Photodynamic and Contact Killing Polymeric Fabric Coating for Bacteria and SARS-CoV-2

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ABSTRACT: The development of low-cost, non-toxic, scalable antimicrobial textiles is needed to address the spread of deadly pathogens. Here, we report a polysiloxane textile coating that possesses two modes of antimicrobial inactivation, passive contact inactivation through amine/imine functionalities and active photodynamic inactivation through the generation of reactive oxygen species (ROS). This material can be coated and cross-linked onto natural and synthetic textiles through a simple soak procedure, followed by UV cure to afford materials exhibiting no aqueous leaching and only minimal leaching in organic solvents. This coating minimally impacts the mechanical properties of the fabric while also imparting hydrophobicity. Passive inactivation of Escherichia coli (E. coli) and methicillin-resistant Staphylococcus aureus (MRSA) is achieved with >98% inactivation after 24 h, with a 23× and 3× inactivation rate increase against E. coli and MRSA, respectively, when green light is used to generate ROS. Up to 90% decrease in the infectivity of SARS-CoV-2 after 2 h of irradiated incubation with the material is demonstrated. These results show that modifying textiles with dual-functional polymers results in robust and highly antimicrobial materials that are expected to find widespread use in combating the spread of deadly pathogens.

KEYWORDS: antimicrobial, antiviral, textile, SARS-CoV-2, singlet oxygen, PDMS

INTRODUCTION

In healthcare settings, hospital-acquired infections (HAIs) result from pathogen transmission between patients and healthcare workers, primarily through contaminated surfaces.1,2 HAIs account for up to 100 000 deaths in the United States each year.3 Both Escherichia coli (E. coli) and methicillin-resistant Staphylococcus aureus (MRSA) infections are major sources of HAIs.4 In addition, the recently emerged severe acute respiratory syndrome coronavirus 2 (SARS-CoV-2), responsible for over 3.5 million deaths worldwide (as of June 1, 2021), continues to spread in indoor settings such as hospitals and long-term care facilities.5–8 As a precautionary measure to limit aerosol droplet transmission of SARS-CoV-2, widespread adoption of non-medical cloth facemasks has been recommended by world health authorities and has immensely reduced the spread in indoor settings.9,10 While SARS-CoV-2 is primarily spread through aerosolized droplets, the virus can survive up to 1 week on contaminated face masks and 2–3 days on steel surfaces.11–13 Exposed personal protective equipment (PPE) remains a possible vector of disease transmission for many pathogens and necessitates the proper use of PPE to prevent contamination.9 Functionalized textiles with self-disinfecting or self-cleaning properties may play a key role in limiting the spread of these diseases.14–16 Methods to prepare antimicrobial surfaces include cationic functionalization using amine or phosphonium groups, quaternary ammonium compounds (QACs), microscale surface patterning, or the addition of metal nanoparticles.17–21 These materials inactivate pathogens without the need for external stimulation either through contact lysis or gradual release of metal ions.17,22 Hydrophobic coatings can also be used to reduce microbial adhesion and prevent biofilm formation.23 Antimicrobial photodynamic inactivation (aPDI) is an alternative strategy for antimicrobial textiles where light stimulates a photosensitizer to generate reactive oxygen species (ROS) from atmospheric O2. The generated species can be either free radicals/radical ions (Type I) or singlet oxygen (1O2, Type II) and cause nonspecific and irreversible damage to microbial membranes and intercellular components.24,25 aPDI materials are effective against multiple types of pathogens including bacteria, viruses, and fungi and remain effective against antibiotic-resistant bacteria.26–29 These sensitizers have been previously incorporated onto textiles through electrostatic interactions, covalent tethering, or encapsulation in a cross-linked polymer matrix.27,30

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Here, we present a dual-functional approach to antimicrobial textiles utilizing a single polymer with both antimicrobial functionalities (primary amines) and a covalently attached photosensitizer that can generate $^{1}O_2$ on irradiation with green light. This provides both passive and active modes of antimicrobial action. In the context of healthcare environments, we envision the passive antimicrobial action to provide a level of baseline protection, while the active mode enhances the antimicrobial activity when required for rapid disinfection in high-risk settings. Huang et al. previously demonstrated a dual-functional approach using the sequential deposition and reaction of $\varepsilon$-polylysine and zinc phthalocyanine onto cellulose fabric, requiring harsh chemical reaction conditions and lacking cross-linking of the polymer.\(^\text{31}\) Allan et al. recently demonstrated dual-functional ZnO/crystal violet antimicrobial polyurethane where ZnO nanoparticles were found to be antimicrobial both in the dark and under irradiation.\(^\text{32}\) Here, we demonstrate the coating of synthetic and natural textiles at room temperature using only a soak procedure, followed by UV cross-linking to affix the polymer onto the fabric. The treated textiles demonstrate only minimal loss of mechanical properties for high polymer loadings and exhibit high degrees of hydrophobicity, which may prevent bacterial adhesion. We demonstrate the passive antimicrobial activity against E. coli and MRSA as well as a rapid enhancement of bacterial and SARS-CoV-2 viral inactivation using aPDI through the generation of $^{1}O_2$ using visible light.

### RESULTS AND DISCUSSION

#### Preparation and Physical Characterization

To modify textile samples, fabrics were treated by room-temperature soaking in a solution containing the solubilized polymer and the cross-linking photosensitizer, followed by 405 nm irradiation to fix the polymer onto the fabric through $^{1}O_2$ cross-linking. This procedure omits any harsh chemical reagents or high temperatures that might damage the textile integrity.

The antimicrobial polymer (PRB) used in these experiments was prepared by condensing (7 wt % aminopropylmethylsiloxane)-dimethylsiloxane copolymer (PNH$\_7$) with 0.048 molar equivalents of Rose Bengal lactone (RBL) (Figure S1). Both components used in this work have been assessed as non-toxic and safe for human use.\(^\text{33,34}\) We have previously demonstrated that this system can be photocross-linked from a liquid to an elastomeric solid via $^{1}O_2$ oxidation of the primary amine functionalities into imine cross-links.\(^\text{35}\) However, this process photobleaches the RBL, leaving it unavailable for further $^{1}O_2$ production. To overcome this limitation, we use tetraphenylporphyrin (TPP) as a sacrificial $^{1}O_2$ source (Figure S2). In a thin-film experiment, TPP was selectively photo bleached over RBL using 405 nm irradiation, followed by bleaching of...
RBL using 530 nm light (39 and 8 mW/cm² at 4 cm, respectively), demonstrating that we can orthogonally excite one dye molecule over the other using narrow light sources (Figure S3).

PRB and TPP were combined using toluene and diluted with tetrahydrofuran (THF) to afford solutions of 1, 5, and 13% polymer weight %, denoted as P1, P5, and P13, respectively. Cotton fabric was soaked in these solutions for 10 min, followed by 30 min of 405 nm irradiation per side open to air to afford samples C/P1, C/P5, and C/P13 (Figure 1a). A sample of C/P13 soaked in THF before irradiation gave a relative molar ratio of RBL:TPP of 3.3:1 based on the absorbance intensities, closely matching the initial solution composition. The mass of the polymer on the fabric increased linearly with the polymer solution wt % to a maximum of 0.24 g PRB per gram of fabric for P13 (Figure 1b). The change in the mass of the treated fabric after soaking in THF was used to determine the extent of polymer cross-linking and attachment to the fibers. At low mass loadings (P1), and for samples that were not exposed to 405 nm light, > 99% of the loaded polymer was soluble in THF, indicating an unsuitable degree of cross-linking (Figure 1b). The percentage of the soluble material decreased to 13 and 7%, respectively, for C/P5 and C/P13, demonstrating a greater cross-linking extent. C/P13 soaked in water or an aqueous solution of laundry detergent overnight showed no mass change after drying and no coloration of the supernatant. Scanning electron microscopy (SEM) images of C/P13 show a reduction in surface roughness while still retaining distinct fibers (Figure S4). All coated samples became hydrophobic after treatment with the maximum contact angle of C/P13 being 147 ± 1° (Figure S5). The same procedure for C/P13 was used to treat other natural and synthetic fibers including linen, polyester, denim, silk, and paper, highlighting that only covalent bonds between the polymer chains are formed and that the chemical composition of the textile is not altered or relevant for the coating procedure (Figure S6). Mass loadings varied from 0.12 (denim) to 0.23 (paper) grams of polymer per gram of material, attributed to differences in material morphology and porosity (Figure S7). All coated materials were hydrophobic after treatment, with water contact angles ranging from 131 ± 2 (silk) to 142 ± 3° (polyester) (Figure S7).

**Mechanical Characterization.** The impact of polymer coating on the mechanical properties of the fabrics was tested for cotton and polyester, the most elastic textile used in our work. Strips were cut to a uniform size and coated using P1, P5, or P13. Their elongation at break and break stress were determined using an Instron system in the tensile mode (Figure 1c). An 18% decrease in elongation at break (128 ± 3 to 105 ± 7%) and a 13% decrease in break stress (102 ± 2 to 87 ± 5 kPa) were observed for the highest polymer loading on cotton (P13), with only 1% decreases in both values for polyester coated with the same solution (Figure S8 and Table S1). Cross-linked PRB by itself has an elongation at break of 117 ± 8% and a break stress of 550 kPa. We attribute the retention of mechanical properties to the mild nature of the treatment process and inherent flexibility of the siloxane polymer. Thermogravimetric analysis of C/P13 compared with untreated cotton showed an increase in the onset temperature (T_o) of 14 °C from 304 °C (untreated) to 320 °C (treated) (Figure S9). The thermal degradation of polyester before and after treatment showed little difference, with a T_o of 397 °C for both samples. The T_o of photocross-linked PRB is 423 °C.

**Fourier Transform Infrared, Absorption, and Emission Measurements.** Fourier transform infrared (FT-IR) spectroscopy was used to characterize the treated samples. P13 shows new vibrational peaks at 1258 and 793 cm⁻¹ that correspond with the PNH₂⁻⁻⁻⁻⁻ siloxane backbone, confirming the presence of the polymer (Figure S10). All other tested materials coated with P13 show the same vibrational peaks, with no samples showing vibrations unattributable to PNH₂⁻⁻⁻⁻⁻ or the textile itself (Figure S11). White cotton becomes pink after treatment owing to the RBL present in the system (Figure 1d). The appearance of black-colored cotton treated with the sample procedure does not change to the eye (Figure S12).

The transmittance spectrum of C/P13 shows two peaks at 525 and 415 nm attributed to the absorbance of RBL and TPP, respectively (Figures 2a, S2). Excitation at these wavelengths results in emission spectra characteristic of RBL and TPP, revealing that TPP is not fully photobleached during the UV cross-linking procedure and that both chromophores remain emissive in the treated textiles (Figure 2a). 1O² exhibits diagnostic phosphorescence centered at 1270 nm. The near-IR emission spectra of a thin film of cross-linked PRB and treated C/P13. 1O² phosphorescence at 1270 nm is observed for both materials as a result of RBL excitation at 525 nm.
static cuvette containing C/P13 and the aqueous uric acid solution was irradiated using 530 nm light and the change in absorbance was measured as a function of irradiation time (Figure S13). A 55% decrease in the absorbance at 292 nm was observed after 130 min of irradiation (Figure S14). A control sample of C/P13 prepared without RBL showed no decrease in absorbance on irradiation, while a sample with twice the RBL molar equivalence had an 81% decrease in absorbance after 130 min. No absorbance above 375 nm was observed during these experiments, indicating that RBL was not released into solution and remained attached to the fabric. A sample of C/P13 irradiated continuously with green light for 24 h no longer generated sufficient quantities of $^{1}$O$_2$ to degrade uric acid under these conditions, highlighting a potential limitation of the use of organic photosensitizers for long-term applications (Figure S15). However, these results confirm that $^{1}$O$_2$ can be generated from our treated fabric in sufficient quantities to oxidize dissolved or suspended molecules. While RBL has high selectivity for producing $^{1}$O$_2$ over other ROS (Type II), diffusion distances of $^{1}$O$_2$ are on the order of tens of nanometers.39,40 The efficacy of substrate oxidation for the bound RBL may indicate the formation of subsequent reactive species such as peroxides or hydroxyl radicals resulting from quenching of $^{1}$O$_2$ that possess greater diffusion distances.

**Contact Antimicrobial Activity.** We have previously demonstrated that $^{1}$O$_2$ cross-linked PNH$_2$-7 induces cell lysis in mammalian cells as well as increased antimicrobial efficacy versus *E. coli* compared to MRSA.41 Initially, for the study of our treated textiles, we investigated the antimicrobial activity versus *E. coli* and MRSA bacterial strains in the dark, examining only the passive contact antimicrobial activity. Coated fabric samples of C/P13 were cut to different sizes and challenged with a fixed concentration suspension of bacteria under dynamic shaking conditions overnight at 37 °C, followed by dilution and plating onto agar to compare the number of colony forming units (CFUs) against a control with no added fabric (Figure 3a). Incubation using a large bacterial volume was chosen over droplet-based measurements to allow for comparison of different sample masses as well as consistency across passive and active antibacterial and antiviral testing. However, this experimental setup is expected to reduce microbial contact time and therefore reported efficacy relative to droplet-based techniques, which encourage microbe—surface interactions and more closely mimic real-world conditions. A 31 ± 8% decrease in *E. coli* CFUs was observed for a 4 cm$^2$ sample, increasing to a 99 ± 2% decrease for 18 cm$^2$. Owing to the heterogeneous nature of our samples and dynamic agitation during incubation, a larger surface area is expected to increase the frequency of polymer—bacteria contact and result in the greater decrease in CFUs. To confirm that this antibacterial activity is a result of the primary amine groups, (23 wt % aminopropylmethyldiethoxysiloxane)-dimethyldiethoxysiloxane copolymer (PNH$_2$-23) was prepared in an identical fashion to PNH$_2$-7 and coated onto cotton (Table S2). A 4 cm$^2$ sample of this material resulted in a 98 ± 3% reduction in CFUs, a 3 times greater activity than for PNH$_2$-7. No inhibition was observed for a 1 cm$^2$ sample of this material. Samples challenged with MRSA under the same conditions resulted in >99% ± 1 reduction in CFUs for 18 cm$^2$ samples for PNH$_2$-7. At lower fabric loadings, there was decreased effectiveness against MRSA compared with *E. coli* for both polymer types, with the higher amine content polymer resulting in greater inactivation compared with the lower amine content polymer (Table S3). These experiments demonstrate the passive antimicrobial activity of our treated textiles and suggest that the antimicrobial activity is directly related to the weight % of amine functionality in the polymer.

**aPDI Antibacterial Studies.** Antimicrobial experiments performed in the dark can interrogate selectively the passive contact killing effect of the amine groups, but aPDI from the photogenerated $^{1}$O$_2$ cannot be isolated from the passive effect. Time-dependent experiments were run in tandem for C/P13, irradiating one set of samples with 530 nm light (passive and active effects) while others remained in the dark (passive only). Incubation was carried out at room temperature, with other conditions the same as for the dark-only experiments. Aliquots were drawn and diluted, and the relative number of CFUs at a given time was compared between the light and dark samples (Figure 3b). No difference in CFUs was observed between the dark and light samples in the absence of C/P13. After 30 min, there were 85 ± 9% (dark) and 3.0 ± 0.6% (light) CFUs remaining relative to t = 0 for *E. coli* and 95 ± 3% (dark) and 35 ± 3% (light) for MRSA. This 28× and 3× increase in disinfection rate for *E. coli* and MRSA, respectively, demonstrates that C/P13 has strong aPDI activity with a greater rate than the passive amine contact killing. No CFUs were observed for either of the irradiated samples at t = 240 min. Differences in the rate of $^{1}$O$_2$ inactivation of *E. coli*
compared with MRSA are consistent with reported sensitivities arising from differences in cell wall composition and antioxidant content.42

**aPDI Antiviral Studies.** aPDI materials which are effective against bacteria have also been demonstrated to be effective at inactivating enveloped and non-enveloped viruses.29 Recently, Ghiladi et al. have demonstrated the use of metalloporphyrins and Rose Bengal encapsulated in photocross-linked polymers for the inactivation of human coronaviruses on textiles.43 To investigate the antiviral properties of our treated material, we tested C/P13 against SARS-CoV-2 in a manner analogous to the aPDI bacterial experiments. Dilutions of SARS-CoV-2 were incubated with treated fabric samples or without fabric in the dark or under constant 530 nm light for 2 h (Figure 4a).

To assess the viral infectivity, a susceptible liver cell line (Huh-7.5.1) was infected with SARS-CoV-2 stock diluted in complete media was incubated with fabric samples in the dark or light (530 nm irradiation) conditions for 2 h at room temperature. The treated virus dilutions were used to (i) infect Huh-7.5.1 cells, and the resulting infectivity was measured by staining for intracellular virus nucleocapsid levels after 48 h of infection and (ii) the treated virus was also added to Vero E6 cells for measuring PFU using the plaque assay. Experiments were performed in triplicate. (b) Relative intracellular viral nucleocapsid levels of SARS-CoV-2 dilutions following incubation with fabric and infection using different MOI of Huh-7.5.1 cells. (c) SARS-CoV-2 viral load following incubation of 1.5 × 10⁴ PFU/mL with fabric under light and dark conditions. Circles represent individual replicates, and green bars are 95% CI. One-way ANOVA with Tukey’s multiple comparisons correction presented; **p = 0.001 and ****p < 0.0001. Representative viral plaques of (d) dark sample and (e) 530 nm irradiated sample.

Previous studies have reported the effect of ¹⁸O₂ on enveloped virus particles, which likely impede the viral entry and fusion through modification of the viral membrane lipid and glycoprotein components.46–49 The predicted effect is
consistent with our observed reduction in SARS-CoV-2 infectivity of HuH-7.5.1 cells, reduction in viral plaque counts on Vero E6 cells, and reduced plaque size following incubation of the virus with aPDI.

**CONCLUSIONS**

We have successfully demonstrated the coating and subsequent photocross-linking of a siloxane copolymer onto textiles using a room-temperature procedure in approximately 1 h. Excitation of a sacrificial porphyrin molecule results in $^{1}O_2$ oxidative cross-linking of the amine-containing polysiloxane to give hydrophobic fabrics with low leaching in organic solvents and only minor decreases in tensile strength and elongation at break. This procedure is amenable to synthetic fabrics (polyester) as well as natural fabrics (cotton, linen, and silk) and paper. Antimicrobial inactivation from the surface amine functionalities was demonstrated to depend on the weight % of amine groups on the polymer, with an inactivation of >98% observed for *E. coli* and MRSA. The RBL attached to the polymer enables aPDI through irradiation with green light, resulting in 28× and 3× increases in *E. coli* and MRSA inactivation rates, respectively, compared with the samples left in the dark. While further work is needed to improve the photostability of the employed photosensitizer, we anticipate that the simplicity of this treatment procedure and compatibility with a wide array of textiles may broaden the applicability of polymer-based antimicrobial coatings in healthcare settings.

We also demonstrated the antiviral activity of the photo-activated polymer against SARS-CoV-2, resulting in up to 90% decrease in infectious virus compared to untreated and dark-treated fabric. Although vaccines and universal masking in healthcare settings have greatly reduced SARS-CoV-2 transmission, the emergence of variants with increased transmissibility and higher patient viral loads warrants development and testing of additional measures for preventing transmission in high-risk settings. $^{30,51}$ The use of treated masks that provide a barrier as well as an active means of viral inactivation may further prevent the spread of highly transmissible SARS-CoV-2 variants to susceptible individuals. Importantly, due to the nonspecific antiviral mechanism of $^{1}O_2$, we predict that these treated fabrics will have broad-antiviral activity and far-reaching applications against many important human-enveloped viruses transmitted in healthcare settings such as influenza A virus and other coronaviruses.

**ASSOCIATED CONTENT**

 Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsami.1c14178.

General experimental setup and procedures, synthesis, antimicrobial and antiviral procedures, spectroscopic characterization (UV−vis and FT-IR), SEM images of the treated materials, water contact angles, and images of the treated material (PDF).

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**Notes**

The authors declare the following competing financial interest(s): The authors have filed a patent on this work.

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