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APPLICATION OF *IN-SITU* HYDROTHERMAL SYNTHESIS FOR THE FUNCTIONALISATION OF COTTON/POLYESTER FABRIC WITH THE INCLUSION COMPLEX OF Β-CYCLODEXTRIN AND ESSENTIAL TEA TREE OIL

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Textiles are often used as a medium for the application of active substances through various processes to achieve antimicrobial and wellness properties for medical and cosmetic purposes. The unique structure of β-cyclodextrins, which enables the formation of inclusion complexes, has led to significant commercial applications in areas such as pharmaceuticals, cosmetics, and the textile industry. A key advantage of β-cyclodextrins is their environmental friendliness — they are biodegradable, non-toxic, and do not pollute wastewater systems. This study investigated the formation of inclusion complexes between β-cyclodextrin and tea tree essential oil and their binding to cotton/polyester fabrics by *in situ* hydrothermal synthesis. Part of the treated fabrics was subjected to a care procedure according to ISO 6330:2012, using the 6330 6N programme and the standard ECE detergent (WFK 88030) to evaluate the durability of the treatment. Changes at the physico-chemical level before and after washing were analysed using FTIR-ATR spectroscopy, while absorption and antimicrobial properties were tested on untreated, treated, and washed treated fabrics, taking into account their intended use in hospital environments. The results indicate effective binding of the β-cyclodextrin tea tree oil inclusion complex to the cotton/polyester fabric by *in situ* hydrothermal synthesis, with the treatment being remarkably durable after laundering. These results emphasise the potential for further research to develop functional, high-performance advanced textiles for use in medical fields.

Keywords: cotton/polyester fabrics, inclusion complexes between β-cyclodextrin and tea tree essential oil, *in situ* hydrothermal synthesis

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INTRODUCTION

The textile industry is currently undergoing a significant transformation towards sustainability and improved functionality. There is a growing demand for high-performance textile materials that go beyond the basic function of clothing and offer additional benefits to the end user. Functional textiles represent an intersection of textile technology, chemistry, and biotechnology, with a focus on solutions that improve the interaction between textiles and human skin. Of particular interest are cosmetotextiles – textile products with integrated cosmetic effects, such as skin moisturisation, soothing, or antibacterial protection [1, 2]. In this context, essential oils play an important role due to their bioactive properties, although their application is often challenged by their volatility and sensitivity to environmental conditions [3]. Tea tree essential oil (Melaleuca alternifolia) is known for its strong antibacterial, antiviral, and antifungal effects [4,5]. Due to their high volatility and susceptibility to external influences, essential oils require special protection and controlled release methods. One of the most effective approaches is the formation of inclusion complexes with cyclodextrins, especially β-cyclodextrin [6]. This cyclic oligosaccharide has a hydrophilic outer surface and a hydrophobic inner cavity, which makes it an ideal carrier for volatile, hydrophobic molecules such as essential oils. The inclusion complex increases stability, prolongs the bioactive effect, and enables the controlled release of active ingredients. In combination with textile substrates — especially cotton/polyester blends — innovative materials with a wide range of functional properties can be developed. Textiles treated in this way can have antibacterial, deodorising, UV-protective, and anti-inflammatory effects [5].

This study focuses on the application of *in-situ* hydrothermal synthesis as an environmentally friendly method for the functionalisation of textiles [6]. This process involves the direct formation of the β-cyclodextrin/tea tree oil inclusion complex on the textile surface under elevated temperature and pressure. The binding of β–β-cyclodextrin to cotton/polyester fabric, primarily to the cellulose component in the given blend, can be achieved through the application of polycarboxylic acids as crosslinking agents. The most extensively studied is 1,2,3,4–1,2,3,4-butanetetracarboxylic acid (BTCA), which demonstrates durable binding of the β–β-cyclodextrin inclusion complex primarily with cellulose in cotton/polyester blends, mainly due to the reactivity of its functional groups. The process is based on a two–step esterification, where in the first step a cyclic anhydride is formed, followed by a reaction with the hydroxyl groups of cotton and/or cyclodextrin in the second step. In this way, ester bonds are formed, ensuring the durability of the treatment even after multiple washing cycles [7].

Hydrothermal synthesis reduces the need for organic solvents and minimises the environmental impact. At the same time, it ensures high reproducibility of the treatment and permanent functionalisation of the textiles. The use of β-cyclodextrin also supports sustainability, as it is a non-toxic and biodegradable compound [6, 7, 8].

Incorporation into an inclusion complex makes the tea tree oil more stable and resistant to external influences, which prolongs its functional activity [5]. The application of such functional textiles is particularly important in the fields of medicine, sports, and protective textiles. It also enables the development of everyday clothing with added value for the wearer. Cotton-polyester fabrics have proven to be a suitable substrate due to their balanced ratio between natural and synthetic properties, and the possibility of fixing the inclusion complex to the fibres further expands the application potential [9]. The use of cross-linking agents ensures long-lasting functionality, even after several care cycles. This technology paves the way for the development of textiles that not only protect the skin, but also actively contribute to health and the comfort of the wearer. The combination of *in-situ* synthesis, natural bioactive compounds, and cyclodextrins provides a sustainable basis for a new generation of intelligent textile materials.

MATERIAL AND METHODS

For the research presented in this paper, the following fabrics were used: cotton/polyester (50%/50%) in satin weave with a weft density of 26 threads cm^{-1}, and cotton/polyester (50%/50%) in plain weave with a weft density of 20 threads cm^{-1}. The description of the samples and their designations are provided in Table 1.

Table 1. Specification and labeling of samples

Sample	Label
Cotton/polyester blend in plain weave, untreated sample	P_PES_P
Cotton/polyester blend in plain weave, treated with β-cyclodextrin–tea tree essential oil at 70 °C in a bath	P_PES_P_BCD_TT
Cotton/polyester blend in plain weave, treated with β-cyclodextrin–tea tree essential oil at 70 °C in a bath, and washed sample after treatment	P_PES_P_BCD_TT_W
Cotton/polyester blend in satin weave, untreated sample	P_PES_A
Cotton/polyester blend in satin weave, treated with β-cyclodextrin–tea tree essential oil at 70 °C in a bath	P_PES_A_BCD_TT
Cotton/polyester blend in satin weave, treated with β-cyclodextrin–tea tree essential oil at 70 °C in a bath, and washed sample after treatment	P_PES_A_BCD_TT_W

The inclusion complex of β-cyclodextrin (CycloLab R&D Ltd.) with tea tree essential oil (Sigma Aldrich) was prepared by applying mechanical force using a Retsch MM 400 vibrating mill, with 50% essential oil added in proportion to the mass of β-cyclodextrin. The mechanical-chemical synthesis was carried out at a frequency of 25 Hz for 10 minutes. Subsequently, the β-cyclodextrin-tea tree oil inclusion complex was heated in a microwave oven at a power of 80 W for 3 minutes to promote the reaction and further dry the complex itself [10]. The prepared β-cyclodextrin inclusion complexes with tea tree essential oil were used to functionalise the above substances by an impregnation process with a padding effect of approximately 100%, followed by in situ synthesis in a bath with the following composition:

- 70 g/L 1,2,3,4-butanetetracarboxylic acid (BTCA) (Sigma Aldrich)
- 65 g/L sodium hypophosphite monohydrate (SHP) (Sigma Aldrich)
- 1 g/L Felosan RG-N (nonionic wetting agent) (Bezema)
- 50% β-cyclodextrin–tea tree essential oil complex relative to the fabric weight

To ensure the binding of the inclusion complexes to the cotton/polyester fabrics, 1,2,3,4-butanetetracarboxylic acid (BTCA) was added to the bath, with sodium hypophosphite monohydrate (SHP) serving as the catalyst.
The impregnated samples, together with the bath, were placed in a Teflon-covered container and kept in a drying oven at 70 °C for 20 hours. The bath pH prior to oven treatment was 3.0, while after the reaction in the oven, the pH decreased to 2.76.
Upon completion of the reaction, the samples were removed and padded using a laboratory padder (Benz, TKF 15/M350 + LFV/2 350R). The samples were then air-dried. Thermocondensation of the samples was performed in a laboratory oven (Benz, TKF 15/M350 + LFV/2 350R) at 180 °C for 90 seconds.
A portion of the treated and thermocondensed samples was subjected to washing. Washing was carried out in an industrial washing machine (Wascator, Electrolux) in accordance with ISO 6330:2012 standard, program 6330 6N, using ECE standard detergent without optical brighteners but with phosphates (WFK 88030) at 75 °C for 80 minutes. The washing process was performed to evaluate the durability of the treatment. After washing, the samples were air-dried.
Physico-chemical changes in cotton samples were investigated using Fourier transform infrared spectroscopy in the attenuated total reflectance technique (FTIR-ATR) (Perkin Elmer, Spectrum 100 software). Four scans with a resolution of 4 cm^{-1} between 4000 cm^{-1} and 380 cm^{-1} were performed for each sample, and the spectra obtained were processed using the Spectrum 100 software package, Perkin Elmer.
The moisture transfer capability of untreated and treated samples was evaluated according to the AATCC TM 195 – 2017 method. Moisture absorption is an essential property of textile materials, as it significantly influences the thermo-physiological comfort of clothing during wear. In general, cotton fiber is considered a hygroscopic and hydrophilic fiber. The equilibrium moisture content (regain) of cotton is approximately 8.5%, indicating its high moisture absorption capacity. Due to these properties, cotton fiber is less prone to static electricity accumulation, which further contributes to the comfort of garments made from cotton [11]. To evaluate the moisture transfer behavior of both untreated and treated samples, tests were conducted using a Moisture Management Tester (MMT, ATLAS). Untreated and treated fabric samples were cut to dimensions of 6 × 6 cm and conditioned for 24 hours at 21 ± 1 °C and 65 ± 2% relative humidity. During testing, the textile specimen was placed flat between two horizontal, concentric sensors (upper and lower). A precisely defined amount of testing solution—used to monitor changes in electrical conductivity—was dispensed at the center of the specimen, with the face side positioned against the upper sensor. The test solution can move in three directions: Radial spreading on the face side, Vertical penetration through the fabric, and Radial spreading on the back side. Throughout the test, changes in the electrical resistance of the specimen were continuously recorded, and a summary of the measured parameters was used to evaluate the fabric's moisture transfer capability [12]. A single test cycle lasts 2 minutes and provides data on the following parameters: overall moisture management capacity (OMMC), wetting time, wetting speed, maximum wetted radius, absorption rate, and the accumulated one-way transport index [13].
The antimicrobial efficacy of the samples before treatment, after treatment, and following the washing cycle was evaluated in accordance with the AATCC 147 method,

with modifications recommended by experts. The samples were prepared under sterile conditions and subjected to testing. Each sample, in triplicate, was exposed to the action of the bacteria *Staphylococcus aureus* and *Escherichia coli* at concentrations of 10^8–10^6 bacteria/mL of nutrient agar (Colony Forming Units, CFU/mL), as well as the fungus *Candida albicans*. The treatment effectiveness was assessed using a qualitative method based on the formation of an inhibition zone, i.e., an area in which the antimicrobial agent from the tested substrate eliminated microorganisms present on the nutrient agar [14]. Achieving inhibition according to this method represents a highly stringent requirement imposed on treated textile materials.

RESULTS AND DISCUSSION

The results obtained from the Fourier Transform Infrared Spectroscopy with Attenuated Total Reflectance (FTIR-ATR) are presented in Figures 1 to 4.
In Figures 1 and 2, the spectral bands of tea tree essential oil and β-cyclodextrin are shown separately, with labeled peaks to facilitate identification of changes in specific wavelength ranges of the treated and untreated samples.

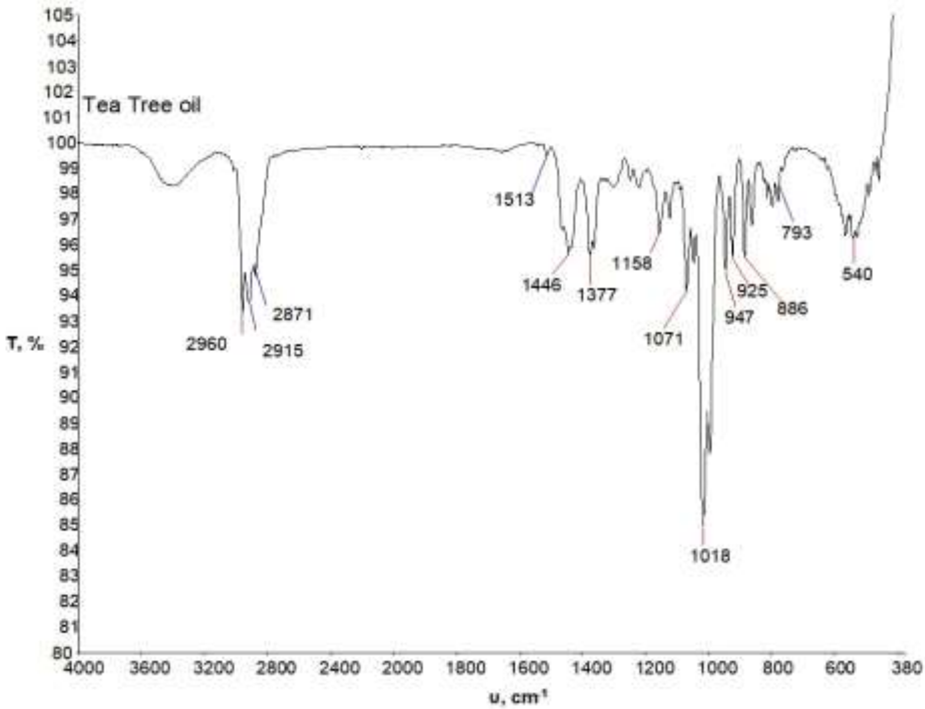

Figure 1. Spectral curve of tea tree essential oil recorded by FTIR-ATR.

The pronounced peaks observed at 2960 cm⁻¹, 2915 cm⁻¹, and 2871 cm⁻¹ correspond to C–H stretching vibrations. The strongly expressed peak at 1018 cm⁻¹ indicates C–C, C–OH, and C–H ring vibrations. The peaks at 886 cm⁻¹ and 793 cm⁻¹ suggest the presence of terpinen-4-ol [15].

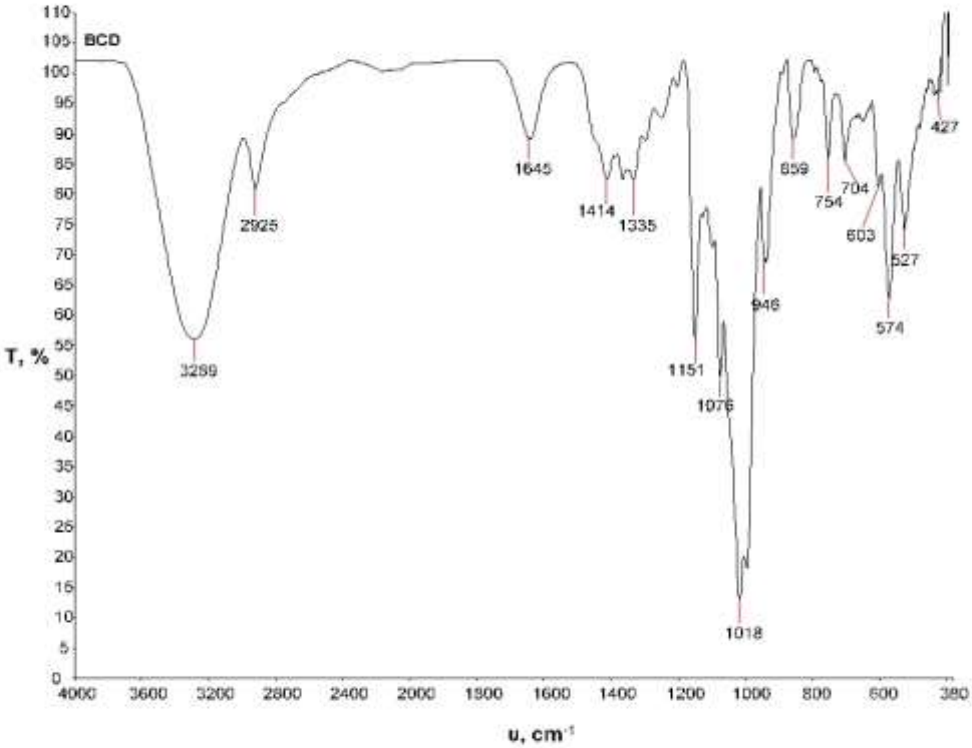

Figure 2. Spectral curve of β-cyclodextrin (BCD) recorded by FTIR-ATR.

The peak at 3289 cm⁻¹ is attributed to the accumulation of water molecules within the hydrophilic cavities of the cyclodextrin molecule. The peak at 2925 cm⁻¹ is associated with C–H and O–H stretching vibrations of cyclodextrin. The peak at 1018 cm⁻¹ corresponds to C–H and C–O–C stretching vibrations, while the peak at 1645 cm⁻¹ arises from O–H stretching of adsorbed water [16].

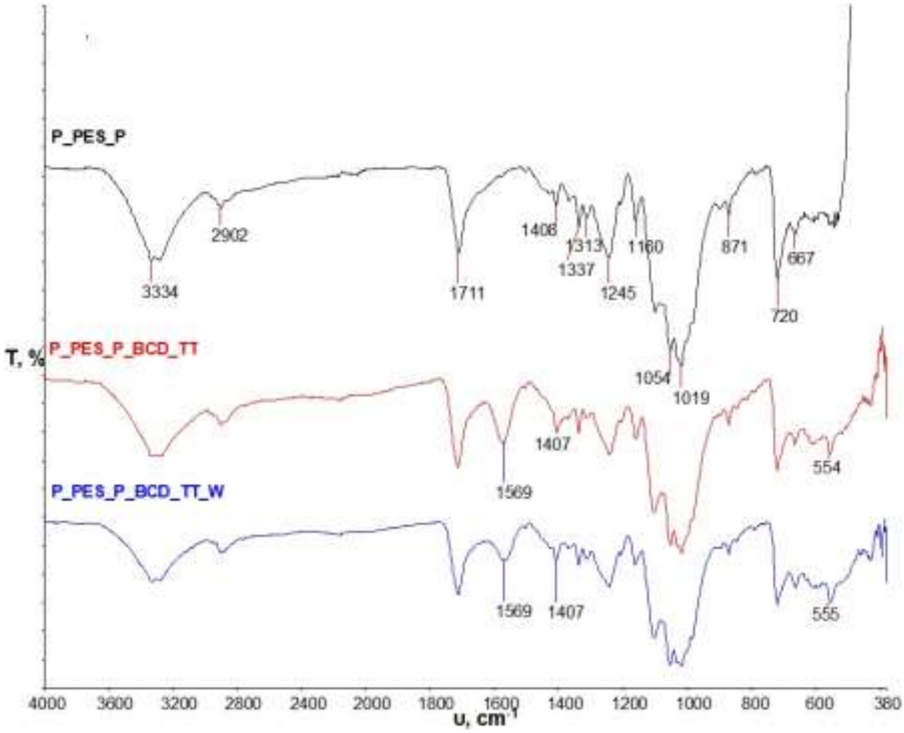

Figure 3. Spectral curves of the untreated (P_PES_P), treated (P_PES_P_BCD_TT), and treated washed sample (P_PES_P_BCD_TT_W) of the cotton/polyester fabric in plain weave.

In all the samples of cotton-polyester blends analysed, both in satin and plain weave, a sharp peak is observed at 1711 cm^{-1} (Figures 3 and 4), which is caused by vibrations within the ester groups present in the polyester. Furthermore, in the treated samples subjected to FTIR-ATR analysis, a peak appears in the wavenumber range of 1560 – 1590 cm^{-1}, indicating the presence of carboxyl groups originating from 1,2,3,4-butanetetracarboxylic acid. This acid mediates the cross-linking of the β-cyclodextrin/tea tree essential oil inclusion complex with cellulose during in situ synthesis. In addition, distinct peaks at 555 cm^{-1}, 554 cm^{-1}, 558 cm^{-1}, and 557 cm^{-1} are visible in all treated and washed samples, which can be attributed to bending and out-of-plane vibrations between aromatic rings within C–C bonds as well as C–H bending in the monoterpene structure of γ-terpinene [7, 15]. It is important to note that even after the washing cycle (samples P_PES_P_BCD_TT_W, Figure 3, and P_PES_A_BCD_TT_W, Figure 4), the spectral profiles of the treated samples remain unchanged. In particular, the peak at 1509 cm^{-1} and 1575 cm^{-1} is clearly distinguishable in the washed samples (P_PES_P_BCD_TT_W, Figure 3, P_PES_A_BCD_TT_W, Figure 4), which confirms that no structural changes occurred after washing, indicating stable crosslinking.

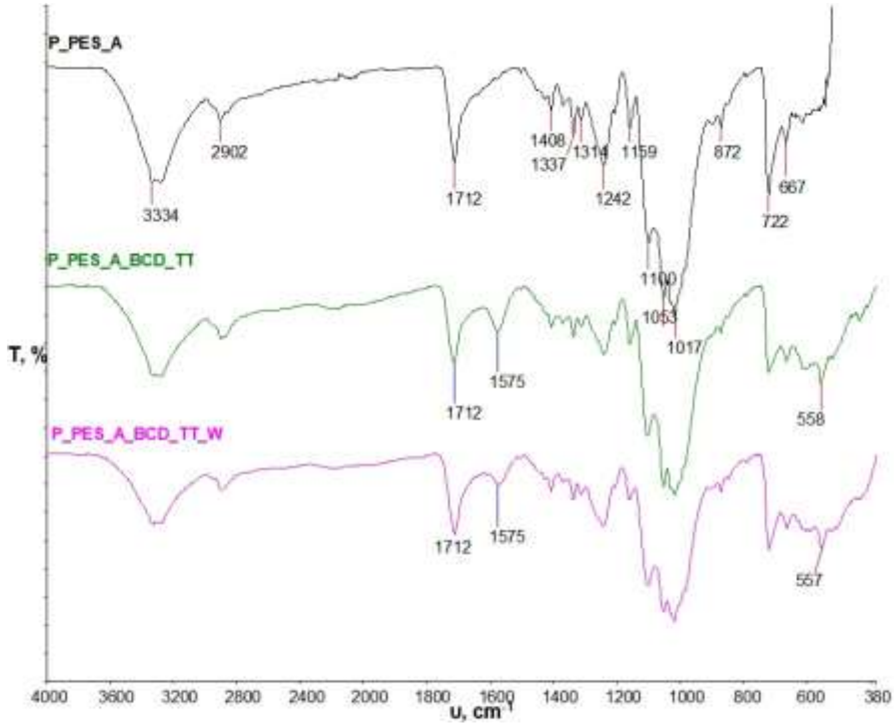

Figure 4. Spectral curves of the untreated (P_PES_A), treated (P_PES_A_BCD_TT), and treated washed sample (P_PES_A_BCD_TT_W) of the cotton/polyester fabric in satin weave.

Table 2 presents the moisture management properties of the samples P_PES_P, P_PES_A, P_PES_P_BCD_TT, and P_PES_A_BCD_TT, measured using a Moisture Management Tester (MMT). The untreated plain weave sample (P_PES_P) exhibits a longer wetting time on the top surface compared to the same sample in satin weave (P_PES_A). This can be attributed to the lower number of interlacing points and the smoother surface of the satin weave, which facilitates faster liquid spreading, i.e., surface wicking. Consequently, the wetting time on the bottom surface of the untreated satin weave sample (P_PES_A) is shorter (2.3713 s) compared to the plain weave sample (P_PES_P, 5.616 s). The top-surface absorption rate and moisture spreading speed are also higher in the satin weave sample (P_PES_A), resulting in a larger top-surface wetting radius compared to the plain weave sample (P_PES_P). Conversely, the bottom-surface absorption rate is slightly higher in the plain weave sample (P_PES_P). This is likely due to the weave structure: plain weave has more interlacing points, allowing for easier capillary liquid transport, whereas satin weave, with fewer interlacing points, has larger capillary spaces that trap more air. Furthermore, the satin weave fabric sample exhibits a slightly higher Accumulative One-Way Transport Index (%), while its Overall Moisture Management Capacity (OMMC) is slightly lower compared to the plain weave sample. Interestingly, the results presented in Table 2 show that both treated samples (P_PES_P_BCD_TT and P_PES_A_BCD_TT) demonstrate identical values across all measured parameters. This suggests a significant effect of the inclusion complexes of β-cyclodextrin and tea tree oil on fabric hydrophilicity and moisture management. Such findings are particularly important considering the potential application of these newly developed samples in hospital environments [7, 17, 18].

Table 2. Moisture management properties of untreated samples in plain weave and satin weave (P_PES_P, P_PES_A) and of the same samples after finishing (P_PES_P_BCD_TT, P_PES_A_BCD_TT)

	P_PES_P	**P_PES_A**	**P_PES_P_BCD-TT**	**P_PES_A_BCD-TT**
Wetting Time – Top Surface (s)	5.9593	2.278	2.9328	2.9328
Absorption Rate – Top Surface (%/s)	70.6766	83.6562	56.0425	56.0425
Wetted Radius – Top Surface (mm)	20	30	22.5	22.5
Spreading Speed – Top Surface (mm/s)	3.8088	7.9669	5.4599	5.4599
Wetting Time – Bottom Surface (s)	5.616	2.3713	3.0422	3.0422
Absorption Rate – Bottom Surface (%/s)	75.2511	74.2551	58.7217	58.7217
Wetted Radius – Bottom Surface (mm)	20	30	22.5	22.5
Spreading Speed – Bottom Surface (mm/s)	4.7181	7.5601	5.416	5.416
Accumulative One-Way Transport Index (%)	95.9004	96.34	111.7374	111.7374
Overall Moisture Management Capacity (OMMC)	0.5828	0.4332	0.565	0.565

Table 3. Antimicrobial efficacy of untreated samples (P_PES_P, P_PES_A), the same samples after finishing (P_PES_P_BCD_TT, P_PES_A_BCD_TT), and after the washing cycle (P_PES_P_BCD_TT_W, P_PES_A_BCD_TT_W).

Label	***S. aureus***	***Escherichia coli***	***Candida albicans***
P_PES_P	-	-	-
P_PES_P_BCD_TT	+/-	+/-	+ (2 mm)
P_PES_P_BCD_TT_W	+/-	+/-	+/-
P_PES_A	-	-	-
P_PES_A_BCD_TT	+/-	+/-	+ (1 mm)
P_PES_A_BCD_TT_W	+/-	+/-	+/-

- No antimicrobial efficacy
+/- Antimicrobial efficacy visible on the fabric; no presence of bacteria or fungi beneath it, but no inhibition zone developed
+ High antimicrobial protection, visible inhibition zone

The results shown in Table 3 confirm the antimicrobial activity of all treated and washed samples against both Gram-positive and Gram-negative bacteria, as well as excellent antifungal activity against Candida albicans. The treated sample P_PES_P_BCD_TT exhibited an inhibition zone of 2 mm, while a slightly smaller zone of 1 mm was observed in the sample P_PES_A_BCD_TT. After the washing cycle, the samples retained their antimicrobial activity against all tested microbial strains.

CONCLUSION

The effectiveness of the binding of the inclusion complex of β-cyclodextrin and tea tree essential oil to polyester cotton fabric in plain and satin weave and the wash durability of the treatment were confirmed by the results of Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR). Changes in the spectral bands of the treated and washed treated samples were observed, as evidenced by the appearance of new peaks and variations in the intensity of existing peaks compared to the untreated sample.

The results of the moisture management tests show that the treated samples in plain and satin weave (P_PES_P_BCD-TT, P_PES_A_BCD-TT) belong to the category of materials with excellent moisture management properties. They are characterized by rapid wetting, rapid absorption, a large liquid spreading area on both the front and back of the fabric, rapid wicking on the back of the fabric, and an excellent ability to transport liquid in one direction. From these results, it can be concluded that the treated samples have hydrophilic and hygroscopic properties.

The antimicrobial analysis, carried out according to the AATCC 147 standard, showed good antimicrobial efficacy of all treated and washed samples against the Gram-positive bacterium *S. aureus*, the Gram-negative bacterium *E. coli,* and the fungus *C. albicans*, without the development of an inhibition zone. However, samples P_PES_P_BCD-TT and P_PES_A_BCD-TT showed an inhibitory effect against C. albicans, indicating a high antimicrobial protection of the fabrics treated with the β-cyclodextrin-tea tree oil inclusion complex.

All these properties of the treated and laundered polyester–cotton fabrics in plain and satin weave indicate their potential applicability in hospital environments.

Acknowledgement

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Izvod

PRIMENA IN-SITU HIDROTERMALNE SINTEZE ZA FUNKCIONALIZACIJU TKANINE PAMUK/POLIESTAR INKLUZIVNIM KOMPLEKSOM BETA-CIKLODEKSTRINA I ETARSKOG ULjA ČAJEVCA

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Tekstilni materijali se često koriste kao nosači aktivnih supstanci kroz različite postupke radi postizanja antimikrobnih i wellness svojstava za medicinsku i kozmetičku primenu. Jedinstvena struktura β-ciklodekstrina, koja omogućava formiranje inkluzivnih kompleksa, dovela je do značajne komercijalne primene u farmaceutskoj, kozmetičkoj i tekstilnoj industriji. Ključna prednost β-ciklodekstrina je njihova ekološka prihvatljivost — biorazgradivi su, netoksični i ne zagađuju sisteme otpadnih voda. U ovom radu ispitivano je formiranje inkluzivnih kompleksa između β-ciklodekstrina i etarskog ulja čajevca, kao i njihovo vezivanje za tkanine pamuk/poliestar metodom in-situ hidrotermalne sinteze. Deo obrađenog materijala podvrgnut je postupku održavanja u skladu sa standardom ISO 6330:2012, koristeći program 6N i standardni ECE deterdžent (WFK 88030), radi procene trajnosti obrade. Promene na fizičko-hemijskom nivou pre i posle pranja analizirane su primenom FTIR-ATR spektroskopije, dok su apsorpciona i antimikrobna svojstva ispitivana na neobrađenom, obrađenom i opranom obrađenom materijalu, uzimajući u obzir njihovu moguću primenu u bolničkom okruženju. Rezultati pokazuju efikasno vezivanje inkluzivnih kompleksa β-ciklodekstrin–etarsko ulje čajevca za tkaninu pamuk/poliestar primenom in-situ hidrotermalne sinteze, pri čemu obrada zadržava visoku trajnost i posle pranja. Dobijeni rezultati naglašavaju potencijal za dalja istraživanja u razvoju funkcionalnih, visoko performansnih naprednih tekstila za medicinsku upotrebu.

Ključne reči: tkanina pamuk/poliestar, inkluzivni kompleksi β-ciklodekstrin–etarsko ulje čajevca, in-situ hidrotermalna sinteza