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**Dodecyltriphenylphosphonium Bromide Antimicrobial Finishing and
Pigment Coloring of Polyester Fabrics in One-Bath**

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Abstract

This study employed the one-bath pad-dry-cure process using a binder to fix antimicrobial dodecyltriphenylphosphonium bromide (12-TPPB) and pigment onto polyester fabric, thereby avoiding the high energy consumption and low efficiency of traditional multi-step finishing processes. This approach enabled the simultaneous coloration and antimicrobial functionalization of polyester fabric, making it suitable for outdoor textiles requiring both color and antimicrobial properties. The minimum inhibitory concentrations (MIC) of synthesized 12-TPPB against *E. coli* and *S. aureus* were 8 and 2 mg/L, while the minimum bactericidal concentrations (MBC) were 64 and 16 mg/L, indicating broad-spectrum antimicrobial activity. The modified polyester fabrics exhibited excellent antimicrobial properties, retaining $\geq 99.9\%$ activity against *S. aureus* and $\geq 90\%$ against *E. coli* after 50 washing cycles. Importantly, the addition of 12-TPPB did not affect pigment coloration of polyester fabrics. This study offers a new approach for the simultaneous pigment coloration and functionalization of polyester fabrics, which has practical value for application in the outdoor textile industry.

Keywords Quaternary phosphonium salt; Polyester fabric; Antibacterial; Pigment; Laundering durability

1. Introduction

Textiles are widely used in everyday life, and among them, polyester fabrics have become the most widely used substrate for outdoor textiles due to their cost-effectiveness, high tensile strength, and chemical resistance^{1,2}. However, their lipophilic and hydrophobic nature causes their surface to readily absorb organic residues such as sweat and sebum, making them prone to bacterial proliferation and increasing the risk of odors and skin infections³. Although polyester fabrics offer outstanding mechanical and chemical stability, they lack inherent antimicrobial properties⁴. In outdoor environments, bright, high-visibility colors significantly improve wearer visibility and safety, serving as protective factors as important as antimicrobial properties⁵. Consequently, multifunctional polyester-based fabrics combining excellent mechanical properties, long-lasting antimicrobial activity, and vibrant colors have become the preferred choice for enhancing health and safety in outdoor settings. Currently, coating with binders is an ideal method for fabric modification, as it effectively prevents issues such as reduced fiber strength and surface irregularities caused by chemical grafting^{6,7}. Due to its simplicity and low cost, it has been widely adopted⁸. Traditional sequential finishing suffers from interference between steps. When dyeing precedes antimicrobial treatment, dyes occupy active sites, resulting in poor adhesion and wash fastness of the antimicrobial agent. Furthermore, high temperatures induce thermal migration, altering color shade⁹. When antimicrobial treatment is performed before dyeing, the antimicrobial agent interferes with dye adsorption, resulting in reduced color depth. This step-by-step process consumes

significant amounts of energy and water¹⁰. Thus, traditional multi-step processes are inherently prone to process conflicts and resource waste, making it imperative to develop a one-step process.

Natural antimicrobial agents have attracted significant attention due to their excellent biocompatibility, but they suffer from poor durability¹¹⁻¹⁴. Although inorganic antimicrobial agents, such as silver nanoparticles, are highly effective, they pose risks of cytotoxicity and environmental bioaccumulation¹⁵⁻¹⁷. Due to their diverse molecular structures and high design flexibility, organic antimicrobial agents have become the most widely used antimicrobial materials today, primarily including biguanides, quaternary ammonium salts, and quaternary phosphonium salts¹⁸. Quaternary phosphonium salts (QPS) are highly effective, broad-spectrum organic antimicrobial agents^{19,20}, exhibiting good chemical inertness and biosafety²¹. The mechanism of action of QPS is similar to that of quaternary ammonium salts²². However, because phosphorus atoms have a larger radius and lower electronegativity than nitrogen atoms, QPS are more capable of adsorbing onto negatively charged bacterial membranes, resulting in superior antimicrobial performance²³.

In this study, alkyl-substituted triphenylphosphonium bromide antimicrobial agents with alkyl chain lengths of 8, 12, and 16 were synthesized. Antimicrobial testing revealed that 12-TPPB exhibited the best antimicrobial performance. This study marks the first application of 12-TPPB in antimicrobial finishing and pigment coloring of polyester fabrics. The one-bath process avoids the interference between steps inherent to traditional multi-step methods (Fig. 1). We systematically evaluated the

antimicrobial efficacy and durability of 12-TPPB-modified fabrics, as well as their tensile properties, softness, and hand. The resulting fabrics not only exhibit excellent antimicrobial performance and durability but also maintain good color stability and coloration properties, while retaining desirable basic performance characteristics.

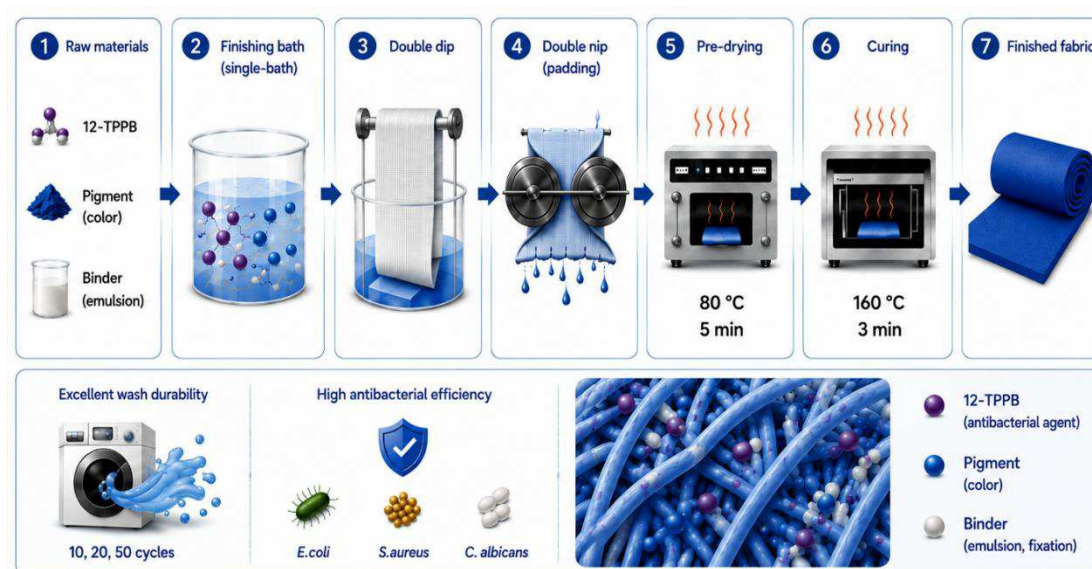


Fig. 1. Schematic diagram of the treatment process for antimicrobial colored polyester fabric of 12-TPPB

2. Experimental section

2.1 Materials

Polyester was purchased from Suzhou Fengdahang Textile Co., Ltd. triphenylphosphine (TPP, 98%) was purchased from Jinan Tudafei Trading Co., Ltd. bromo-n-octane (99%), bromo-dodecane (98%), bromohexadecane (97%) were purchased from Shanghai McLean Biochemical Technology Co., Ltd., dichloromethane (99.9%) was purchased from Jinan Sbaite Biotechnology Co., Ltd., and anhydrous diethyl ether (analytical grade, AR) was purchased from Qingdao Zhengye Reagent & Instrument Co., Ltd. VAE-707 emulsion (vinyl acetate-ethylene copolymer emulsion)

was purchased from Shandong Xuri Chemical Co., Ltd. A commercially available nonionic pigment (Archroma (China) Co., Ltd.) was used as the colorant. *Staphylococcus aureus* (CMCC(B)44102), *Escherichia coli* (CMCC(B)26003), and *Candida albicans* (ATCC10231) were purchased from the Beijing Microbial Culture Center.

2.2 Synthesis of 8-TPPB, 12-TPPB, and 16-TPPB

Triphenylphosphine (1.2 eq) and bromoalkanes with different alkyl chain lengths (1.0 eq) were stirred and refluxed for 24 h at 130 °C under a nitrogen atmosphere²⁴. Next, the crude product was dissolved in the minimum volume of dichloromethane and precipitated with ice-cold ether. The precipitate was filtered and washed with ice-cold ether. Finally, the resulting products were dissolved in dichloromethane, and the solvent was removed under vacuum to yield alkyltriphenylphosphonium bromide antimicrobial agents with different alkyl chain lengths, designated as 8-TPPB, 12-TPPB, and 16-TPPB, respectively. The synthesis procedure is shown in Fig. 2.

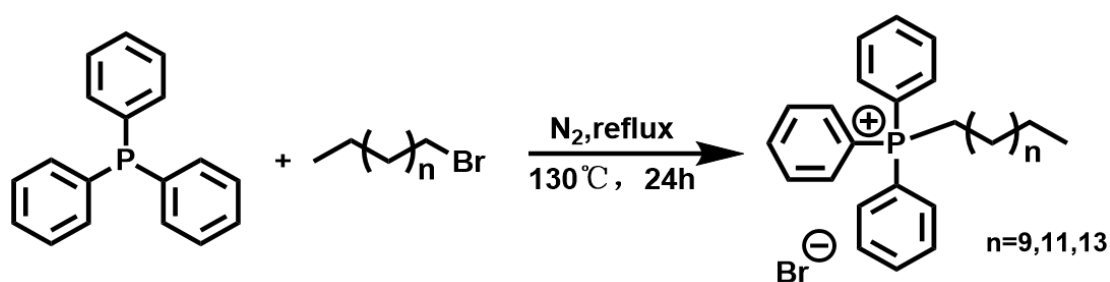


Fig. 2. Synthesis of antimicrobial agents 8-TPPB, 12-TPPB, and 16-TPPB

2.3 Finishing Processes for Polyester Fabrics

The polyester fabric was immersed in a 1% NaOH solution and treated at 100 °C for 1 hour to remove impurities. It was then thoroughly rinsed with deionized water until the pH reached neutral and dried at 60 °C. The resulting fabric was designated as PET.

Three finishing solutions were prepared, containing 1 wt%, 2 wt%, and 3 wt% of 12-TPPB, each with 5% nonionic binder and 1% pigment. For each solution, the components were weighed, placed in a beaker, and mixed with deionized water under magnetic stirring (500 rpm, room temperature) to obtain a stable working solution. The PET was completely submerged in the respective finishing solutions. After ensuring that the PET was thoroughly wetted, treated using a two-dip, two-roll process (with a liquid pickup rate of $80 \pm 5\%$) to ensure even distribution of the working solution on the fabrics. After processing, the PET was first pre-dried at 80 °C for 5 min and then baked at 160 °C for 3 min. This treatment caused the binder to cross-link and cure, thereby immobilizing the antimicrobial agent and pigment on the fiber surface. Finally, the PET was thoroughly washed to remove excess pigment and unfixed agents, the resulting fabrics, dried at 80 °C, were designated as 1PETDQ, 2PETDQ, and 3PETDQ. Two additional finishing solutions were prepared separately: one containing only 12-TPPB (2 wt%) and 5% nonionic binder, and the other containing only pigment (1%) and 5% nonionic binder. Both solutions were treated using the same process to produce two control samples, designated as PETQ and PETD, respectively.

2.4 Characterization

Fourier-transform infrared (FTIR) spectroscopy (Thermo Fisher Scientific, USA), ¹H nuclear magnetic resonance (¹H NMR, 400 MHz, Bruker, Germany), and X-ray photoelectron spectroscopy (XPS, K-Alpha system, Thermo Fisher Scientific, USA) were used to characterize the chemical groups, molecular structures, and surface chemical compositions of the samples. Thermal stability was evaluated using

thermogravimetric analysis (TGA) on a HITACHI STA200 system (Hitachi, Japan). Detailed TGA data are provided in the supplementary material (Fig. S1). A scanning electron microscope equipped with an energy dispersive spectrometer (SEM-EDS, JSM-7800F, Nippon Electronics, Japan) was used to examine the microstructure and elemental distribution of the samples. The MIC and MBC of quaternary phosphonium salts against *S. aureus* and *E. coli* were determined using the broth dilution method²⁵. In accordance with GB/T 31713-2015, the antibacterial activity of the samples was evaluated using the zone of inhibition (ZOI) method. In accordance with GB/T 20944.3-2008, the antimicrobial activity of the modified PET was tested using the shaking method. The wash fastness of the fabric was measured using an SW-20B washing machine in accordance with AATCC Test Method 61-2013 (Test 1A). The color difference (ΔE) was calculated using Equation (1), where ΔL^* is the difference in lightness, and Δa^* and Δb^* are the differences in the red-green and yellow-blue coordinates, respectively. According to the literature^{26,27}, $\Delta E > 1$ typically indicates a visually detectable color difference, and $\Delta E \leq 5$ suggests no significant color change after immersion in acidic, alkaline, or aqueous solutions²⁸. The tensile strength (Instron 5967 universal testing machine, Instron, USA), flexibility (ring method), wrinkle recovery angle (M5010 wrinkle recovery tester), contact angle (AFES contact angle measuring instrument, Shanghai AFES, China), breathability (FX3300IV air permeability tester, TEXTTEST, Switzerland), and fabric hand (FES-3-10 instrument, NuCybertek, Mexico) were measured.

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

3. Results and discussions

3.1 Structural Characterization of 8-TPPB, 12-TPPB, and 16-TPPB

The FTIR spectra of 8-TPPB, 12-TPPB, and 16-TPPB were shown in Fig. 3. The peaks at 2917 cm^{-1} and 2849 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of the methylene ($-\text{CH}_2-$) groups, respectively. The characteristic peaks of the salt were located at 1590 cm^{-1} , 1436 cm^{-1} , and 1112 cm^{-1} , which corresponded to the skeletal vibration of the benzene ring, the asymmetric bending vibration of the saturated C-H bond, and the stretching vibration of the P-C bond, respectively. In addition, out-of-plane bending vibration of the $-(\text{CH}_2)_n-$ group ($n \geq 4$) was observed at 721 cm^{-1} . Figure 2 (b-d) showed the ^1H NMR spectra of 8-TPPB, 12-TPPB, and 16-TPPB, which further confirmed the chemical structure of the alkyl triphenylphosphonium bromides. The positions marked in the structural formula correspond to the numbers shown in the spectra. These synthetic 8-TPPB, 12-TPPB, and 16-TPPB exhibited similar structures, differing only in the length of their alkyl chains, which resulted in variations in the ^1H NMR spectra and the observed hydrogen counted at the 2-proton (1.1 ppm) peak. It was worth noting that an increase in alkyl chain length led to a corresponding increase in the number of hydrogen atoms at these positions^{29,30}. The successful synthesis of 8-TPPB, 12-TPPB, and 16-TPPB was confirmed by FTIR and ^1H NMR.

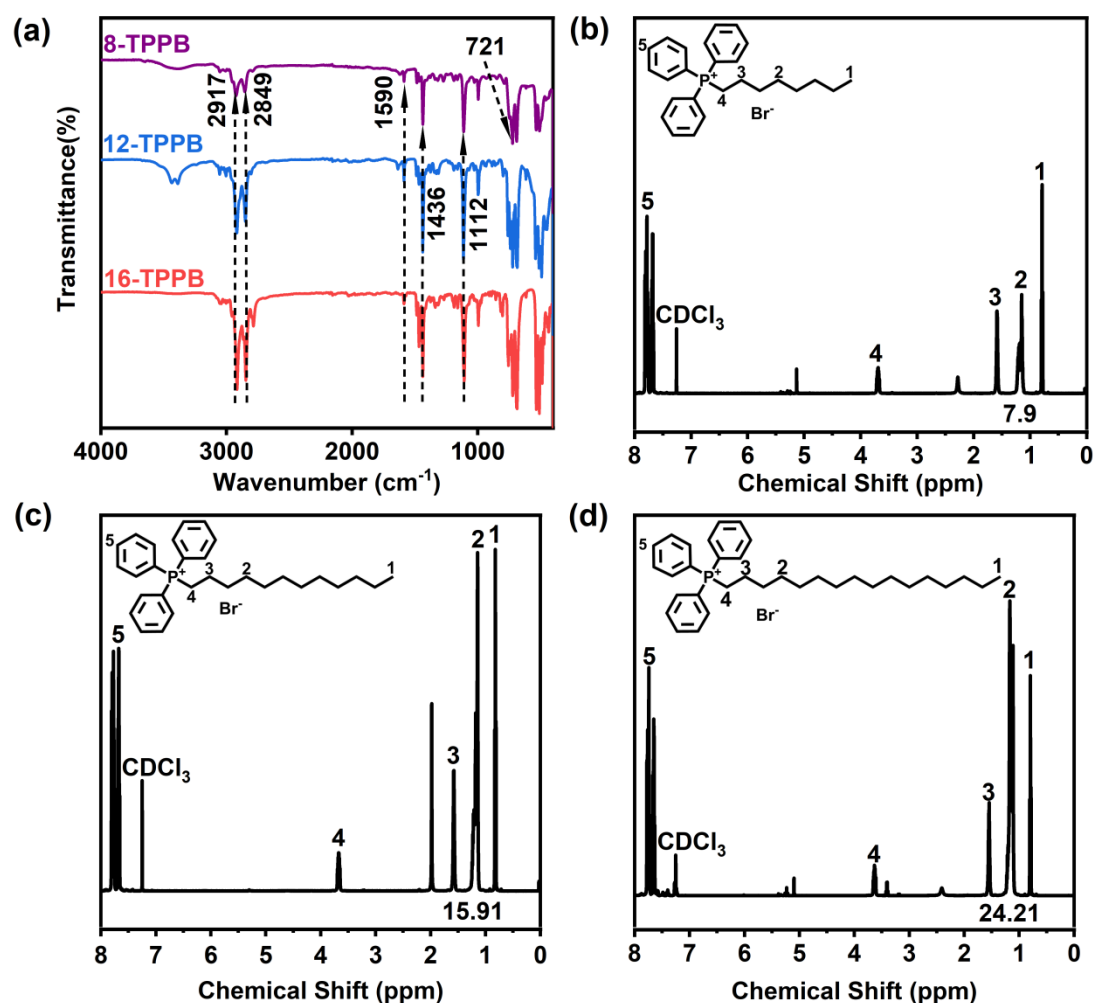


Fig. 3. FTIR spectra (a) and ^1H NMR spectra (b-d) of 8-TPPB, 12-TPPB, and 16-TPPB

3.2 Analysis of the Antimicrobial Properties of 8-TPPB, 12-TPPB, and 16-TPPB

MIC and MBC are key indicators for evaluating antimicrobial efficacy. The MIC and MBC values for 8-TPPB, 12-TPPB, and 16-TPPB are shown in Figs. 4 and 5. The MIC value of 8-TPPB against *S. aureus* was 4 mg/L, and the MBC value was 256 mg/L. The MIC values of 12-TPPB and 16-TPPB against *S. aureus* were both 2 mg/L, with MBC values of 16 mg/L and 32 mg/L, respectively. The MIC of 8-TPPB against *E. coli* was 128 mg/L, and the MBC was 1024 mg/L, the MIC of 12-TPPB against *E. coli* was 8 mg/L, and the MBC was 64 mg/L, the MIC of 16-TPPB against *E. coli* was 32 mg/L, and the MBC was 128 mg/L. Thus, 12-TPPB exhibited the highest antibacterial activity.

199 In contrast, 8-TPPB with a shorter alkyl chain exhibited significantly weaker
200 antimicrobial activity, whereas the activity of 16-TPPB with a longer alkyl chain did
201 not increase further but declined. This finding suggested that 12-TPPB was the optimal
202 chain length for this class of quaternary phosphonium salts to exhibit the best
203 antimicrobial performance. Alkyl chains that are too short lack sufficient lipophilicity
204 to disrupt cell membranes effectively. Conversely, excessively long chains may render
205 the molecule too hydrophobic, reducing dispersion or permeability and thereby
206 decreasing activity. Therefore, 12-TPPB was selected for functional finishing of the
207 subsequent fabric.

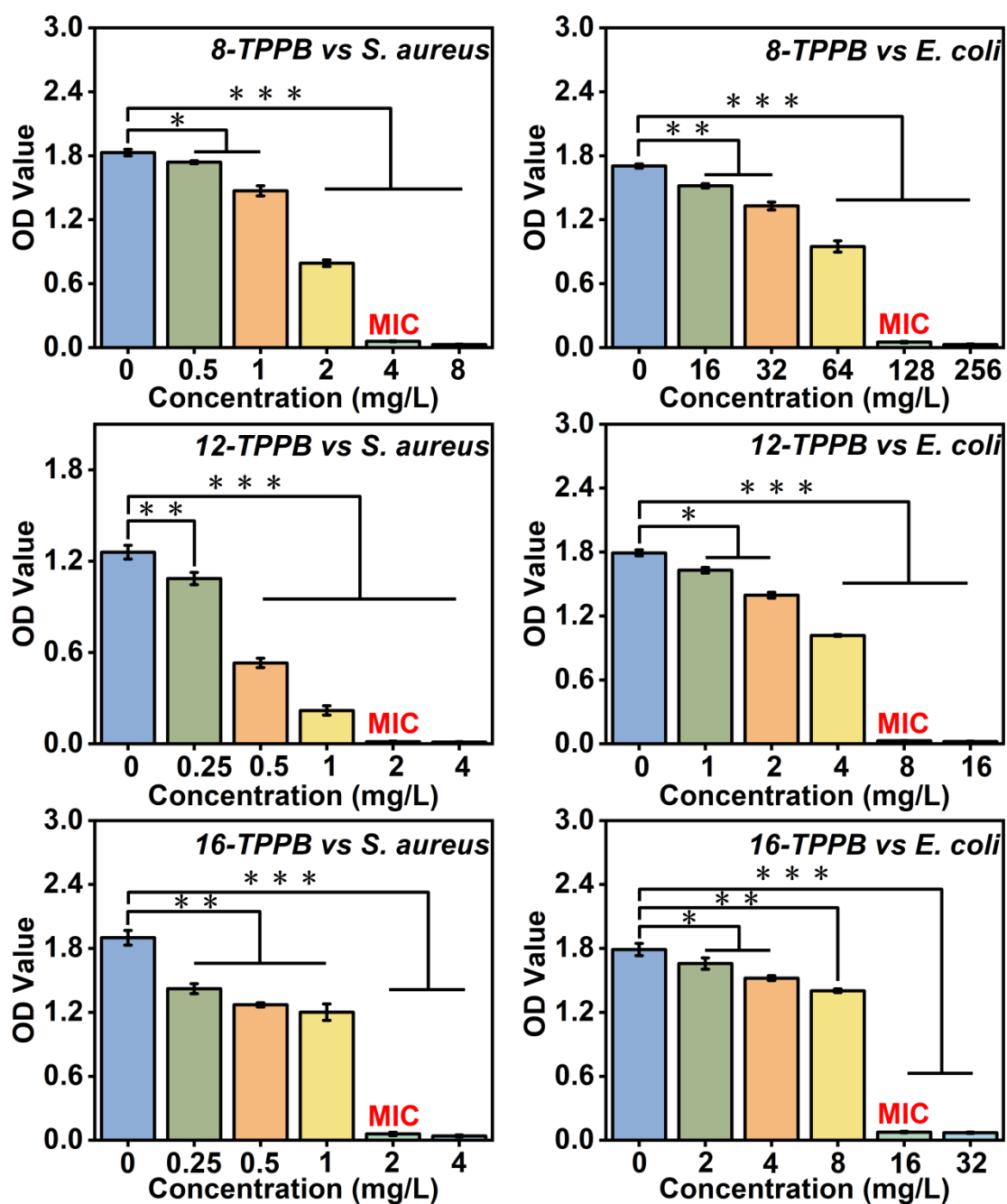


Fig. 4. MIC values of 8-TPPB, 12-TPPB, and 16-TPPB against *E. coli* and *S. aureus*

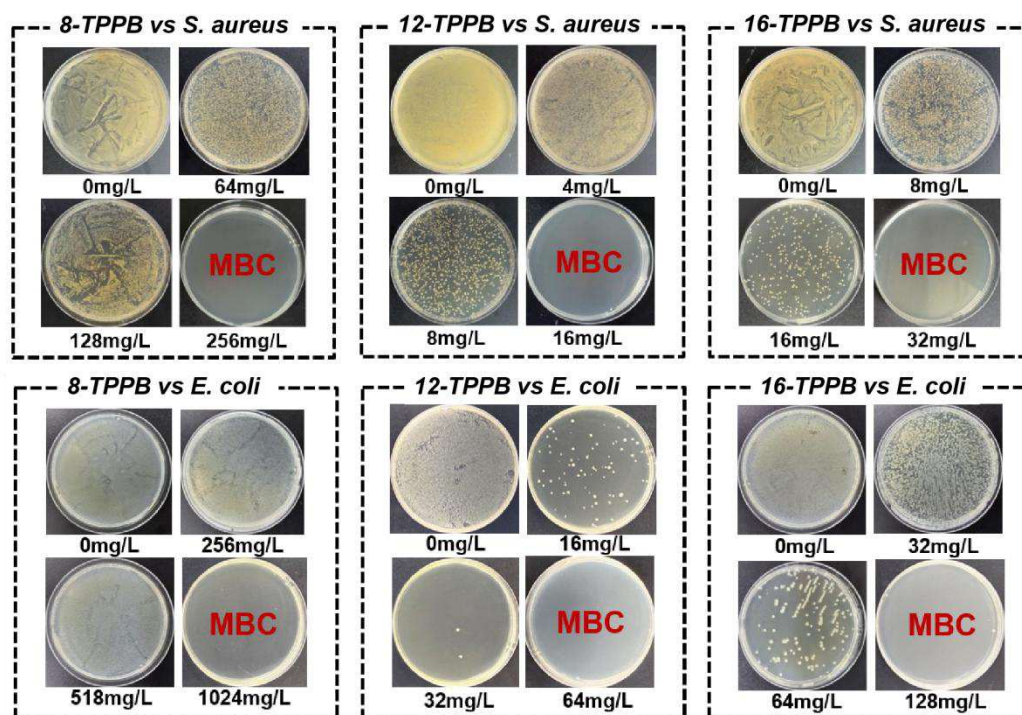


Fig. 5. MBC values of 8-TPPB, 12-TPPB, and 16-TPPB against *E. coli* and *S. aureus*

3.3 Structural Characterization of PETDQ

Figure 6(a) showed SEM images of the PETD and PETDQ samples. Figure 6(b) showed that phosphorus was uniformly distributed on the surface of PETDQ. Figure 7(a) showed the FTIR spectrum of PETDQ, with PETD used as a control. PETDQ exhibited two new characteristic peaks at 2920 cm^{-1} and 2851 cm^{-1} . These two peaks were attributed to the stretching vibrations of the symmetric and asymmetric $-\text{CH}_2$ bonds in the long chain. The peaks at 1450 cm^{-1} and 1113 cm^{-1} were characteristic peaks of the salt, corresponding to the skeletal vibration of the benzene ring and the stretching vibration of the P-C bond, respectively. Figure 7(b) showed the full spectrum of PETDQ, revealing characteristic peaks for the elements C, O and P. Further peak fitting of the high-resolution spectrum of P_{2p} revealed a doublet corresponding to C-P and P^+ . As was shown in Fig. 7(c), the two peaks at binding energies of 133.4 eV and 132.5 eV

corresponded to the $2p^{1/2}$ and $2p^{3/2}$ orbitals, indicating that 12-TPPB had been successfully loaded onto the fabric.

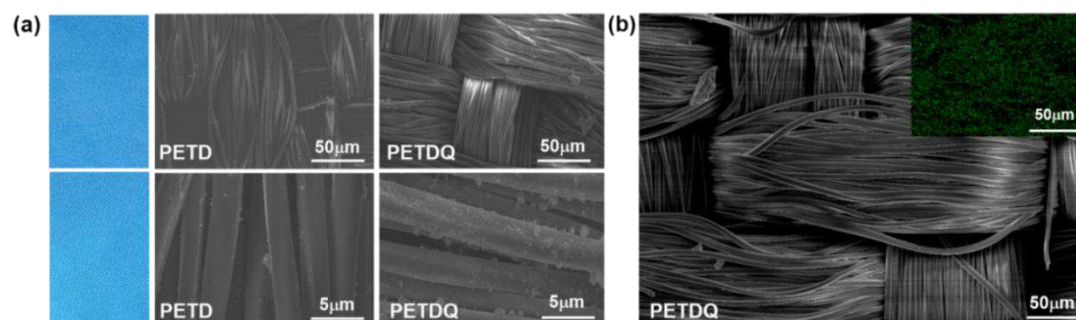


Fig. 6. SEM of PETD and PETDQ (a), and EDS of PETDQ (b)

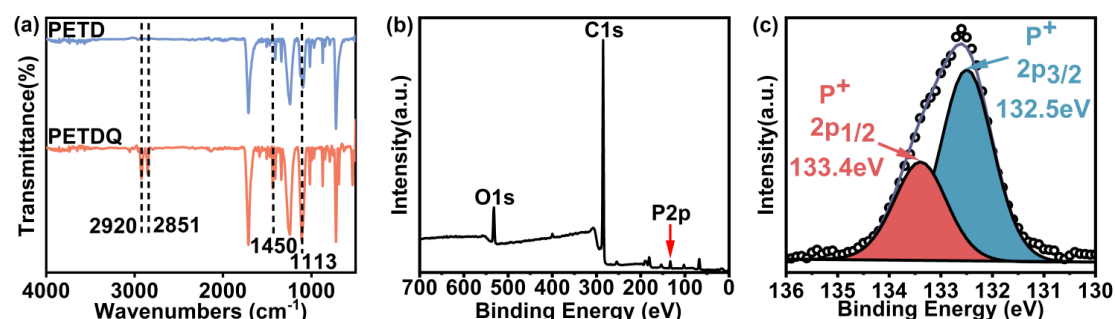


Fig. 7. FTIR spectra of PETD and PETDQ (a), XPS full survey spectrum (b), and high-resolution spectra of P_{2p} (c)

3.4 Analysis of the Antimicrobial Properties of PETDQ

As shown in Fig. 8(a), the agar plates for *E. coli*, *S. aureus*, and *C. albicans* all exhibited numerous contiguous colonies. The ZOIs for 12-TPPB against *S. aureus*, *E. coli*, and *C. albicans* were 4.0 mm, 1.5 mm, and 3.5 mm, respectively. According to Chinese national standard GB/T 31713-2015, ZOI widths of less than 1 mm were classified as non-leaching, those of 1-5 mm as low-leaching, 5-10 mm as moderate-leaching, and > 10 mm as high-leaching. Thus, 12-TPPB was classified as a low-leaching antimicrobial agent, indicating that the antimicrobial agent was stably loaded onto the fabric surface and provided durable antimicrobial performance. The ZOI of 12-TPPB against *S.*

aureus was significantly larger than that against *E. coli* and *C. albicans*, indicating that it was most effective against this bacterium. This difference is primarily attributed to variations in the isoelectric points of the tested microorganisms. The isoelectric point of *S. aureus* was approximately 2-3, that of *E. coli* was 4-5, and that of *C. albicans* was approximately 6³¹. Microorganisms exhibit a negative charge in near-neutral or slightly alkaline environments. *S. aureus*, which carries a significant negative charge, exhibits a stronger affinity for positively charged quaternary phosphonium salts, thereby enhancing the antibacterial effect against *S. aureus*. Figure 8(b) and 8(c) showed the antimicrobial efficiency of PETDQ prepared using antimicrobial finishing agent solutions at concentrations of 1%, 2%, and 3%, respectively. Figure 8(b) showed digital photographs of agar plates corresponding to different concentrations of antimicrobial agents. The untreated PET fabric samples exhibited numerous contiguous colonies. In contrast, antimicrobial fabrics treated with 12-TPPB showed a significant reduction in the number of microbial colonies on agar plates, demonstrating strong antimicrobial efficacy. At a concentration of 1%, the antibacterial activity against *E. coli* was approximately 97.5%, and virtually no colony growth was observed for *S. aureus* and *C. albicans*. At 2% and 3%, the antibacterial rate reached 99.9% in both cases. Therefore, we selected the finishing solution containing 2% antimicrobial agent for subsequent experiments on PETDQ fabric.

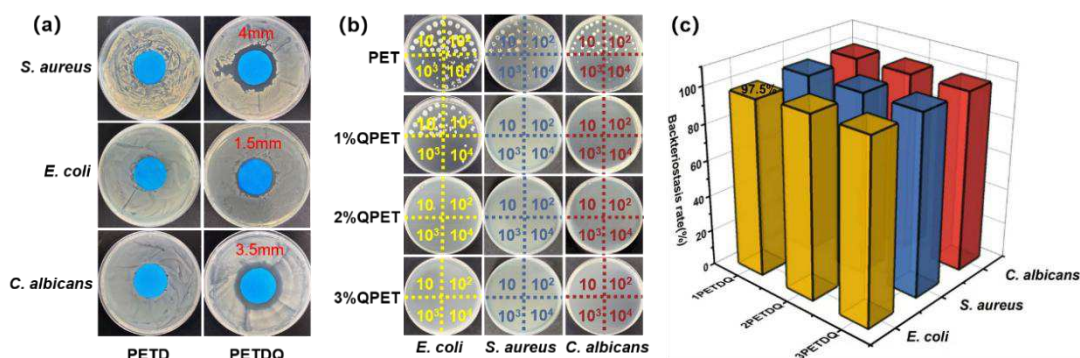


Fig. 8. ZOI photographs of polyester fabric (a) and digital photos of agar plates having microbial suspensions with polyester fabric (b). bacteriostatic rates of polyester fabric of different concentrations (c)

3.5 Wash Fastness of PETDQ

As shown in Fig. 9(a), after 10, 20, and 50 home laundering cycles, no significant bacterial colonies were observed on the agar plates following co-cultivation with microorganisms on the 12-TPPB-treated antimicrobial fabric. As shown in Fig. 9(b), after 50 wash cycles, PETDQ demonstrated high wash durability, with antibacterial rates against *S. aureus*, *E. coli*, and *C. albicans* greater than or equal to 99.9%, 90%, and 97%, respectively. This indicated that the modified polyester fabric met the AAA-grade antimicrobial durability requirements. As shown in Fig. 9(c), ΔE between PETD and PETDQ was less than 1.0, indicating that the addition of the antimicrobial agent did not affect the fabric's color. As shown in Fig. 9(d), after 50 washes, ΔE increased to 1.56, still far below the perceptible color change threshold of 5. This indicated that the fabric retained its color well after repeated washing.

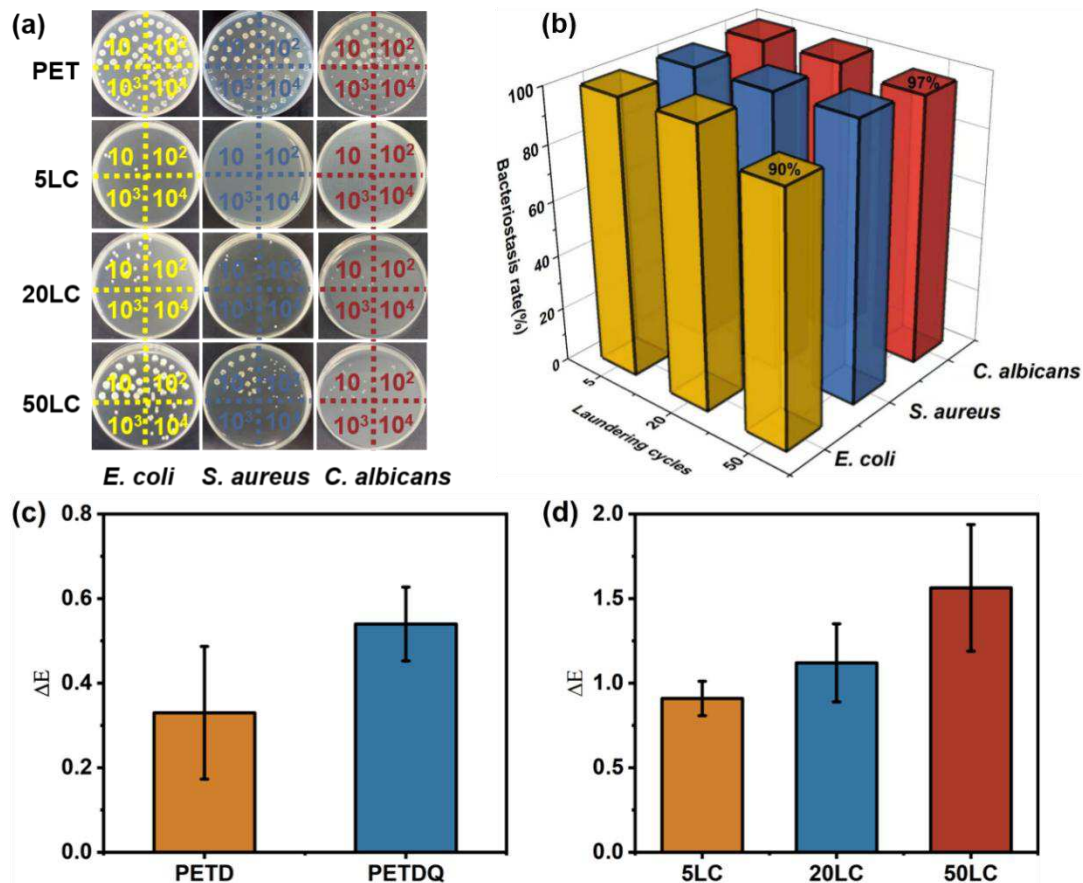


Fig. 9. Digital photos of agar plates having microbial suspensions with PETDQ after laundering cycles (a). bacteriostatic rates of PETDQ versus the number of laundering cycles (b) and color difference after laundering (c, d)

3.6 Analysis of wear performance

Figure 10(a) and (b) showed the mechanical properties of PET, PETD, and PETDQ. Compared to PET, the warp tensile strength of PETD increased from 563 N to 598 N, and that of PETDQ further increased to 615 N. The warp elongation at break increased from 27% to 28.8%. The tensile strength of the weft yarn increased from 362 N to 401 N, while that of the PETDQ reached 557 N. The elongation at break of the weft yarn increased from 5% to 9%. This indicated that the finishing process not only did not reduce the mechanical properties of the fibers but actually strengthened them due to the

dense surface coating and heat treatment. Figure 10(c) showed the contact angles of
 PET, PETD, and PETDQ. The contact angle of PET was 60°, while that of PETD
 increased to 75° and that of PETDQ increased to 80°, indicating a slight increase in
 hydrophobicity. As shown in Fig. 10(d), the air permeability of the modified fabrics
 remained essentially unchanged compared to PET. As shown in Fig. 10(e), the wrinkle
 recovery angles of PET, PETD, and PETDQ increased significantly, which was
 primarily attributed to the antimicrobial coating, which enhanced the elasticity of the
 fibers.

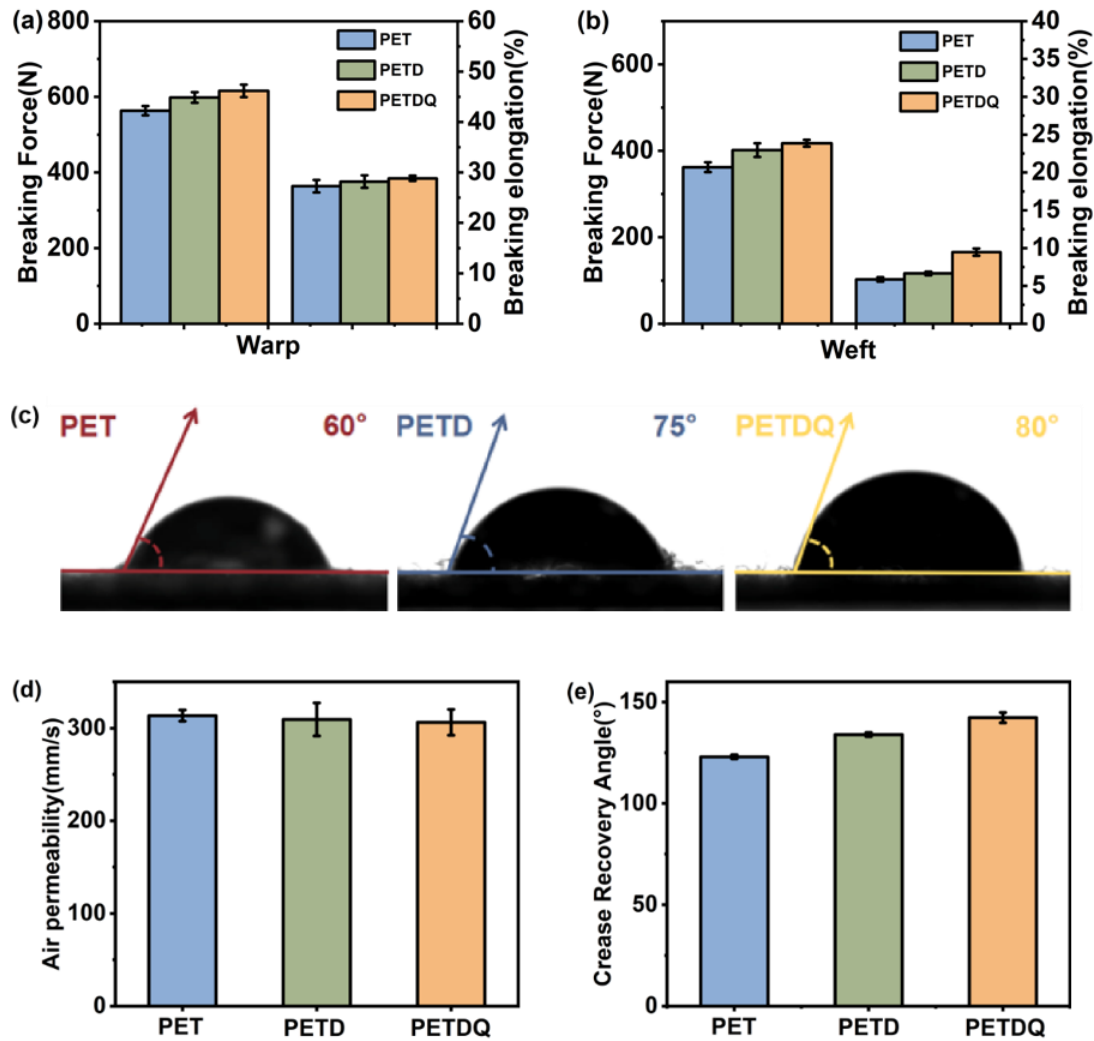


Fig. 10. Tensile test (a, b), hydrophilicity (c), air permeability (d) and crease recovery
 angle of PET, PETD and PETDQ (e)

Figure 11(a) showed the changes in flexibility for PET, PETD, and PETDQ. The lower the height, the better the flexibility. The circular height of PETD (2.8 cm) was comparable to that of PETDQ (2.9 cm), suggesting that the antimicrobial agent had little effect on fabric flexibility. Figure 11(b) showed the hand of PET, PETD, and PETDQ. Some key tactile parameters included polyester fabric softness, resilience, and smoothness. It could be observed that smoothness and drape improved progressively. PET, PETD, and PETDQ scored 43, 45, and 46, respectively. This indicated that PETDQ had a pleasant texture and felt similar to PET. There was a significant improvement in drape, with the score increasing from 14 to 25. A significant decrease in wrinkle recovery, particularly for PETDQ, which dropped to 65, was attributed to the additional internal friction introduced by the polymer coating formed on the surface. When the PETDQ was creased, friction occurred within the coating or at the interface between the coating and the fibers, which dissipated energy and prevented the fabric from quickly returning to its original smooth state. Elasticity was significantly improved, increasing from 28 to 45. These results indicate that PETDQ was smooth, well-draped, and highly elastic. It should be noted that the wrinkle recovery angle (Fig. 10e) and the wrinkle recovery score from the hand test (Fig. 11b) reflect different aspects of fabric deformation behavior and are therefore not contradictory.

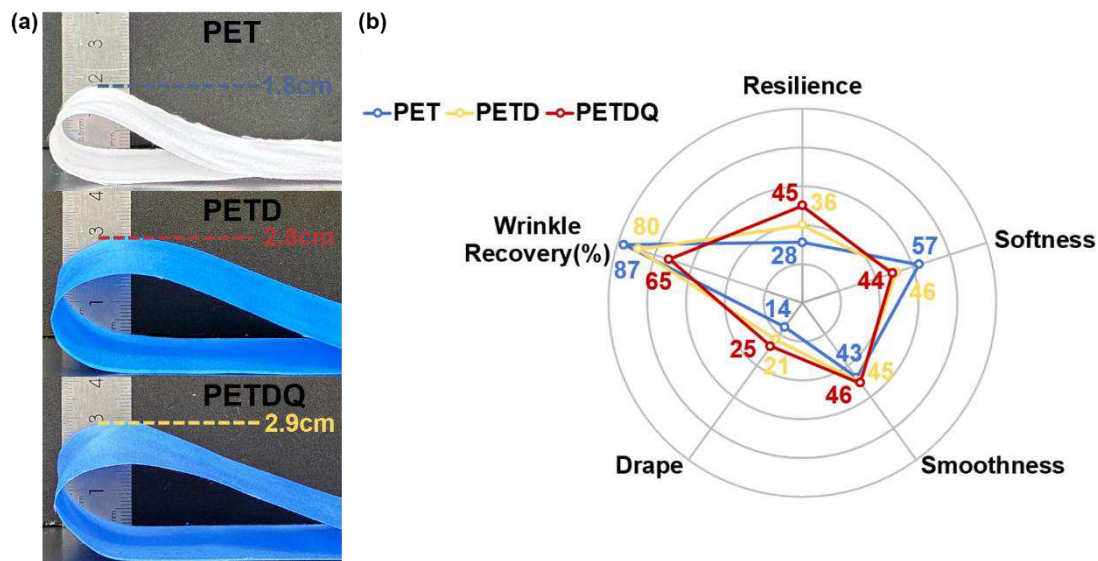


Fig. 11. Softness (a), hand assessment (b) of PET, PETD and PETDQ

4. Conclusion

In this study, the antimicrobial agent 12-TPPB was successfully synthesized, and PETDQ was prepared using a pad-dry-cure process. 12-TPPB exhibited MIC of 8 mg/L (against *E. coli*) and 2 mg/L (against *S. aureus*), with corresponding MBC of 64 mg/L and 16 mg/L, respectively, demonstrating strong bactericidal activity. PETDQ-modified fabrics demonstrated excellent antimicrobial efficacy and wash resistance. After 50 washes, the antimicrobial activity still met the AAA-grade standard as defined by Chinese textile standards. The color of the PETDQ showed no significant change, and there was no noticeable impact on its wear performance. Relative to the PETD control, PETDQ finishing had little effect on the fabric's basic properties, including mechanical strength, breathability, surface wettability, softness, and hand. This study reports the first application of 12-TPPB as an antimicrobial agent and pigment coloring on polyester fabrics, filling a gap in the field of 12-TPPB antimicrobial finishing for polyester fibers. The finishing method is simple, rapid, and cost-effective. 12-TPPB

could be used in blended spinning to impart lasting antimicrobial properties to the fibers themselves, laying the groundwork for the development of high-performance antimicrobial fabrics for medical, athletic, and decorative applications.

Author contributions Jiao-Jiao Ji: Investigation, Formal analysis, Writing-original draft. Mei-Yu Ci: Methodology, Investigation. Jin-Zhi Kuang: Methodology, Investigation. Lin Zhang: Conceptualization, Supervision, Funding acquisition, Writing-review & editing.

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Data availability Data will be made available on request.

Competing interests The authors declare no competing interests.

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