. The differences in the diameter of the zone of inhibition reported between the polyester fabrics treated with lavender, thyme, and clove could be attributable to bacterial susceptibility discrepancies between the samples. The antibacterial activity of treated polyester fabrics with thyme was the highest; this could be due to thyme’s
significant antioxidant effect. Furthermore, the examined samples are clearly efficient against *Staphylococcus aureus* (G+) growth that is higher than that of *Escherichia coli* (G−). This could be due to *E. coli*’s thick cell wall layer, which prevents antibacterial active substance from penetrating their cell walls and interfering with their metabolic system.

**Fragrance evaluation (fastness of fragrance to washing)**

A panel of five judges assessed the fragrance on the treated fabric before and after a certain number of washing cycles (Table 6). The judges scratched the fabric with their fingernails to break some of the capsules and smell the swatch. Then they wrote a “Yes” if there was a very strong, strong, medium, or light smell present, or a “No” if there was no fragrance present. As indicated in Table 5, all samples (regardless of the type of essential oil used) had a very strong aroma before washing. After 20 and 50 days, a strong fragrance was detected, (the storage conditions of samples at temperature 25°C and 1 atmospheric pressure), but after 60 days, or five washings, a medium scent was detected. The aroma was found on all samples even after five washings. After five washing, the perfume faded slightly due to a decrease in the fragrance concentration on
the fabric. Because β-cyclodextrin molecules are capable of building inclusion compounds with essential oils that fit into the cone-shaped hydrophobic cavity, the aroma was much stronger on samples with a higher number of capsules, regardless of the number of washing cycles.

**Conclusions**

Essential oils such as lavender, thyme, and clove are derived from natural sources and used to treat polyester fabrics to give them a pleasant odor and antibacterial qualities, that is, medicinal textiles. β-cyclodextrin molecules can combine with essential oils to generate inclusion compounds that fit into the hydrophobic cavity’s cone shape. The exhaustion approach may be used to successfully apply microcapsules containing essential oils of lavender, thyme, and clove to polyester materials. Antibacterial activity of treated polyester fabrics with essential oils, lavender, thyme, and clove against *Staphylococcus aureus* and *Escherichia coli*, that is medicinal textile. A strong
Table 5. Effect of lavender, thyme, and clove essential oils on antimicrobial properties of finishing polyester fabrics, dried at 100°C in the oven for 4 min and cured at 130°C for 3 min.

| Sample                             | Inhibition zone diameter (mm/1 cm sample) |
|------------------------------------|------------------------------------------|
|                                    | Escherichia coli (G⁻) | Staphylococcus aureus (G⁺) | Aspergillus flavus (Fungus) | Candida albicans (Fungus) |
| Untreated polyester                | 5.5                        | 5.2                        | 0.0                         | 0.0                        |
| Treated polyester fabrics with lavender. | 21                         | 23                         | 0.0                         | 0.0                        |
| Treated polyester fabrics with thyme.    | 23                         | 24                         | 0.0                         | 0.0                        |
| Treated polyester fabrics with clove. | 14                         | 14                         | 0.0                         | 0.0                        |

Figure 8. SEM image of treated polyester fabrics with (a) lavender, (b) thyme, and (c) clove essential oil, respectively.

Figure 9. The antibacterial and fungus capabilities of treated polyester fabric.
smell was discovered after 50 days, whereas a medium aroma was discovered after 60 days or five items of washing. The originality of this study stems from its capacity to meet the economic feasibility of medical textiles, which are more expensive than conventional textiles, while still achieving the goal of antibacterial good odor fabrics.

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