

Development of self-cleaning and antibacterial properties on cotton fabric using silver nanoparticles and PFOTS

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Abstract

The demand for superhydrophobic fabric with self-cleaning and antibacterial features as potential practical applications has been rising steadily with the passage of time. In this research work, the wet chemical method is used to manufacture cotton with a superhydrophobic coating. Thus, Nanoparticles of silver were produced by using an in situ method. Afterward, the fabric is treated with a solution of perfluorooctyltriethoxysilane to make it superhydrophobic. The cotton fabric performs remarkably with exceptional hydrophobicity as it had a contact angle of $157 \pm 1^\circ$ and less than 10° in sliding angle. Water droplet's dynamic behavior is also studied so that the superhydrophobic fabric development can be proven. The fabric shows excellent chemical durability by performing chemical tests. The antimicrobial and self-cleaning properties are greatly enhanced by the superhydrophobic coating on cotton. Particle density on the superhydrophobic cotton before and after self-cleaning was found 5.1 and 0.4 mg/cm². In addition, the treated cotton fabric also exhibited superior antibacterial properties against *Escherichia coli* bacteria. Therefore, the developed multifunctional cotton fabric can be used in real-life applications.

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Highlights

- Cotton fabric enables to repelling of dirt, oil, and water-based stains more effectively.
- Silver nanoparticles possess potent antibacterial properties.
- Incorporation of PFOTS controls the release of silver into the environment.
- Versatile applications from healthcare and hospitality to outdoor and athletic apparel.

KEYWORDS

in situ method, medical applications, multifunctional properties, nanoparticles, superhydrophobic

1 | INTRODUCTION

The use of functional textile materials has been on the rise recently. The advantages of functional textile materials are: high air permeability, mechanical strength, flexibility, and a plethora of surface modification options that might induce multifunctional properties—have led to their widespread adoption as substrates. Among the different textile substrates, cotton fabric is mostly used.¹ The surface modification of cotton fabric allows multifunctional uses such as superhydrophobicity, antibacterial, self-cleaning, physiotherapy, antistatic, and anti-ultraviolet fabrics, moisture absorption, and so forth.^{2,3}

The antimicrobial and self-cleaning characteristics of cotton raised interest in the development of a superhydrophobic coating on the fabric.⁴ The surfaces that create a contact angle of water droplets greater than 150° are known as superhydrophobic surfaces and it is naturally found in flora and fauna. Many attempts have been made to make a fabric superhydrophobic. A superhydrophobic fabric with a water contact angle of 160 ± 1° was created by a group of researchers employing TiO₂ and perfluorodecyltriethoxysilane (PFOTS).⁵ For oil–water separation and self-cleaning, they created a robust flower-shaped TiO₂-coated cotton fabric in a separate study. The fabric was superhydrophobic.⁶ Furthermore, a superhydrophobic fabric was created with a water contact angle of 161 ± 2° utilizing ZnO nanorods and dodecyltrimethoxysilane (DTMS).⁷ Tissues coated with silica nanoparticles and tetraethyl orthosilicate (TEOS) are also superhydrophobic.⁸ Fabrics made with fluoroalkyl siloxane are also superhydrophobic.⁹ Also, for oil–water separation, superhydrophobic cotton was made utilizing poly (dimethyl siloxane) and SiO₂ NPs.¹⁰ Moreover, a fabric with a contact angle of 173° that is superhydrophobic was created utilizing TEOS and SiO₂.^{11,12}

Problems with durability, environmental impacts, and ineffective methods of merging functions are common in

the current state of research on self-cleaning and antibacterial textiles. Using silver nanoparticles and perfluorotriethoxysilane (PFOTS), this study fills these gaps by creating a scalable, environmentally friendly, and long-lasting method that simultaneously treats antibacterial and hydrophobic properties. This work's originality comes from the integrated approach it takes, which provides a novel and useful way to improve the efficiency and durability of cotton fabrics. Besides the superhydrophobic properties, the self-cleaning property is also very important. Self-cleaning is known as cleaning the dirt and dust particles from the surface without any applied external forces. Regarding the self-cleaning technology, the formation of spherical water droplets may remove the dust particles.^{13–15} Recently nanotechnology has played an important role in developing self-cleaning properties. In this work, at first, the cotton fabric was treated with NaOH to induce the functional groups (—OH) on the fabric surface. After that, the fabric was immersed in an AgNO₃ solution, followed by treatment with ascorbic acid. Here, ascorbic acid aids the formation of silver nanoparticles (AgNPs). Then the AgNPs-coated cotton fabric was modified with 1H, 1H, 2H, and 2H PFOTS to introduce the hydrophobicity as well as self-cleaning properties. Moreover, the antibacterial properties were also examined against *Escherichia coli* bacteria.

2 | MATERIALS AND METHODS

2.1 | Materials

A 100% cotton knitted fabric with an area density of 127.4 g/m² was sourced in Lahore, Pakistan. Before the use of cotton, distilled water and ethanol were used to clean the cotton. Several chemicals were purchased from Sigma-Altrich, including sodium hydroxide, ascorbic acid, silver nitrate, and PFOTS.

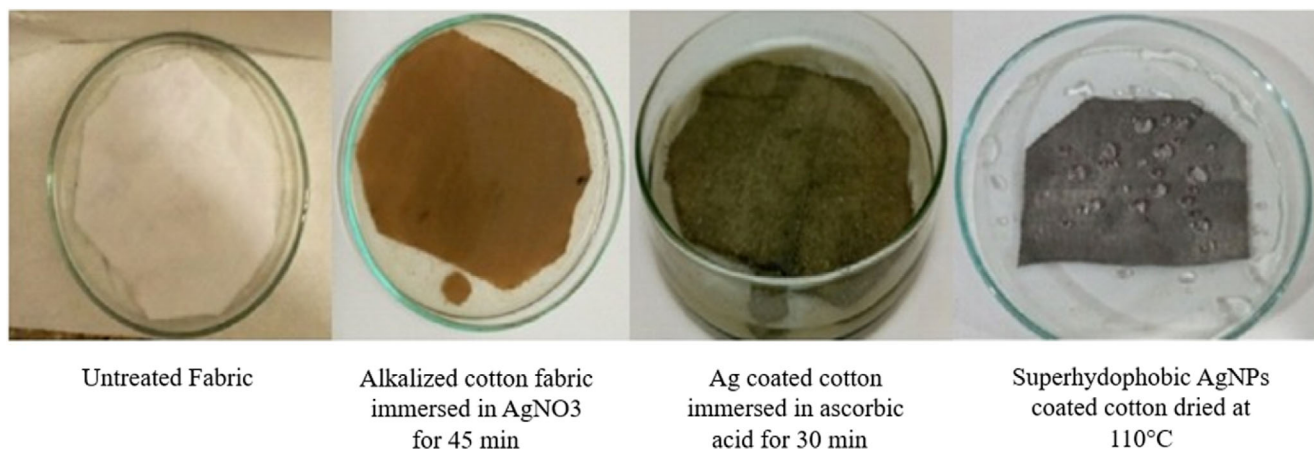


FIGURE 1 Preparation of superhydrophobic cotton fabric using PFOTS and AgNPs.

2.2 | Fabric preparation

It begins by immersing clean cotton textile in a solution of NaOH at room temperature for 10 min, effectively alkalinizing the fabric. Distilled water is applied on the alkalinized fabric's external surface to eliminate any excess NaOH. Next, the fabric is immersed in a 0.5 mol/L AgNO_3 solution for 45 min, allowing for the silver ions to bind with the fabric. Following this step, the fabric is placed in a solution of 0.1 mol/L ascorbic acid for an additional 30 min to reduce any excess silver ions on the surface of the fabric. Lately, the fabric is dried in an oven at 65°C for 45 min, ensuring that it is completely dry and ready for use. This comprehensive treatment ensures that the cotton textile is properly prepared and enhanced for its intended purpose Figure 1.

2.3 | Fabrication of PFOTS

A solution containing 1H, 1H, 2H, and 2H PFOTS was used to immerse the cotton fabric coated with AgNPs for 60 min at room temperature. Following this treatment, the cotton fabric that had been treated was initially dried at 80°C for 15 min before being cured for an extra hour at 110°C .

Cotton fabric's superhydrophobicity was achieved by the sample immersing in a solution of AgNO_3 and a solution of PFOTS. Table 1 indicates the samples prepared and their respective treatment.

The superhydrophobic-coated cotton fabric was produced by varying the molar ratios of AgNO_3 , which is crucial for creating surface roughness through the formation of silver nanoparticles. These nanoparticles enhance the micro and nanoscale texture of the fabric, a key factor in achieving superhydrophobic properties. Subsequently, the fabric was treated with PFOTS, which imparts

hydrophobicity by forming a low surface energy coating on the cotton fibers. The combination of the rough surface provided by AgNO_3 and the hydrophobic layer from PFOTS results in a fabric with a water contact angle greater than 150° , indicating excellent superhydrophobicity and self-cleaning capabilities.

2.4 | Characterization

Uncoated and coated fabrics were analyzed by using a contact angle measuring instrument (DataPhysics OCA 15EC). The surface morphology of cotton samples was examined through a scanning electron microscope (SEM) (Bruker D8 Advance). Functional groups were analyzed by using Fourier transform infrared (FTIR) spectroscopy. A contemporary regular washing method (sonication) for 2 h with different solvent solutions was adopted for the improvement of mechanical stability. The wettability was checked when the fabric was dried. An experiment was conducted to evaluate the superhydrophobic fabric's self-cleaning ability following ASTM D7334-08. This was done by applying water to the fabric's surface and then spreading dust particles on it. *E. coli* bacteria were used to determine the antibacterial properties of both untreated and coated cotton fabric. A total of 14 g nutrient agar dissolved in 500 mL of distilled water. Boiled for 10 min at 120°C until its color changes from yellow to transparent then it was cooled down. Equally spread on plates. A $150\ \mu\text{L}$ of each bacterium was marbled on each plate using cotton swabs and left until dried. By using a cork borer, four wells of 8 mm diameter were made in plates. Different samples of fabric were loaded in wells and incubated for 24 h at 37°C then a zone of inhibition (ZOI) was observed around the wells. ISO 20645:2004 standardized method was used for antibacterial testing.

3 | RESULTS AND DISCUSSION

3.1 | SEM and EDX analysis

Silver nanoparticles and 1H, 1H, 2H, and 2H PFOTS-coated cotton samples surface morphology is stated in Figure 2A,B. The SEM image provided evidence of effective modification of the cotton surface. Inherent roughness was created by fabric yarns on the cotton surface where the gaps were filled by water. It is observed that higher surface scratches were found in coated fabric compared to uncoated fabric.

The elemental composition of superhydrophobic-coated cotton was examined using energy dispersive spectrometry (EDS). Figure 2C shows the presence of C, O, Ag, F, and S on superhydrophobic cotton. The results of EDS confirm the even coating of PFOTS and AgNPs on samples. The table shown in Figure 2C confirms the presence of 17.75 wt % of C, 23.66% of O, 10.31% of F, 2.33% of S, and 45.95% of Ag in the coated cotton fabric. The cotton surface had an inherent roughness due to the inducement of coated materials confirmed by the EDS test that was able to fill the gaps with water. The number of surface scratches on coated fabric was significantly higher than that on uncoated fabric.

TABLE 1 Sample preparation and treatment.

NaOH treatment	AgNO ₃ treatment	Ascorbic acid treatment	PFOTS treatment	Drying and curing
10 min in 0.1 mol/L NaOH	45 min in 0.5 mol/L AgNO ₃	30 min in 0.1 mol/L ascorbic acid	60 min in 0.02–1.00 mol/L PFOTS	15 min at 80°C, then 1 h at 110°C

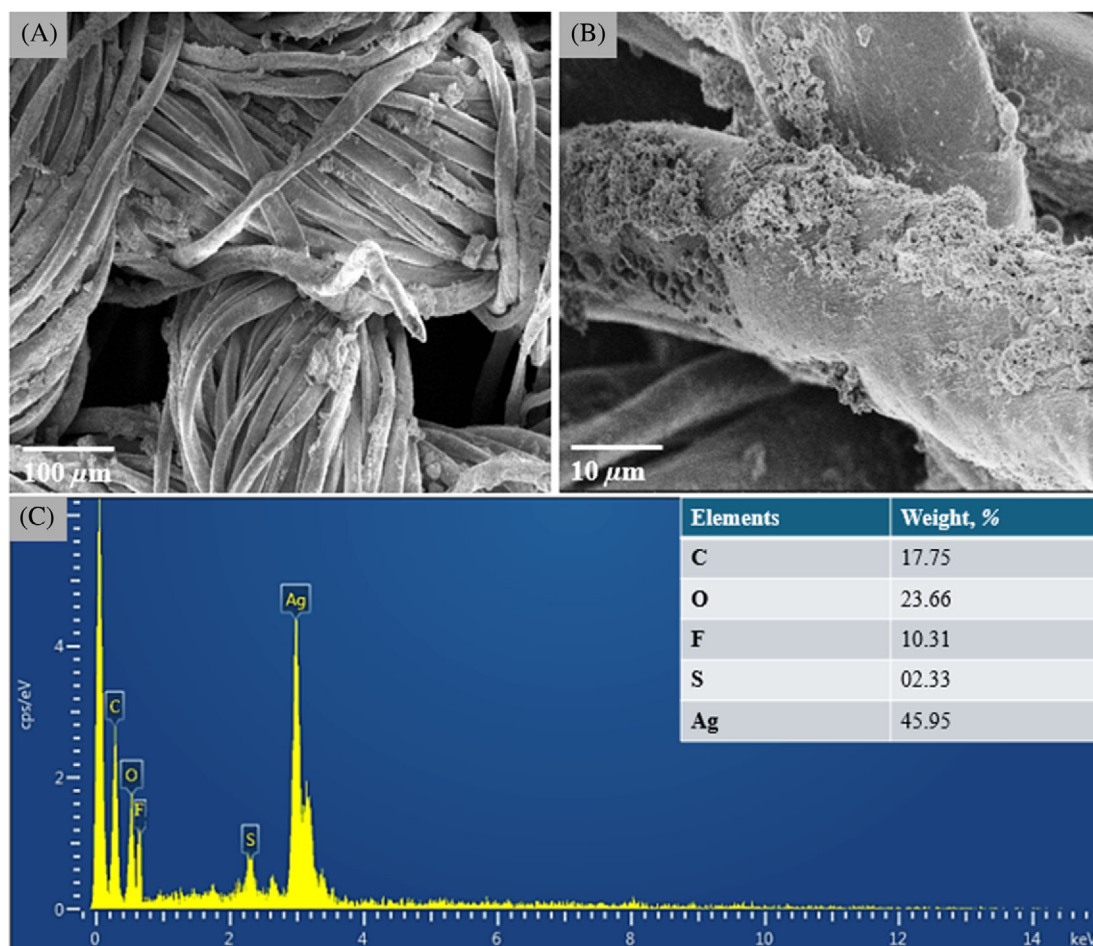


FIGURE 2 Surface morphology of PFOTS-coated sample at 500× (A) and 4000× (B) magnifications and energy dispersive spectrometry spectrum (C).

3.2 | FTIR analysis

The functional group on superhydrophobic-coated cotton was studied using FTIR. The comparative visual of the

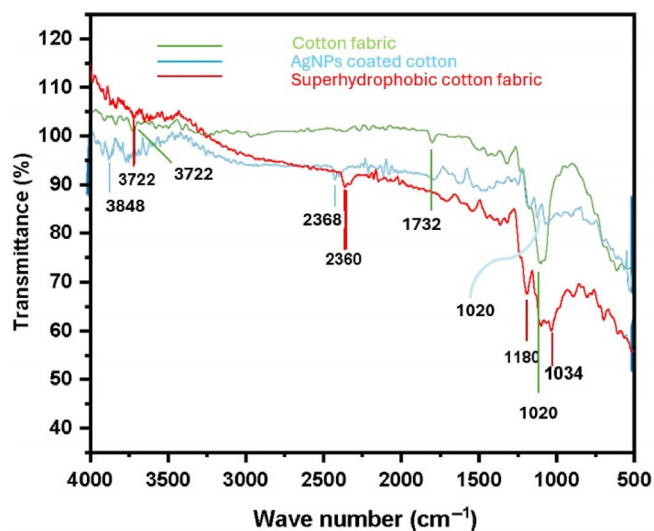


FIGURE 3 Uncoated cotton fabric, AgNPs-coated cotton fabric, and PFOTS-coated samples Fourier transform infrared spectroscopy analysis.

FTIR analysis of uncoated cotton fabric, AgNPs-coated cotton fabric, and superhydrophobic-coated cotton fabric is illustrated in Figure 3.

Various peaks have emerged at various wave numbers. The presence of the hydrophilic tail of PFOTS on the surface can be observed by the peaks at 1034, 2368, and 3722 cm^{-1} which correspond to the $-\text{C}-\text{C}-$, CO_2 , and $-\text{C}-\text{H}-$ functional groups, respectively. Water and dirt are repelled by the stable hydrophobic backbone that is provided by the $-\text{C}-\text{C}-$ bonds.¹⁶ When combined with water, the CO_2 groups produce a mild acid that dissolves organic stains, improving the self-cleaning process.¹⁷ The hydrophobicity, which is the ability to repel water and dirt, can be enhanced by the $-\text{C}-\text{H}-$ groups.¹⁸ The roughness of the fabric surface creates air pockets. These pockets increase the water contact angle and achieve the superhydrophobicity of a fabric.^{19–22}

3.3 | Droplet impact behavior

The nature of droplets that impact the superhydrophobic fabric at different velocities is illustrated in Figure 4. A pancake-shaped water droplet was formed at low velocity.

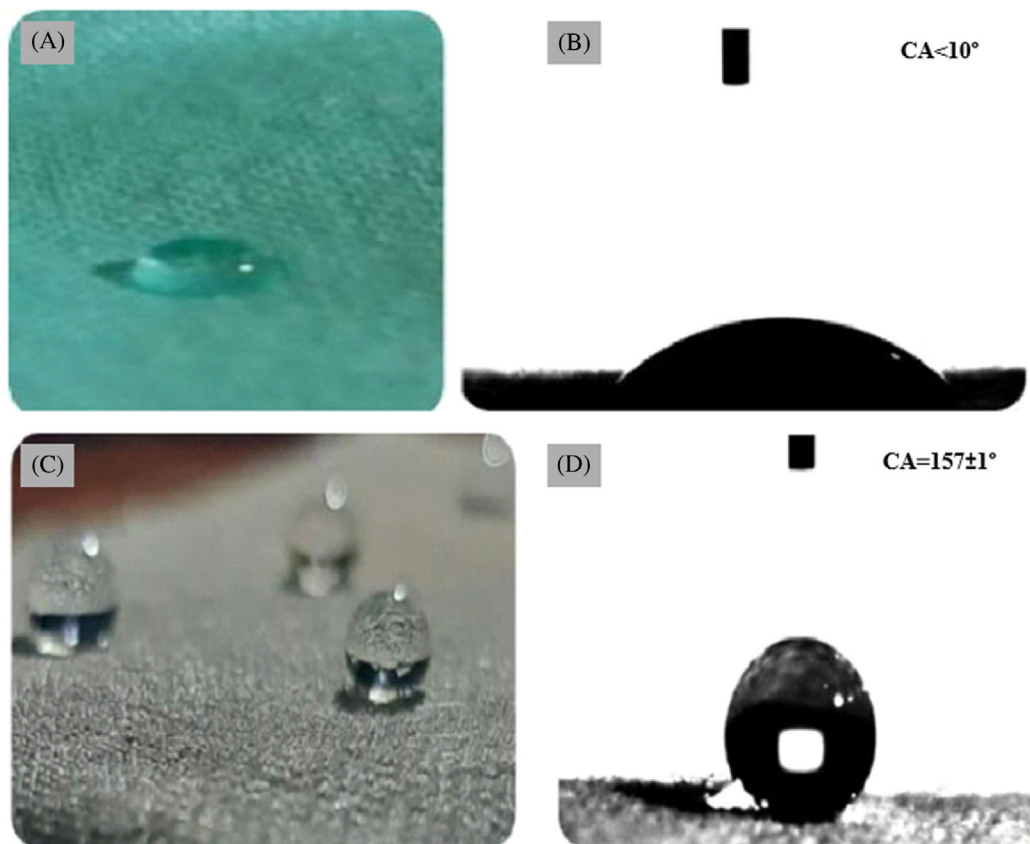


FIGURE 4 The contact angle of untreated cotton fabric (A, B) and contact angle of PFOTS-coated cotton (C, D).

The shape of the water droplet is changed when it hits the solid surface of the superhydrophobic samples and also restores K.E. and bounces back. A barrier is created by air pockets on the fabric's surface.²³ Cotton that has been modified to be superhydrophobic displays water-repellent properties after being modified, with a water contact angle of $157 \pm 1^\circ$ and a water sliding angle below 5° .

3.4 | Self-cleaning efficiency

Figure 5 represents the self-cleaning property of coated and uncoated samples and the results are shown in Table 2. Dust particles were spread on both samples.

Water drops were subjected to the samples. The size of dust particles was 5 and 20 μm . Particle density on the superhydrophobic cotton before and after self-cleaning was 5.1 and 0.4 mg/cm^2 . Water droplets stick to the sample surface when they are subjected to the exterior surface of the uncoated sample.

Additionally, floating water moves on the surface of the samples whereas the samples remain dirty leaving muddy water. Water droplets immediately roll when its fall on the superhydrophobic-coated surface. Furthermore, as they fall to the ground, water droplets pick up all the dust and particles, washing them away. Fabrics coated with superhydrophobic agents have remarkable abilities to clean themselves.²⁴

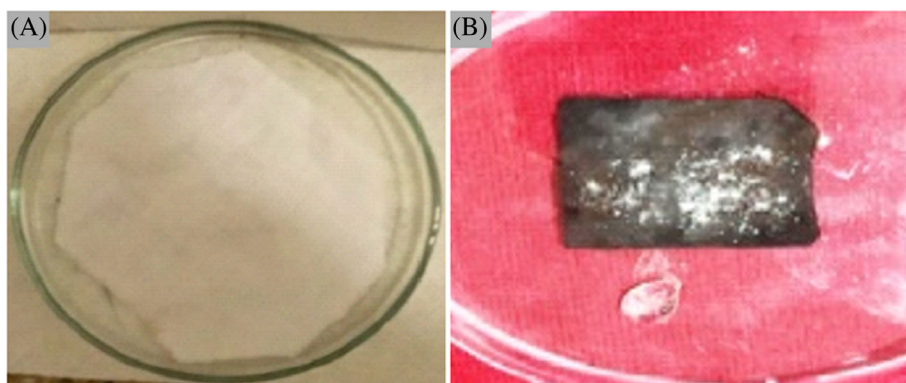


FIGURE 5 Self-cleaning performance of uncoated cotton fabric (A) and PFOTS-coated cotton fabric (B).

TABLE 2 Self-cleaning efficiency of coated and uncoated cotton fabrics.

Sample type	Dust particle size (μm)	Particle density before cleaning (mg/cm^2)	Particle density after cleaning (mg/cm^2)	Self-cleaning observation
Uncoated cotton	5–20	5.1	4.96	Water droplets stick to the surface; sample remains dirty with muddy water.
PFOTS-coated cotton	5–20	5.1	0.4	Water droplets roll off immediately, picking up and washing away dust particles.

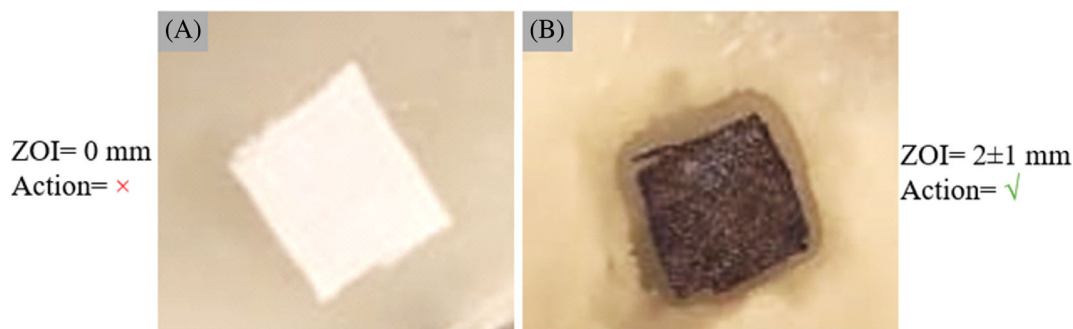


FIGURE 6 Antibacterial performance of uncoated (A) and coated (B) samples against *Escherichia coli* bacteria at 37°C after 48 h incubation.

3.5 | Analysis of antibacterial properties

The ISO 20645:2004 standard method was employed for determining the antibacterial activity. Cellulosic materials have limited practical use because of their susceptibility to microbial development. *E. coli* bacteria were used to analyze the antimicrobial activity of superhydrophobic-coated samples through quantitative methods. Bacterial growth on fabric samples of untreated and superhydrophobic coated are shown in Figure 6. Here, it is observed that there was no bacterial growth around the coated samples as there was a 2 mm ZOI that confirms it eliminated the bacterial growth. However, there was the development of bacteria in noncoated samples after 24 h of incubation.

There is less bacterial growth around the cotton fabric when AgNPs are used because of their well-known antibacterial properties.^{25,26} Light irradiation inhibits the development of bacteria by reducing their e^-/h^+ generation. By degrading the bacterial protein, the h^+ in Ag's valence band forms ZOI which inhibits the growth of microbes.

4 | CONCLUSION

The present work extensively focuses on functionalization techniques of cotton fabric for real-life applications. In this study, the wet chemical method is chosen to develop superhydrophobic fabric. SEM, EDX, and FTIR analysis showed the successful coating of silver nanoparticles and perfluorooctyltriethoxysilane solution. The water contact angle and water sliding angle for the coated fabric are $157 \pm 1^\circ$ and less than 10° respectively. The superhydrophobic cotton fabric also does a great job of cleaning itself. The created functional fabrics were also very effective in killing the bacteria *E. coli*. The newly developed multifunctional technical fabric has excellent antibacterial and hydrophobic qualities, making it suitable for use in medical and athletic settings.

AUTHOR CONTRIBUTIONS

Mahnoor Khan, Muhammad Adnan saeed, Sultan Ullah, and Md. Reazuddin Repon have contributed to conceptualization, methodology, characterization, validation, original draft preparation, and visualization. Arnob Dhar Pranta, Nurzod Yunusov, and Md Monir Hossain have contributed to materials, manuscript editing, and reviewing. All authors have read and agreed to the published final version of this manuscript.

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CONFLICT OF INTEREST STATEMENT

The authors have no relevant financial or nonfinancial interests to disclose.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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