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Recent Advances in Textile Functionalization Using Essential Oil-Based-Microcapsules with Antimicrobial Properties

Nedavni napredek funkcionalizacije tekstilij z mikrokapsulami na osnovi protimikrobnih eteričnih olj

Scientific review/Pregledni znanstveni članek

Received/Prispelo 2–2025 • Accepted/Sprejeto 3–2025

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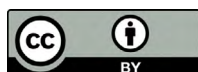
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Abstract

Antimicrobial textiles are functionalized textiles designed to inhibit or terminate the growth of microorganisms. In light of the increasing emphasis on eco-friendly processes, the application of essential oils presents a viable alternative to synthetic drugs (antibiotics). The aim of this study was to evaluate recent advances in microencapsulation methods of essential oils with antimicrobial activity that can be applied on medical textile for dermal use by employing the PRISMA methodology. Essential oils have been microencapsulated using various methods: coacervation, spray-drying, emulsion method and *in situ* polymerization. Among these, coacervation is still extensively utilized, though associated scale-up challenges persist. Many essential oils have demonstrated antibacterial properties against Gram-positive (*Staphylococcus aureus*, *Bacillus subtilis*) and Gram-negative (*Pseudomonas aeruginosa*, *Escherichia coli*, *Klebsiella pneumoniae*) bacteria, as well as antifungal activity (*Candida albicans*). The growth inhibition of these microorganisms was assessed in the presence of the following essential oils and their active substances with the highest biological-antimicrobial activity: cinnamon (trans-cinnamaldehyde), lime (α -terpineol, terpineol, and limonene), tea tree (terpinen-4-ol), rosemary (1,8-cineole and α -pinene), peppermint (l-menthol, menthone, methyl acetate and limonene), lavender (linalool and linalyl acetate), thyme (carvacrol) and clove (eugenol). The findings indicate that functionalized textile with microcapsules exhibits enhanced antibacterial activity against Gram-positive bacteria compared to Gram-negative bacteria (*Escherichia coli*), which could be attributed to the bacteria's thick wall. However, there is a notable lack of data regarding cytotoxicity and the sensory evaluation of functionalized textile. The potential utilization of essential oils was explored in the development of eco-friendlier functionalized textile with antimicrobial



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properties. However, additional research is required to maximize the antimicrobial activity of microcapsules to overcome challenges in the scale-up to pilot process, and to improve the immobilization in textiles.

Keywords: antimicrobial properties, sustainability, essential oils, microcapsules, textile functionalization

Izvleček

*Protimikrobne tekstilije so funkcionalizirani materiali, zasnovani za zaviranje ali preprečevanje rasti mikroorganizmov. Ker si prizadevamo za uporabo okolju prijaznih tehnologij, je uporaba eteričnih olj mogoča kot alternativa sintetičnim zdravilom, kot so antibiotiki. Namen raziskave je bil oceniti razvoj metod mikroenkapsuliranja eteričnih olj s protimikrobnim delovanjem, ki jih je mogoče s pomočjo metodologije PRISMA uporabiti pri pripravi medicinskih tekstilij za dermalno uporabo. Eterična olja so bila mikrokapsulirana z različnimi tehnikami, vključno s koacervacijo, sušenjem z razprševanjem, emulzijsko metodo in polimerizacijo in situ. Med omenjenimi metodami je koacervacija še vedno najpogostejše uporabljena kljub izzivom, povezanim z razširitvijo procesa na industrijsko raven. Posamezna eterična olja izkazujejo širokospektralno protibakterijsko delovanje proti grampozitivnim (*Staphylococcus aureus*, *Bacillus subtilis*) in gramnegativnim bakterijam (*Pseudomonas aeruginosa*, *Escherichia coli*, *Klebsiella pneumoniae*) ter protiglivično učinkovitost proti *Candida albicans*. Najvišjo biološko-protimikrobno aktivnost so pokazale aktivne snovi eteričnih olj cimeta (*trans*-cinamaldehyd), limete (*α*-terpineol, *terpineol*, *limonen*), čajevca (*terpinen-4-ol*), rožmarina (*1,8-cineol*, *α*-*pinen*), poprove mete (*L*-*mentol*, *menton*, *metil acetat*, *limonen*), sivke (*linalol*, *linalil acetat*), timijana (*karvakrol*) in klinčkov (*evgenol*). Ugotovljeno je bilo, da mikrokapsulirane funkcionalizirane tekstilije izkazujejo učinkovitejše protibakterijsko delovanje proti grampozitivnim bakterijam kot proti gramnegativnim, kar je mogoče pripisati razliki v strukturi celične stene mikroorganizmov. Kljub spodbudnim rezultatom pa so podatki o citotoksičnosti in senzoričnih lastnostih protimikrobnih tekstilij še vedno omejeni. Potrebne so nadaljnje raziskave za povečanje protimikrobne učinkovitosti mikrokapsul, optimiziranje postopkov industrijske izdelave in izboljšanje vezave mikrokapsul na tekstilna vlakna.*

Ključne besede: protimikrobne lastnosti, trajnost, eterična olja, mikrokapsule, funkcionalizacija tekstilij

1 Introduction

Antimicrobial resistance (AMR) is one of the major global public health threats of the 21st century, and is characterized by the reduction in the efficacy of antibiotics [1]. AMR has been exacerbated by COVID-19 pandemic, due to the over prescription of antimicrobial agents by physicians and the easy availability of over-the-counter (OTC) antibiotics in pharmacies and drug stores [2]. According to the CDC's July 2024 Report on Antimicrobial Resistance Threats, AMR increased by 20% during the COVID-19 pandemic relative to the pre-pandemic period [2]. Globally, bacterial infections on the skin and subcutaneous tissues rank as the sixth leading infectious syndrome contributing to the mortality associated with AMR [3].

Natural products, particularly essential oils (EOs), are potential candidates to combat AMR due to their antioxidant and pro-oxidant properties [4]. EOs are plant extracts derived from various parts such as petals and flowers, grasses, seeds, leaves, stems, roots and rhizomes, woods and resins [5]. According to the European Pharmacopoeia (Ph. Eur.) and to the Association Française de Normalisation, an essential oil is defined as a "product obtained from a natural raw materials of plant origin, either through distillation using water or steam, a mechanical process from the epicarp of *Citrus* sp. fruits or through dry distillation [6,7]. The essential oil is separated by physical means from the aqueous phase. EOs are multicomponent systems contain-

ing a variety of volatile, lipophilic and odoriferous chemical compounds, including terpenes, alcohols, sesquiterpenes, amides, phenols, acids, ketones, aldehydes, esters, ethers and oxides [8]. EOs have been extensively investigated for their biological activities [9], including antibacterial properties. Active substances, with natural antimicrobial properties are derived from thyme (*Thymus vulgaris*), oregano (*Origanum compactum*), clove (*Eugenia caryophyllata*), mint (*Mentha piperita*), sage (*Salvia officinalis*), lavender (*Lavandula angustifolia*) and others (Figure 1).

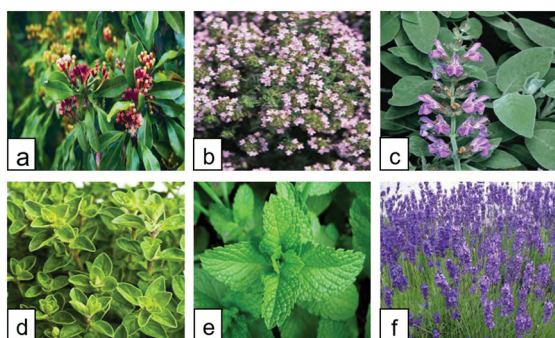


Figure 1: Species from which essential oils with antimicrobial properties can be extracted: a) *Eugenia caryophyllata* (<https://www.pioneerherbal.com>), b) *Thymus vulgaris* (<https://plants.ces.ncsu.edu>), c) *Salvia officinalis* (<https://www.greensmile.cy>), d) *Origanum compactum* (<https://aliksir.com>), e) *Mentha piperita* (<https://www.la-saponaria.com>) and f) *Lavandula angustifolia* (<https://www.seedscape.net.au>)

Due to their fragrance, EOs are widely explored in the textile industry and employed as natural antibacterial agents [10, 11]. Antibacterial activity has been demonstrated against various bacterial strains, including *Escherichia coli*, *Shigella dysenteriae*, *Listeria monocytogenes*, *Bacillus cereus*, *Salmonella typhimurium* and *Staphylococcus aureus* [12]. EOs are thus broadly used in research and development laboratories to design value-added textiles with cosmetic and medical applications. Three groups of antimicrobial textiles can be distinguished: antibacterial textiles that inhibit bacteria growth, antifungal textiles that prevent fungal mycelium and spore germination, and antiviral textiles that modify the virus surface structure [13]. Although commercial antimicrobial products have been developed, the most efficient compounds (silver nanoparticles, triclosan and quaternary ammonium compounds) are regulated under Regulation 528/2012 [14].

While the shift towards sustainability is still in its early stages, consumers increasingly prefer eco-friendly choices. In light of this trend and the looming threat of AMR, the application of EOs can be considered an alternative to synthetic drugs (antibiotics), as it was the case in ancient times. The earliest references to the use of sandalwood and cinnamon essential oils date back to ancient Hindu scriptures called Vedas [15]. The Egyptians used plants for medicinal purposes, surgery, food preservation, mummification and healing practices or massages [16]. A graphical illustration is presented in Figure 2.

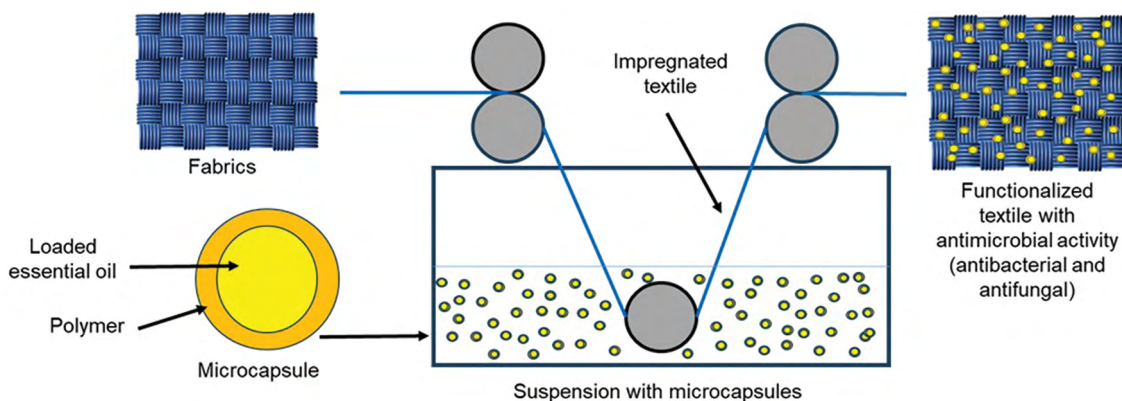


Figure 2: Graphical illustration of medical textile functionalization with microcapsules

However, the main disadvantage of EOs is their susceptibility to environmental conditions (oxygen, light, temperature and humidity), making them prone to decomposition and easy volatilization [17]. These drawbacks can be mitigated through technological and/or formulation modification in the microencapsulation process.

The potential use of microencapsulation technology for encapsulating EOs in medical textile has been previously discussed [18]. Nevertheless, not all microencapsulation methods are suitable for textile applications. Commonly used techniques in textile functionalization are:

- physical: spray-drying and solvent evaporation,
- physico-chemical: simple and complex coacervation and molecular inclusion, and
- chemical: *in situ* polymerization, photopolymerization and interfacial polymerization.

This study addresses recent developments in microencapsulation methods for EOs, types of EOs with antimicrobial activity, textile functionalization methods with microcapsules and tested microorganism strains. The focus is on summarizing the antimicrobial activity of various EOs, the production of microcapsules, and their use in creating eco-friendly, biocompatible, and nontoxic functional antimicrobial textiles with biological, aromatherapeutic and antioxidant properties.

2 Methodology

A comprehensive literature search was conducted using the Scilit database, focusing on the keywords “essential oil”, “antimicrobial activity”, “fabrics” and “antibacterial activity”. The selection criteria included studies published between 2014 and 2024, and was limited to articles in English. Both peer-reviewed journal articles and ‘grey’ literature, such as conference papers, were included. Eligible studies were required to report antimicrobial efficiency testing, microencapsulation methods and textile functionalization techniques with a clear research purpose. For purposes of screening and selection,

the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) was followed. Evaluation was performed based on ‘title and abstract’, followed by qualitative content analysis by two reviewers. Any discrepancies between the reviewers were resolved by triple-checking the articles and through discussion with a third reviewer. After selecting eligible articles, obtained results and cited studies were screened for inclusion. Data extracted from the included studies encompassed microencapsulation techniques, types of essential oils, microorganism strains, types of textile/fabrics and functionalization methods. Due to absence of a standardized quality tool for the assessment of studies involving functionalized textiles with antimicrobial activity, articles with clearly presented relevant data were considered for evaluation.

3 Results

3.1 Results from the qualitative literature search

The following parameters were included in the prior advanced tool search: publication period (2014–2024) and English language. All journal articles that had results on antimicrobial activity of microcapsules and a lack data on antimicrobial activity of functionalized textile alongside textile functionalization were excluded. Book chapters, preprints and review articles were also excluded. A total of 84 studies were identified from the Scilit database with three duplicates. The screening of the title and abstract led to the exclusion of fifteen articles, while eight more were excluded after full-text screening. The final number of relevant studies was 16. The full selection process and outcomes are summarized in Figure 3.

3.2 Characteristics of included studies

Details of included studies and their outcomes are presented in Table 1. Five studies reported [19–21, 24, 32] that their microcapsules were obtained through coacervation, two using the spray-drying process [28, 34], seven using the emulsion method [23, 25–27,

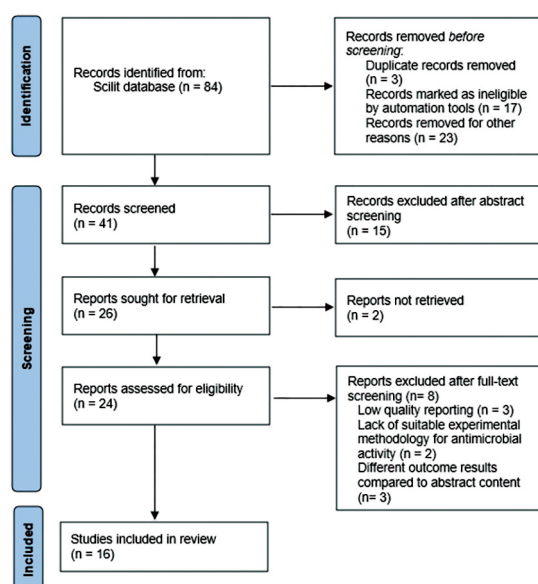


Figure 3: Qualitative literature search results

29–31] and one using the *in situ* polymerization method [33], while one article [22] did not provide data regarding the microencapsulation method. In one study [31], microcapsules with thyme essential oil were obtained as a commercial product. Essential oils from cinnamon [19, 27] and clove [29, 34] were each reported in three studies. Lime [20, 24] and tea tree [21, 22] essential oils were each reported in two studies. Thyme [31], ginseng [30], peppermint [32], eucalyptus [26] and sandalwood [26], and rosemary [28] essential oil were each reported once. The majority of the textile functionalization methods (Table 1) included padding ($N = 4$), pad-dry-cure ($N = 4$), pad-dry method ($N = 2$), printing, finishing, *in situ* procedure and exhaustion process ($N = 1$, each), while one study covered the fibre spinning method. The most commonly tested bacteria were *Staphylococcus aureus* (Gram positive) and *Escherichia coli* (Gram negative). Additionally, antibacterial efficiency against the microorganisms *Klebsiella pneumoniae*, *Staphylococcus epidermis*, *Bacillus cereus* and *Salmonella typhimurium* was assessed. In one article, antifungal activity against *Candida albicans* was evaluated. Cotton fabrics ($N = 9$) were predominantly investigated, followed by viscose fibres, cellulosic fibres, nylon-polyurethane fabrics, linen fabrics, PLA

fabrics and polyester fabrics ($N = 1$, each). Research studies that elucidated both microencapsulation, followed by textile functionalization and extensive characterization (washing durability, sensory evaluation, biocompatibility study and cytotoxicity effects), are limited.

4 Discussion

Results from the systematic review suggest that functionalized textile with essential oil-based microcapsules possess antimicrobial activity. This is further confirmed by different previously conducted literature-scientific reviews that are not part of this systematic review [18, 35–37]. Based on the review analysis, the coacervation method, one of the oldest methods, is widely explored in microencapsulation studies. However, the commercialization of coacervation is hindered by high-costs and a time-intensive multistep manufacturing process (polymer hydration, emulsification, coacervation, shell hardening and drying). The reviewed articles did not provide explicit data regarding advances in industrially feasible and scalable coacervation methods. Therefore, further studies are required to design an industrially scalable coacervation process following up on the work performed by Tang [38].

Considering technological advances, eco-friendlier trends and the optimization of resources, Sharma and Chakraborty [34] optimized process parameters in the spray-drying method using the Design of Experiments approach (DoE), employing the Box-Behnken and Central composite design. This research methodology adds value and should encourage researches to employ DoE in their studies. Beşen [22] suggested that optimization in technological and formulation parameters should be carried out to achieve equally high antibacterial activity against strains, which could be further achieved by using the DoE approach.

Although polyester fibre made of poly(ethylene terephthalate) holds the highest market share ($> 50\%$) in the textile industry, the reviewed data showed that

Table 1: Summary of textile functionalization using microcapsules with antimicrobial activity

Microencapsulation technique	Wall material(s)	Essential oil	Microorganism strains	Functionalization method	Type of fabrics	Ref.
Coacervation	Chitosan	Cinnamon	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Padding	Cellulosic fibres surface	[19]
Coacervation	Alginate and gelatine	Lime	<i>Staphylococcus aureus</i> , <i>Escherichia coli</i> , <i>Klebsiella pneumoniae</i> and <i>Staphylococcus epidermidis</i>	Pad-dry-cure	Cotton fabrics	[20]
Coacervation	Poly(vinyl)-alcohol, gum Arabic and β -cyclodextrin; ethyl-cellulose	Tea tree	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Padding	Cotton fabrics	[21]
No data	Ethyl Cellulose	Tea tree	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Padding	Cotton fabrics	[22]
Emulsion	β -cyclodextrin	Lavender, thyme and clove	<i>Staphylococcus aureus</i> and <i>Escherichia coli</i>	Exhaustion	Knitted polyester fabrics	[23]
Complex coacervation	Chitosan and gum Arabic	Lime oil	<i>Escherichia coli</i> , <i>Staphylococcus aureus</i> , <i>Bacillus cereus</i> and <i>Salmonella typhimurium</i>	Dipping	Cotton fabrics	[24]
Emulsion	Chitosan and β -cyclodextrin	Cinnamon, lavender, thyme and savory	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	<i>In situ</i> procedure	PLA fabric	[25]
Emulsion	Chitosan	Eucalyptus; Sandalwood	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Pad-dry-cure	Cotton fabrics	[26]
Emulsion	Chitosan	Cinnamon + propolis	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Padding	Knitted cotton textile	[27]
Spray-drying	Chitosan-gelatine complex	Rosemary	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Pad dry method	Linen fabric	[28]
Spray-drying	Chitosan-gelatine complex	Rosemary	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Pad dry method	Linen fabric	[28]
Emulsion	Chitosan	Clove	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Pad-dry-cure	Cellulosic fabric	[29]
Emulsion	Melamine-formaldehyde prepolymer	Ginseng oil	<i>Staphylococcus aureus</i> , <i>Klebsiella pneumoniae</i>	Pad-dry-cure	Nylon-polyurethane fabric	[30]
Simple coacervation	Arabic gum	Peppermint oil	<i>Escherichia coli</i>	Finishing process	100% cotton denim fabric	[32]
<i>In situ</i> polymerization method	Melamine-formaldehyde polymer	Lavender, rosemary and sage	<i>Escherichia coli</i> and <i>Staphylococcus aureus</i>	Printing	100% cotton woven fabric	[33]
Spray-drying	Chitosan	Clove oil	<i>Bacillus sp.</i> and <i>Escherichia coli</i>	Pad-dry method	Cotton fabrics	[34]

majority of functionalized textile are cotton fabrics. Cotton fabric is preferred to synthetic fabric as it is biodegradable and naturally occurring. The functionalization of such fabrics with bio-based, green and renewable antibacterial molecules will help improve environmental sustainability.

It is evident from the reviewed articles that *Escherichia coli*, as Gram-negative bacteria, and *Staphylococcus aureus*, as Gram-positive bacteria, were mainly evaluated. Research has proven that functionalized textiles with EO-based-microcapsules exhibit antibacterial activity against *Staphylococcus aureus*. However, the lower inhibition of *Escherichia coli* could be observed in certain studies. This can be attributed to *Escherichia coli*'s thick cell wall, which hinders the penetration of antibacterial compounds.

Despite extensive research on the antimicrobial potential of EOs as a green alternative to antibiotics in engineered-textile, their toxicological effects are still insufficiently investigated. This research area is crucial for the commercialization of EOs-based-functionalized textile, and for meeting requirements for permitted daily exposure contained in the EMA Guidelines (2014) [39] on setting health-based exposure limits for use in risk identification in the manufacture of different medicinal products in shared facilities. Affygility Solutions' catalogue includes monographs for lavender, peppermint and coconut oil, suggesting that the industrial scale-up process of microencapsulation and functionalization methods of textiles are challenging from technological, economical, toxicological and health-based perspectives.

The technological limitations of applied microencapsulation techniques in textile functionalization are mainly attributed to one critical quality attribute (CQA): the particle size distribution of microcapsules. In functionalized-medical textiles, this CQA is directly correlated with a patient's acceptance. More attention must thus be paid to sensory evaluations, which currently lack extensive results. Since EOs are multicomponent systems, the quantitative analysis of assay poses another challenge in commercializing EOs-based-textiles.

5 Conclusion

The microencapsulation of volatile EOs is widely investigated as a promising technique for designing textile with functional properties. Their application in fabric functionalization to produce medical textiles has been intensively explored due to eco-orientated consumerism. This study highlighted the use of essential oils such as cinnamon, lime, peppermint, thyme, lavender, clove and tea tree.

Coacervation is the most commonly employed method for essential oil microencapsulation in textile functionalization, followed by emulsion and spray-drying techniques. Published results indicate durable antimicrobial efficacy against both typical Gram-positive and Gram-negative bacteria, with higher efficacy observed against Gram-positive (*Staphylococcus aureus*) bacteria. One commonly employed textile functionalization method is the pad-dry-cure method.

Functionalized textiles embedded with microcapsules containing essential oils represent a significant potential for applications in medical treatments. Specifically, in the context of dermal wound healing, such textiles can serve as effective agents for the prevention or inhibition of infections and inflammation. Additionally, microcapsules exhibiting antifungal efficacy may be incorporated into athletic socks to reduce the incidence of fungal infections. Furthermore, medical textiles designed with immobilized microcapsules endowed with antimicrobial properties hold promise for the development of advanced military apparel, offering protection against microbial proliferation and the associated risks of infection.

The gaps that remain in the broad and diverse disciplines conducting research on antimicrobial textile with essential oil-based microcapsules must be narrowed over time. Further research arises from identified gaps in the field of textile functionalization, and is highlighted below:

1. The sensory evaluation and dermatological testing of medical textiles with immobilized

microcapsules must be performed in future research studies. If needed, further optimization on the particle size distribution or the overall technological process should be carried out. Such studies would increase the scientific worth of antimicrobial textiles. Dermatological testing should be performed in future studies to assess whether a product causes irritation and inflammation when in contact with the skin.

2. Toxicological studies of essential oils are extremely limited. Scientific-based toxicological studies of essential oils with proven antimicrobial activity must be performed. This would be a prerequisite in setting the dosage in antimicrobial textiles.
3. Studies focusing on release kinetics, mechanism of release, EO concentration and microencapsulation optimization, as well as the functionalization process, optimization and transdermal delivery, are not focused on evaluating the overall benefit of such technological development and the “therapeutic” benefit of value-added textile.
4. Performing stability studies of microcapsules and functionalized textile using microcapsules to set the shelf-life of finished products (functionalized textile). Until now, no literature has been identified that investigates for how long microcapsules maintain their antimicrobial efficacy.

Microcapsule textile functionalization is undoubtedly a challenging task for scientists but is expected to receive more attention in future. To overcome current knowledge gaps, an interdisciplinary approach in research groups is essential for the commercialization of antimicrobial textiles with essential oil-based microcapsules.

Authors' contributions: conceptualization: T.G., S.T., K.A., K.L.; methodology T.G.; data curation T.G., S.T., K.A.; writing-original draft preparation: T.G., S.T.; writing-review and editing K.A., K.L.; visualization S.T.; supervision K.L.

Conflict of Interest: The authors declare no conflict of interest.

Data availability statement: All data analysed in this review were obtained directly from publicly accessible Scilit database using their readily available filters under advanced search.

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