Biointerface Research in Applied Chemistry
Open-Access Journal (ISSN: 2069-5837)

Article
Volume 13, Issue 6, 2023, 591
https://doi.org/10.33263/BRIAC136.591

Effect of CuO Antibacterial Coating on Cotton, Polyester, and Blend Wool Fabrics

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Scopus Author ID 57315067900
Received: 22.12.2022; Accepted: 9.02.2023; Published: 7.04.2023

Abstract: In the present work, the copper oxide nanoparticles (CuO NPs) were successfully synthesized via the sol-gel method to coat on cotton, polyester, and blend wool fabrics. The solution was deposited onto the fabrics by a dip-dry process. The sol-gel synthesis process was confirmed to be successful through the presence of the complex nature of pomegranate rind extract (PRE) in CuO NPs solution as determined by Fourier transform infrared spectroscopy (FTIR) and the existence of copper in all coated fabrics by and dispersive X-ray spectroscopy (EDX) analysis. All the coated fabrics show antibacterial properties towards all three species of gram-positive bacteria, namely *Brevibacterium linens*, *Cutibacterium acnes*, and *Staphylococcus epidermidis*. The maximum inhibition zone was observed at 7 mm on coated blend wool fabric towards *B. linens*. The CuO-coated fabrics have established good antibacterial properties and could be used in medical textiles and everyday clothing to prevent bacterial infection.

Keywords: antibacterial fabric; copper oxide nanoparticles; pomegranate rind extract; sol-gel synthesis.

1. Introduction

Fabrics are important in human life as they fulfill numerous daily practical, economic, social, and aesthetic roles. Choosing the right fabric is vital as it can be a potential site for the propagation of microorganisms which could lead to the deterioration of fabric strength, defacement, and odor [1]. The production of antibacterial fabric is one of the alternatives to avoid such impacts. The antibacterial fabric has been used for decades in the healthcare industry to make surgical gowns, bed linens, bandages and dressings, and curtains in order to sustain high hygiene standards [2]. Thus, it helps to protect the patient from getting a further infection by preventing the growth of the bacteria. Due to health and hygiene awareness, its application also expanded for apparel, home textile, commercial, and industrie[3]. The high demand for better quality products and growing lifestyle, especially in sports and high disposable income, is leading to the increased use of antibacterial textiles. The antibacterial fabric also has an odor-control feature, making it more popular nowadays.

Antibacterial agents from metal-based are used on fabric to inhibit the growth of bacteria. This agent can be introduced into the fabric during the production or finishing process of the fabric by various methods. Sol-gel is one approach to producing metal nanoparticles (NPs). It involves converting a solution from a liquid 'sol' into a solid 'gel' phase. Sol-gel could simplify the process, and it does not require special or expensive equipment, eventually making it more economical [4]. The sol-gel method involves three processes: hydrolysis, condensation, and drying. Firstly, the metal hydroxide solution is produced through hydrolysis of the metal precursor, followed by the condensation process to form three-dimensional gels. Subsequently, the drying process took place and converted the product to xerogel or aerogel based on the mode of drying [5]. The sol-gel method ensures a high-quality coating with any shape, such as thin films, fibers, monoliths, porous membranes, composites, and powders [6].

Copper oxide (CuO) is a metal-based with antibacterial properties. CuO is easy to synthesize, cheap, heat resistant, and stable, thus, making it a suitable agent for metal-based NPs [7]. Three mechanisms associated with antimicrobial activities include the release of copper ions, copper NPs, and biofilm inhibition [8]. CuO exerts its antimicrobial activity through membrane disruption and the production of reactive oxygen species (ROS) [9]. In order to sustain the antibacterial performance efficiency and durability of CuO NPs, it is recommended to be coated with supporting polymer matrices such as chitosan [10], epoxy resin [11], cellulose [12], and bovine serum albumin [13]. According to Sebastian and Arruebo [14], the release of ions becomes more facilitated with the polymer matrix's help to cap the NPs on the substrate. The polymer matrix from the plant also prevents bacteria growth due to the presence of antibacterial compounds such as terpenoids, lectin, flavonoids, quinones, coumarins, and tannins [3]. The pomegranate rind extract (PRE) has shown antimicrobial activity against several highly pathogenic and drug-resistant bacteria strains [15]. Hence, PRE also has high antioxidant activity, which is good for anti-cancer and anti-inflammatory properties [16]. Thus, the use of PRE as a polymer matrix could directly increase the antibacterial properties of the NPs.

The research was conducted to produce antibacterial coating fabrics, whereas its environmentally water-based sol-gel method is cheap and user-friendly. The modified CuO NPs produced were deposited onto cotton, polyester, and blend wool fabrics (20 % of wool, 33 % of Tencel, and 47 % of anti-pilling acrylic) via dip-dry. Using PRE as a polymer matrix could enhance the antibacterial properties and preserve the softness and smoothness of the fabric. Then, the antibacterial properties were compared and evaluated against three species of gram-positive bacteria, namely *Brevibacterium linens*, *Cutibacterium acnes*, and *Staphylococcus epidermidis*.

2. Materials and Methods

The CuO NPs were synthesized using the sol-gel method in an acidic environment using a water-based approach. Copper nitrate ($Cu(NO_3)_2$) with a purity of 99.6% was used as a precursor, citric acid (100% purity) as a stabilizer, and ethylene glycol as a reagent were purchased from Bendosen (Malaysia). In addition, diethyl ether (99.5% purity) was purchased from R&M Chemicals (India). The pharmaceutical grade of pomegranate rind powder was used as a polymer matrix obtained from Ayurvedic Pharmacopoeia, India. The deionized water (used as solvent) and distilled water (used to extract the pomegranate rind) were prepared in the laboratory.

Pomegranate rind was extracted by adding 4 g of pomegranate rind powder into 100 mL of distilled water and boiled for 1 hour at 130 °C. The solution was then filtered using Whatman's No. 1 filter paper. Firstly, 0.59 g of $Cu(NO_3)_2$ was diluted in 20 mL of deionized water. Then, 0.42 g of citric acid, 1 mL of ethylene glycol, and 1 mL of diethyl ether were added and vigorously stirred at 30 °C and 450 rpm with a magnetic stirrer. 10 mL of PRE was then dropwise added into the solution and allowed to react for 24 h under constant stirring and temperature.

The prepared CuO NPs solution was then deposited onto the cotton, polyester, and blend wool fabrics using the dip-dry technique. However, before the coating process was conducted, the fabrics were treated using 95% ethanol to remove the impurities from the fabric. The treatment was carried out in an ultrasonicator for 15 mins at 35 °C. The treated fabrics were then air-dried at ambient temperature for 8 h.

The functional groups present on the surface of CuO NPs solution and PRE were determined using FTIR. It was operated at room temperature with a resolution of 2 cm-1. Spectra were scanned in the region of 4000-500 cm^{-1} with attenuated total reflection (ATR) mode. The SEM-EDX was used to observe the chemical compositions and surface morphology of the fabrics before and after the deposition of CuO NPs.

The antibacterial activities test was conducted using a modified agar diffusion assay. The antibacterial activities of coated and uncoated fabric were evaluated against B. linens, C acnes, and *S. epidermidis*. Mueller Hinton (MH) agar, Tryptone Soya Yeast Extract (TSYE) agar, and Columbia Blood (CB) agar media were used to inoculate *S. epidermidis*, *B. linens*, and *C. acnes*, respectively. The bacteria species with a concentration of 9.0 × 108 CFU/mL was spread on the respective agar. 6 mm of coated fabric, uncoated fabric, and antibiotic disk were placed on the inoculated petri dish and incubated at optimum conditions (Table 1).

Table 1. The culture conditions on different bacteria species

Bacteria species	Antibiotic disc	Agar medium	Culture Conditions		
			Temperature (°C)	Incubation time (h)	Biological process
B. linens	Vancomycin	TSYE	30	72	Aerobic
C. acnes	Gentamycin	CBAB + Expired human blood	37	48	Anaerobic
S. epidermidis	Ampicillin	MH	37	48	Aerobic

3. Results and Discussion

3.1. FTIR analysis.

Figure 1 shows the spectra of PRE and CuO NPs synthesized using the sol-gel method. The strong peak observed at 3354 cm^{-1} is probably associated with −NH of the amino acid compound and bonded −OH groups of carboxylic acid [17]. The asymmetrical stretching of CH_2 appeared at 2950 cm^{-1}, and the symmetrical stretching of CH_2 at 2885 cm^{-1} of modified CuO NPs was related to amide groups [18]. The peaks that appeared at 2136 cm^{-1} of PRE and 2110 cm^{-1} of modified CuO NPs may indicate the presence of alkyne groups associated with the punicalagin compound in PRE [19]. The band appeared at 1640 cm^{-1} and 1534 cm^{-1} representing the C=C stretching vibration of aromatic rings [20] and also C=O of amides and carboxylic groups [17], which probably related to the flavonoid and amino acid contained in PRE [21,22] The peak appeared at 1237 cm^{-1} could be associated with C−O stretching of C−O−C or C−O−H groups in PRE. The band at 1450 cm^{-1} could represent the C−H bending of alkene from the methyl group. The band at 1090 cm^{-1} and 1088 cm^{-1} of CuO NPs could be

related to C−O stretching and −OH deformation of secondary alcohols. At spectra 1196 cm^{-1}, the −OH deformation of tertiary alcohol and stretching of the C−O band probably occurred [23]. The peak that appeared at 667 cm^{-1}, 602 cm^{-1}, and 553 cm^{-1} were related to the vibration of metal-oxygen (Cu−O bond), which confirmed the presence of the monoclinic phase of CuO NPs [18]. The spectrum confirmed the presence of flavonoids, a phenolic, and a wide variety of compounds of PRE which attributed to the antibacterial properties [24].

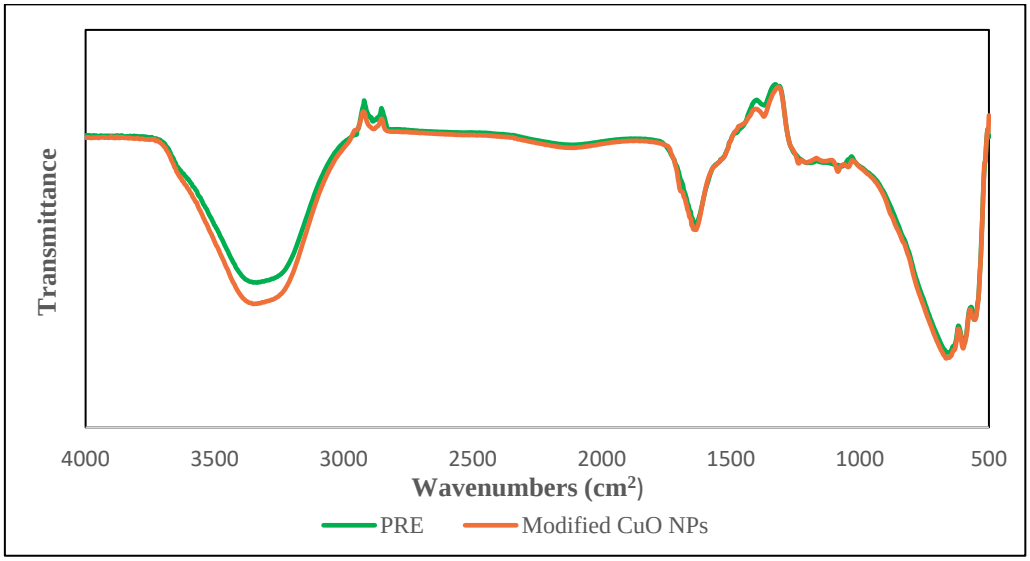

Figure 1. The FTIR spectra of PRE and modified CuO NPs.

3.2. SEM-EDX.

The surface morphology of the coated and uncoated and their chemical compositions were studied using a combination of two effective systems called SEM-EDX. All the coated fabric showed the ununiformed deposition of amorphous CuO NPs in the form of homogenous sol (Figure 2). Based on the observation, all fabric samples showed non-homogenous coating with agglomerated CuO NPs. The CuO NPs were not entirely coated in the fabrics and bound very well to the fabric. This is probably due to the low concentration of CuO precursor. The hydrogen bonds that form between the fabric and CuO NPs are weakened with the limited active site of the CuO NPs resulting in agglomeration of CuO NPs in some places of the fabric [25]. Also, the pH of CuO NPs solution at 4 attributed to the non-homogenous coating due to the insufficient OH^- ions vacant in the sol. Thus, it permitted the nucleation and the growth of CuO, and the formation of particles [26].

The elemental composition of coated cotton, polyester, and blend wool fabrics is shown in Figure 3. Based on the EDX spectrum, only two elements are present in the uncoated cotton and polyester: carbon and oxygen. The uncoated blend wool fabric contained carbon, oxygen, silicon, and sulfur. The silicon element was derived from anti-pilling acrylic[27], while the sulfur element came from wool [28], which consisted of blended wool fabric. The EDX spectrum showed the presence of copper elements in all coated fabric, verifying the CuO deposition on the coated fabric. However, the presence of potassium associated with the PRE was not found in the coated cotton spectra. Hence, due to the consumption of potassium, which acted as an activator to catalyze the reaction of cellulose from cotton fabric and cellulase enzyme from PRE [29].

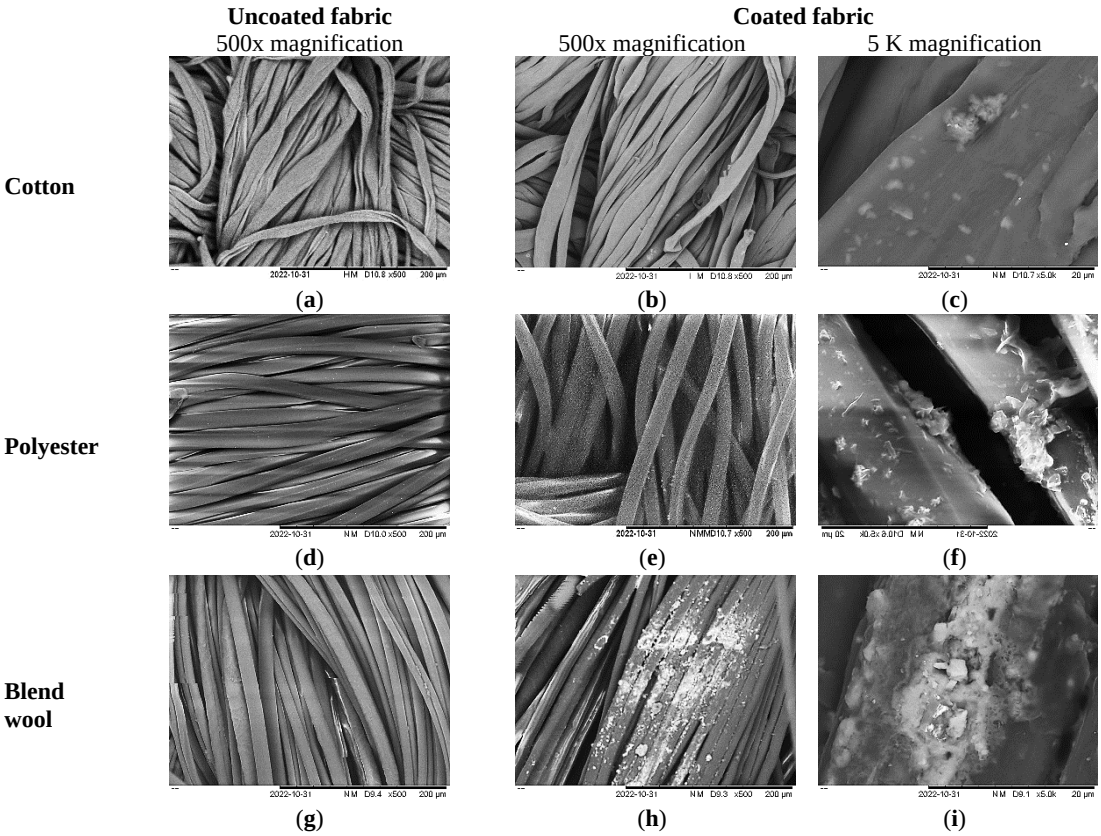

Figure 2. SEM images of **(a)** uncoated cotton fabric at 500x magnification; CuO coated cotton fabric at (**b**) 500x magnification and (**c**) 5 K magnification; **(d)** uncoated polyester fabric at 500x magnification; CuO coated polyester fabric at (e) 500x magnification and (f) 5 K magnification; **(g)** uncoated blend wool fabric at 500x magnification; CuO coated blend wool fabric at (**h**) 500x magnification and (**i**) 5 K magnification.

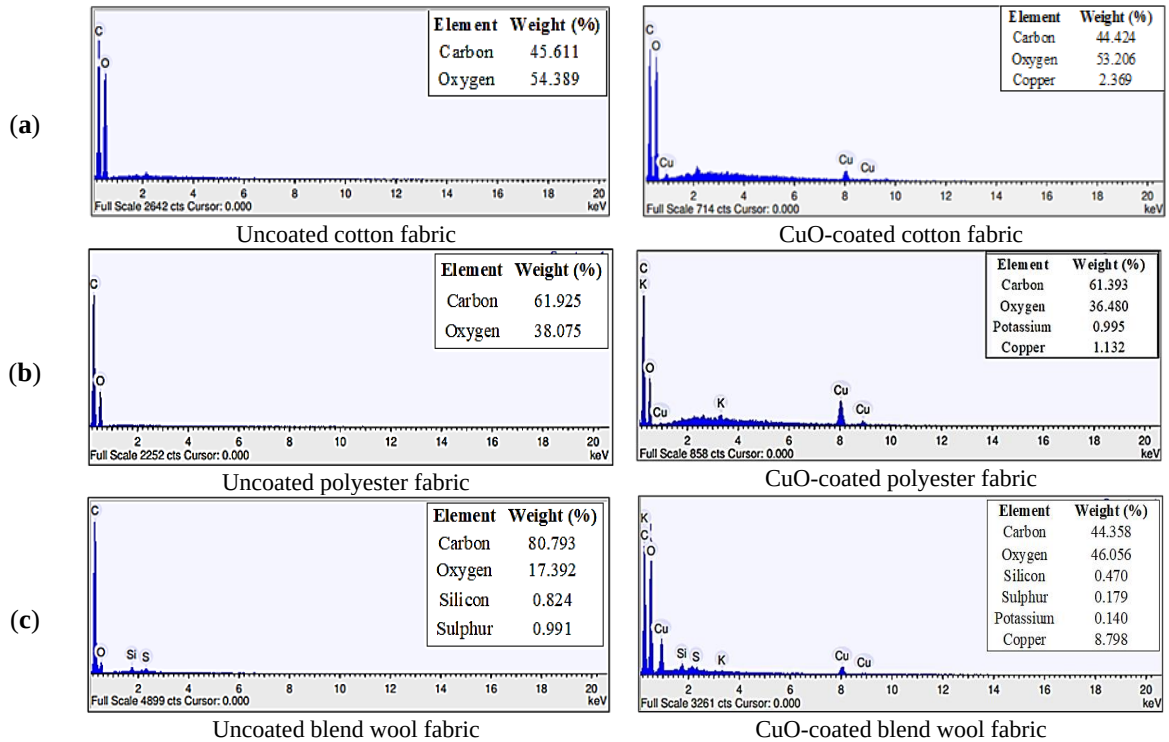

Figure 3. The EDX spectrum of the uncoated fabrics and CuO-coated fabrics.

3.3. Antibacterial activity test.

The CuO-coated fabrics were tested for antibacterial activity against three species of gram-positive bacteria. These bacteria present on the skin's surface are responsible for breaking

down amino acids to create odoriferous compounds. Thus, it could cause unpleasant odors and health problems. Therefore, the production of antibacterial coating could solve the problem. The inhibition zone of the CuO-coated fabric is listed in Table 2. The results showed all the CuO-coated fabrics have antibacterial activity against all tested species of bacteria (Figure 4). The CuO-coated blend wool fabric showed the strongest antibacterial activity against *B. linens* with an inhibition zone of 7 mm, probably the due thickness of the fabric and the agglomerates of CuO NPs on yarn fiber. The minimum inhibition zone observed was 1 mm on CuO-coated cotton and polyester fabric against *C. acnes*. The 1 mm inhibition zone also was observed on CuO-coated polyester fabric against *B. linens*. All the coated fabric samples showed similar trends of antibacterial activity against *S. epidermidis* with an inhibition zone of 2 mm.

Table 2. The inhibition zone value (mm) of antibiotic, coated cotton, coated polyester, and coated blend wool fabrics against *B. linens, C. acnes, and S. epidermidis.*

Bacteria Species	Inhibition Zone (mm)			
	Antibiotic	**Cotton**	**Polyester**	**Blend wool**
B. linens	Vancomycin			
	10	3	1	7
C. acnes	Gentamicin			
	15	1	1	2
S. epidermidis	Ampicillin			
	17	2	2	2

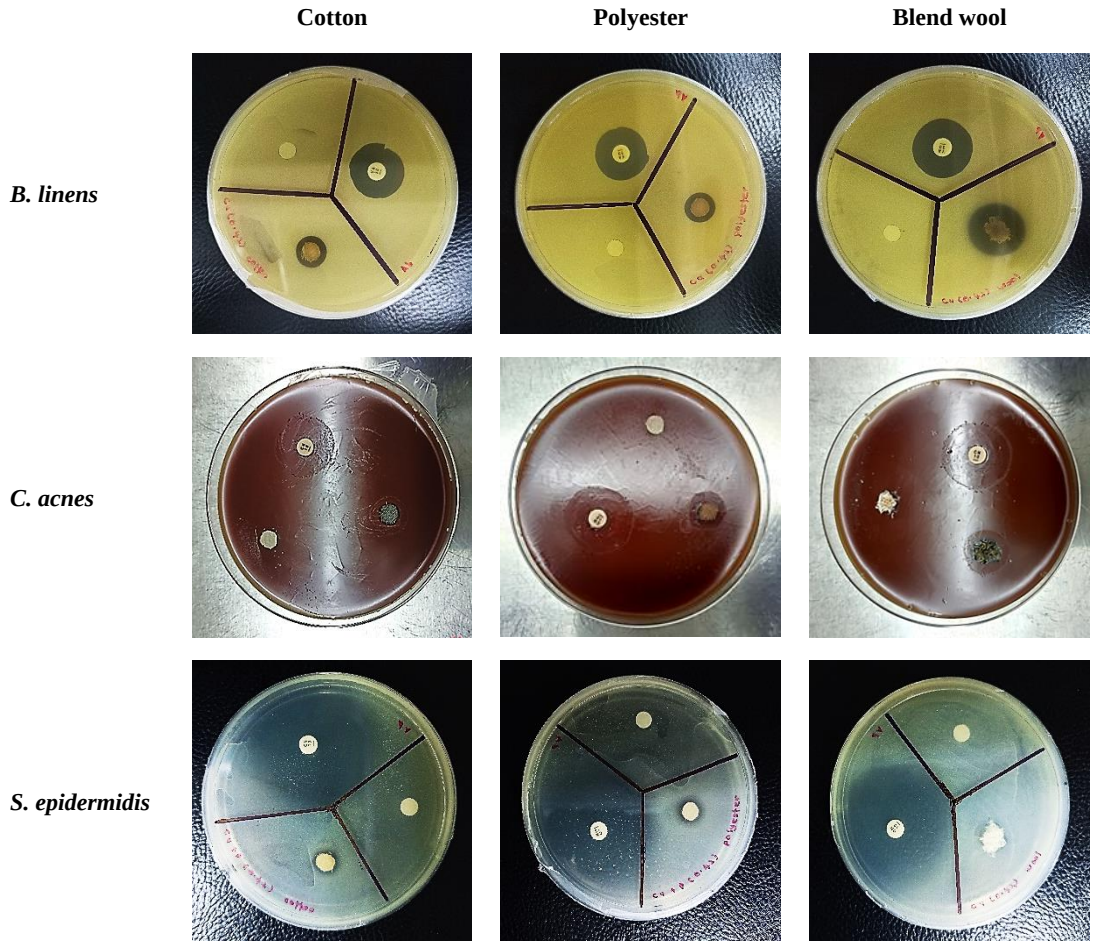

Figure 4. The antibacterial activities of the CuO-coated fabrics against *B. linens, C. acnes,* and *S. epidermidis.*

Various factors such as concentration, chemical composition, surface charge, particle size and shape of NPs, and exposure time of NPs to bacteria species contributed to the bactericidal effect of CuO NPs. Reactive oxygen species (ROS) could be a possible mechanism

for the destructive action of CuO NPs on bacteria. The attachment of CuO NPs to the bacteria cell causes damage to the membrane due to structural and functional interruption, which allows the penetration of the NPs into cells. Hence, leakage of the intracellular component occurs due to oxidative stress and leads to cell functionality loss[30–32]. The death of bacteria cells could also be caused by inhibiting bacteria activities such as enzyme functions, metabolisms, and transportation due to the penetration of Cu^{+} ions into the cell.

The presence of phenolic compounds in PRE also contributes to the antibacterial activity of the NPs [33]. The phenolic have an anti-infective activity due to their potential complex with proteins through hydrogen bonding or covalent linkages [34]. The phenolic, polyphenols, and other components in PRE act as active components, which could decrease the membrane fluidity of bacteria cells and directly damage their cytoplasm [35,36]. Thus, the bactericidal effect of these compounds included the damage of microbial respiratory chain [37], damage to the electron transport mechanisms [36], an increase in the permeability profile of bacteria [32], damage to cellular metabolism [38], and destruction the genetic material and energy production enzymes [36].

4. Conclusions

The antibacterial coating was successfully synthesized using $Cu(NO_3)_2$ as a precursor and pomegranate rind extract as a polymer matrix via a water-based sol-gel method under an acidic condition. The presence of a wide variety of natural compounds in CuO NPs has confirmed the occurrence of hydrolysis and condensation of sol-gel reaction. Furthermore, the formation of the Cu−O bond has assured sol-gel synthesis's success in producing CuO NPs. The EDX analysis verified the deposition of CuO NPs in cotton, polyester, and blend wool fabric with the presence of copper elements in all coated fabrics. The SEM images showed amorphous CuO NPs coating with non-homogeneity of the coating on all fabric types. However, all the fabric samples manage to preserve the softness and smoothness after the coating process, which is important for the comfort of the wearer. Crucially, all the fabric samples imparted good antibacterial properties against all tested species of gram-positive bacteria, which gave bad odor to the human body when interacting with skin and sweat. In the future, further research should focus on the effect of CuO NPs against fungi, viruses, and gram-negative bacteria.

Funding

This research was funded by Toray Science Foundation, Japan, under MTSF Science & Technology Research Grant 2020.

Acknowledgments

The authors wish to extend their appreciation to Universiti Malaysia Pahang (UMP) and Science & Technology Research Institute for Defence (STRIDE), Ministry of Defence, Malaysia, for granting access to the research facilities necessary to conduct this study.

Conflicts of Interest

The authors declare no conflict of interest. The funders had no role in the design of the study, in the collection, analysis, or interpretation of data, in the writing of the manuscript, or in the decision to publish the results.

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