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Cotton Fabric Coating by rGO and Polymethylsiloxane Layer with Antibacterial, Hydrophobic and Photothermal Properties

Lintang Kumoro Sakti[1], Geometry Amal Nur Sheha[1], Cut Putri Saba Miftah Radhiyah[1], Jakariya Nugraha[2], Diana Rakhmawaty Eddy[1], Muhamad Diki Permana[1,3], Iman Permana Maksum[1] and Yusi Deawati[1,*]

[1]*Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Padjadjaran, Sumedang 45363, Indonesia*
[2]*National Research and Innovation Agency, Bandung 40272, Indonesia*
[3]*Center for Crystal Science and Technology, University of Yamanashi, Yamanashi 4008511, Japan*

([*]Corresponding author's e-mail: yusi.deawati@unpad.ac.id)

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Abstract

The cotton fabric, which is widely used as personal protective equipment, is vulnerable to droplets that may contain pathogens. Thus, the functionalization of cotton fabric is usually carried out to get the desired properties. Graphene-based materials have some interesting properties like hydrophobic, photothermal, and antibacterial properties that can be used to modify cotton fabric. This research has made a cotton fabric coated with graphene from coconut shells and polymethyl siloxane. Graphite was obtained from burning coconut shells, and the modified Hummers method was used to get a graphene oxide solution (GO). A graphene oxide layer on cotton fabrics was added by the dip-pad-dry process, followed by reduction using ascorbic acid to produce a reduced graphene oxide (rGO) and immersion of cotton fabrics with methyl trichlorosilane in n-hexane to produce a polymethyl siloxane (PMS) layer. Scanning electron microscope (SEM) and Fourier-transform infrared (FTIR) are used to determine the morphology and functional groups in rGO-PMS-coated cotton fabric. Contact angle measurements to determine the hydrophobicity of the rGO-PMS-coated cotton fabric, which exhibits hydrophobic properties with a contact angle of 146.3 °, which can show self-cleaning ability while preventing water droplets from entering the fabric. Antibacterial activity was carried out using the disc diffusion method against Staphylococcus aureus and delivered a good result. The rGO-PMS-coated cotton fabric also shows photothermal capability when irradiated with infrared lamps.

Keywords: Antibacterial activity, Coating, Cotton fabric, Hydrophobic, Photothermal

Introduction

Healthcare workers (HCWs) are protected by personal protective equipment (PPE) from exposure to highly pathogenic microorganisms through body fluids and respiratory droplets [1]. Cotton fabric is used in surgical gowns, bed sheets, patient pajamas, and health worker uniforms [2]. However, cotton fabric fibers are a medium that is easily overgrown with bacteria [3]. To deal with this issue, cotton fabrics are typically functionalized with additional materials to provide the necessary qualities.

For antipathogenic uses, graphene materials have been recognized as promising alternatives [4]. Graphene can cause membrane and oxidative stress in bacteria [5]. Ag/rGO-coated cotton fabric exhibit hydrophobicity with a water contact angle in the range of 90 - 150 ° [6]. Water droplets will roll over the surface of a layer that has hydrophobic characteristics. It will remove dirt that has adhered to the surface, enabling this layer to self-clean [7]. Moreover, near-infrared optical adsorption in graphene and its derivatives is strong, making them effective photothermal treatment agents [8]. Graphene-based nanomaterials can be employed for photothermal therapy and other treatments since they have inherent near-infrared (NIR) absorption characteristics [9]. Moreover, photothermal treatment has a broad antibacterial range and doesn't lead to bacterial mutations [10].

Graphene oxide (GO) and reduced graphene oxide (rGO) sheets are typically synthesized using Hummers' method, which requires the use of highly pure graphite, which is relatively expensive for mass production of graphene [11]. Graphite is divided into two types: Natural graphite and synthetic graphite, which can be produced through graphitization using heat-induced hydrocarbon precursors [12]. Carbonaceous wastes from natural sources like wood, leaves, bagasse, and fruit can produce graphene [11]. Rice husks and coconut shells are agricultural wastes that have been used as raw materials for sustainable and cheaper graphene production [13-15]. Because of its high content of carbon of 74.3 %, coconut shell can be utilized as a natural source of graphene [16]. GO was produced using a modified Hummers method from the graphite made from coconut shells waste [14].

A silane coupling agent is typically utilized to improve the adhesion of the rGO to the cotton fibers [17]. A combination of rGO and methyl trichlorosilane (MTCS) on cotton fabric can produce functional fabrics with hydrophobic and electroconductive properties [18]. This research aims to make graphene oxide from coconut shells using the modified Hummers method and layer it on cotton fabric, then add a polymethyl siloxane (PMS) layer on graphene-coated cotton fabric with MTCS. Characterization with SEM and FT-IR was performed to determine the character of the cotton fabric after functionalization, contact angle measurements were performed to determine hydrophobicity, and functionalized fabrics were tested for photothermal and antibacterial activity.

Materials and methods

Materials

The materials used in this research are primarily high-purity materials from Sigma Aldrich (St. Louis, MO, USA), including ascorbic acid ($C_6H_8O_6$, 99 %), hydrochloric acid (HCl, 37 %), hydrogen peroxide (H_2O_2, 30 %), methyl trichlorosilane (MTCS, CH_3Cl_3Si, 99 %), n-hexane (C_6H_{14}, 99 %), potassium permanganate ($KMnO_4$, 99 %), sulfuric acid (H_2SO_4, 98 %), distilled water, coconut shell as the main precursor of graphene and cotton fabrics 100 % as substrate.

Coconut shell carbonization

Coconut shells were cleaned and dried in an oven for 24 h at 120 °C, then burned using a furnace (Thermolyne, FB1310M, Thermo Fisher Scientific, Waltham, MA, USA) at 600 °C for 2 h to produce graphite. The recovery for the mass of the coconut shell after burning to obtain graphite was 67.44 %. Graphite was crushed with a mortar and then crushed using a ball mill. Then, the graphite was sieved through a sieve of 80, 120, and 200 Mesh. Graphite that passes through a 200 Mesh sieve will be used to produce graphene oxide.

Preparation of graphene oxide (GO)

This process is carried out using a modified Hummers method from Shateri-Khalilabad and Yazdanshenas [18]'s research. Graphite (1 g) was dissolved with 25 mL of concentrated H_2SO_4 and stirred continuously for 12 h at room temperature. Next, the temperature of the solution was maintained below 10 °C, then 3.5 g of $KMnO_4$ was slowly added. The mixture was stirred in a water bath at 50 °C for 2 h. Then, 70 mL of distilled water was slowly added and followed by the addition of 5 mL of 30 % H_2O_2 while stirring for 30 min. Next, the mixture was centrifuged and purified by rinsing the precipitate using 5 % HCl and then washed three times with distilled water. A total of 200 mL water was added to 1 g of graphene oxide and then sonicated for 1 h to get the 0.5 % graphene oxide solution.

The coating on cotton fabric

This procedure uses the dip-pad-dry method from Shateri-Khalilabad and Yazdanshenas [18]'s with several modifications. The cotton cloth was soaked in 0.5 % graphene for 10 min and then removed. Then, the cotton cloth was pressed using a padder tool (Padding Mangle Lab Textile Padder MU504A, Fanyuan Instrument, Anhui, China). This process was repeated three times to increase the graphene oxide charge on the fabric. The cotton cloth that had been coated with graphene oxide was then dried in an oven at 80 °C for 1 h.

The graphene oxide-coated cloth was immersed in 100 mL of 0.05 M ascorbic acid solution and constantly stirred at 95 °C for 1 h. As a result, the graphene oxide layer is reduced to provide reduced graphene oxide (rGO). Then the graphene-coated cloth was dried in an oven at 80 °C for 1.5 h.

This process aims to make hydrophobic properties. The rGO-coated cotton fabric was immersed in a solution of 0.5 M MTCS in n-hexane with a concentration for 15 min with constant stirring. After that, the fabric was rinsed with water and dried in an oven for an hour at 110 °C for the curing process. As a result, an rGO-polymethylsiloxane (PMS) layer is formed on the cotton fabric.

Characterization of modified fabric

Scanning electron microscopy analysis (SEM, Hitachi SU-3500, Tokyo, Japan) was performed with a voltage of 3.00 kV at 80× and 10,000× magnification. The analysis was carried out to determine the shape of the surface morphology of the fabric. Then, Fourier-transform infrared spectroscopy analysis (FTIR, Perkin Elmer Spectrum 100, Waltham, MA, USA) was performed to determine the functional groups, with a scanning range of 400 - 4,000 cm^{-1}. The hydrophobicity of the fabric surface was characterized using a contact angle gauge (KINO Scientific Instrument SL200KB, Boston, MA, USA) at room temperature. The contact angle value is the average of the 5 measurements.

Anti-bacterial and photothermal activity

Next, the fabric is tested for its antibacterial ability. In this case, the ability of the coated fabric was tested against *Staphylococcus aureus* ATCC 6538. Strain ATCC 6538 is crucial for routine antimicrobial susceptibility testing and investigating the development of biocide resistance [19]. The ATCC 6538 chromosomal is 2,819,210 bp (G + C content, 32.9 %) and the plasmid genomes are 28,078 bp (G + C content, 30.6 %) in size [20]. The method used is the disc diffusion method. All tools used for antibacterial testing are sterilized by heating using an autoclave. After that, nutrient agar (NA) media was put into a petri dish and allowed to harden. Bacterial cultures were inoculated on NA media. A circular fabric with a diameter of 6 mm was placed on the media that had been inoculated with bacteria. Then, the cultures were incubated for 24 h at 37 °C. The inhibition zone formed around the fabric was measured after 24 h of incubation. The result was observed and measured using a caliper. The inhibitory zone is calculated by the following formula:

$$W = \frac{T-D}{2} \quad (1)$$

where *W* is the width of the inhibitory zone (zone without bacterial growth), *T* is the total diameter of the sample and inhibitory zone, and *D* is the sample diameter.

Furthermore, photothermal analysis was carried out to see the ability of the modified fabric to absorb heat so that it has the potential to inactivate bacteria and viruses. A 10×10 cm^2 fabric was placed under an infrared lamp (Philips PAR38E 150W E27, Amsterdam, Netherlands). The heating process is carried out with the near-infrared (NIR) lamp with a wavelength specification of 700 - 1,000 nm with a voltage of 12 V on cotton fabric, rGO-coated cotton fabric, and rGO-PMS-coated cotton fabric for 5, 10, and 15 min. After irradiation, heat detection of the fabric was carried out with a thermal camera (Adafruit AMG8833, New York, USA) with the Raspberry Pi using the Python program.

Results and discussion

Surface morphology of modified fabric

Figure 1(a) shows the morphology of cotton fabric without any treatment. Thus, the cotton fibers look loose. Meanwhile, **Figure 1(b)** shows that adding rGO can make the fabric fiber denser. The deposition of rGO and PMS layers on individual threads of fabric may be seen clearly in SEM pictures obtained at a greater magnification. The rGO and PMS layers had successfully coated over the cotton fabric. In addition, white dots at 10,000× magnification indicate incomplete exfoliation of graphite [18]. **Figure 1(c)** shows an additional layer in the form of PMS, which is characterized by the thickening of the fabric fibers which is clarified at a magnification of 10,000× and the morphology of the fabric is coarser. Surface roughness is an important property of hydrophobicity and self-cleaning [21]. Because hydrophobic surfaces repel certain liquids, they have numerous uses in self-cleaning materials [22].

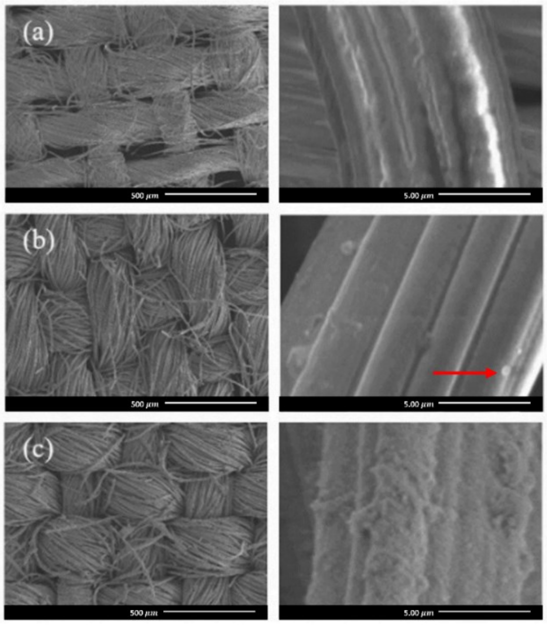

Figure 1 SEM results of 80× and 10,000× magnification for (a) cotton fabric, (b) rGO-coated cotton fabric, and (c) r-GO-PMS-coated cotton fabric.

Functional groups of modified fabric

The FTIR results in this study are shown in **Figure 2**. **Figures 2(a)** and **2(b)** show absorption peaks in the same region, peaks at 3,291 cm^{-1} which indicates the O-H stretching band in hydrogen bonds [23]. Peaks at 1,600 - 1,700 cm^{-1} are from O-H bending bands [24]. Peaks at 1,005 - 1,020 cm^{-1} indicate C-O stretching bonds originating from graphene oxide (GO) and reduced graphene oxide (rGO) [25]. Meanwhile, other peaks at 2,800 - 2,900 cm^{-1} are from the C(sp^3)-H stretching band [26]. Peaks at 1,300 - 1,400 cm^{-1} indicate C-H bonds [27]. Meanwhile, absorption bands at 890 cm^{-1} are stretching bands of CH_2 derived from cotton fabric cellulose [18]. The difference in FTIR analysis results between GO-coated cotton fabric and rGO-coated cotton fabric is that the intensity of the peak associated with oxygen-containing functional groups is reduced. **Figure 2(c)** gives a new absorption peak at 778 cm^{-1} originating from Si-C vibration [28]. The absorption peak of the siloxane layer's Si-O-Si bonds appears in the 1,000 cm^{-1} region that overlaps the C-O bonds [29]. The results of the FTIR spectrum showed that the reaction between rGO and methyl trichlorosilane succeeded in forming a chemical bond between polydimethylsiloxane with rGO which can be seen in **Figure 3**.

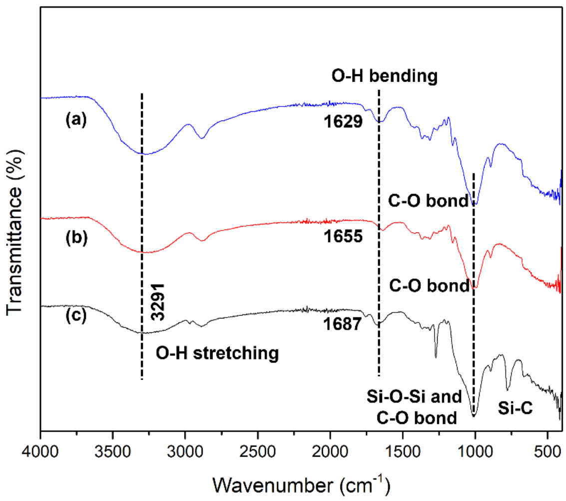

Figure 2 FT-IR results of (a) GO-coated cotton fabric, (b) rGO-coated cotton fabric and (c) rGO-PMS-coated cotton fabric.

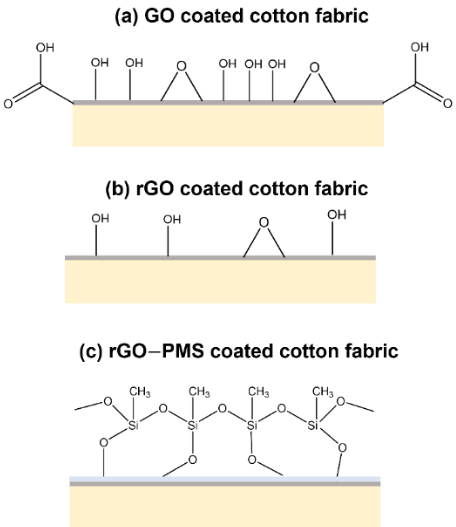

Figure 3 Functional groups on modified cotton fabrics.

Hydrophobicity properties

The water contact angle was measured to determine the wettability of the fabric. The contact angle between 0 and 90 ° indicates hydrophilicity, while more than 90 ° indicates hydrophobicity [30]. The results of the comparison of contact angle measurements on cotton fabrics, rGO-coated cotton fabrics, and rGO-PMS-coated cotton fabric are shown in **Figures 4(a) - 4(c)**.

Based on the analysis results, the contact angle of the cotton fabric is 0 °, so it is hydrophilic. Water droplets can seep directly into the fabric (**Figure 4(d)**). Cotton has hydrophilic properties because of the abundance of hydroxyl groups on its surface to absorb water quickly. In comparison, the rGO-coated cotton fabric has a contact angle of 89.6 °. This property shows that rGO is almost hydrophobic. rGO-coated cotton fabric has hydrophobic properties due to the reduction of the hydroxyl group for rGO [31]. Water droplets can still penetrate the rGO-coated cotton fabric (**Figure 4(e)**). In addition, the contact angle is increasing in the rGO-PMS-coated cotton fabric, which is 146.3 °. At this angle range, the rGO-PMS-coated cotton fabric has hydrophobic properties. The hydrophobicity of the rGO-PMS-coated cotton fabric is caused by the addition of MTCS, which increases the roughness of the nano secondary scale due to the combination of the condensation reaction and hydrolysis on the surface of the fabric substrate [31]. The advantage of this property is that it prevents droplets from seeping through the fabric, as seen in **Figure 4(f)**. This property is essential because the droplets from the cough or sneeze usually contain infectious pathogens.

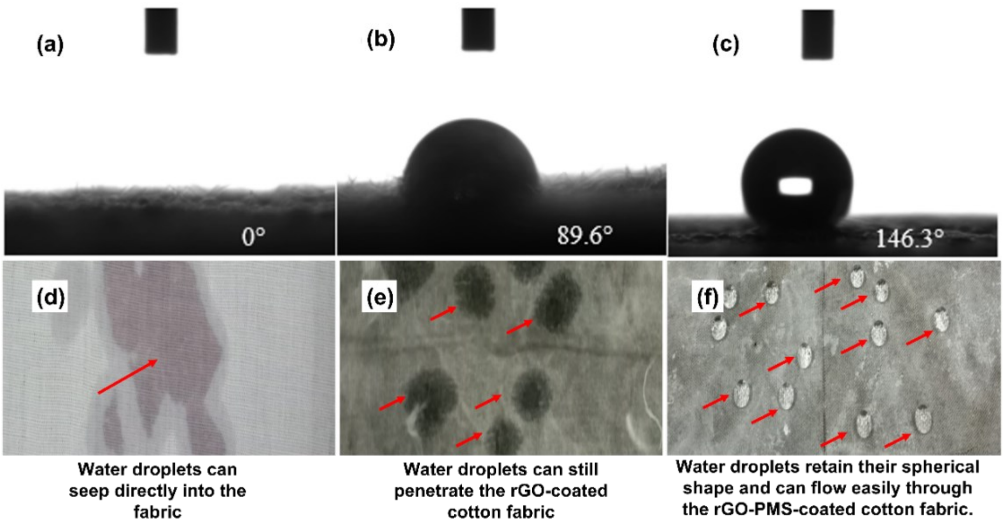

Figure 4 Contact angle measurement on (a) Cotton fabric, (b) rGO-coated cotton fabric, (c) rGO-PMS-coated cotton fabric and (d) - (f) its interaction with water droplets, respectively.

Antibacterial activity test

Cotton cloth can become a medium for bacterial growth when in contact with the skin due to its large surface area and hydrophilicity. Graphene has been shown to have a broad spectrum of antibacterial activities against pathogenic bacteria. In principle, graphene combines physical and chemical mechanisms in its antibacterial activities [32]. Physically graphene can damage the cell walls and membranes of microorganisms. Graphene can also isolate microorganism cells from their microenvironment, which can cause cell death [33].

Meanwhile, chemical interactions between graphene and microorganisms can produce reactive oxygen species that cause cells to be under oxidative stress. This interaction can cause the phenomenon of electron transfer flowing from the outer surface of the microbe so that the microbe is under oxidative stress and microbial cell death [34]. **Figure 5** shows the antibacterial activity using the bacteria *Staphylococcus aureus* ATCC 6538 with the disk diffusion method. *Staphylococcus aureus* belongs to the most critical and dangerous pathogens in Healthcare Acquired Infections, putting a substantial burden on the healthcare system [35].

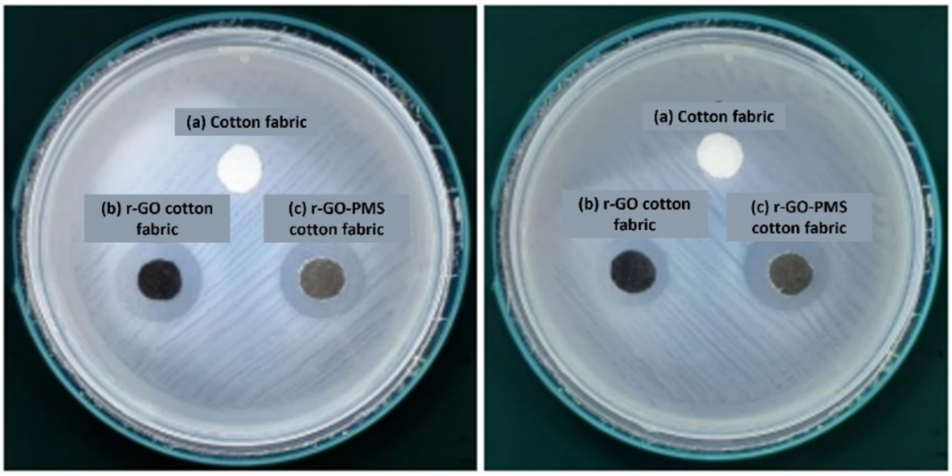

Figure 5 Fabric antibacterial activity against *Staphylococcus aureus* ATCC 6538 (a) Cotton fabric, (b) rGO-coated cotton fabric and (c) rGO-PMS-coated cotton fabric. The disk diffusion test was repeated 2 times to ensure the repeatability of the antibacterial activity of cotton fabrics.

Figure 5(a) showed no inhibition zone around the cotton fabric, which indicated the absence of antibacterial activity on the cotton fabric. In addition, **Figure 5(b)** shows the inhibition zone of rGO-coated cotton fabric with an average diameter of 16.95 mm. Meanwhile, in **Figure 5(c),** rGO-PMS-coated cotton fabric has an inhibition zone with an average diameter of 18.33 mm (**Table 1**). It proves that rGO-coated cotton fabric and rGO-PMS-coated cotton fabrics have good antibacterial activity. The addition of PMS layers also shows an increase in the inhibitory zone.

Table 1 Antibacterial activity test of the modified cotton fabric against *Staphylococcus aureus*.

Sample	Diameter of inhibition zone (mm)		Average of inhibition zone (mm)
Cotton fabric	0	0	0
r-GO-coated cotton fabric	15.50	18.40	16.95
r-GO-PMS coated cotton fabric	18.55	18.10	18.33

Photothermal property

The photothermal feature of the modified fabric was observed to determine the sterilization ability of viruses or bacteria that may remain on the surfaces of the fabric. The NIR lamp was chosen because infrared energy causes molecular vibrations that the molecular movement becomes fast and causes the internal temperature of the object that absorbs this energy to increase. In addition, reduced graphene oxide (rGO) absorbs NIR strongly, making it a good candidate for photothermal therapy [36].

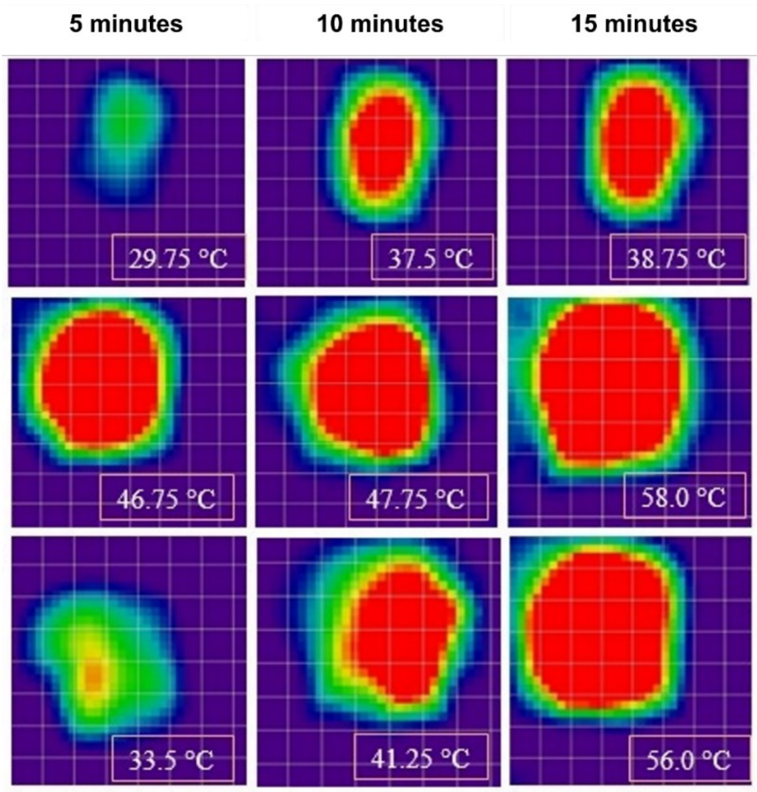

Figure 6 Photothermal result (a) Cotton fabric, (b) rGO-coated cotton fabric and (c) rGO-PMS-coated cotton fabric for 5, 10 and 15 minutes. The red color represents the hottest area, followed by the yellow, green and blue colors representing the coldest areas.

Figure 6(a) shows that the temperature of ordinary cotton fabric only produces a temperature of 38.75 °C when heated for 5 min. Meanwhile, the rGO-coated cotton fabric produces a maximum temperature of 58 °C when illuminated for 5 min, as shown in **Figure 6(b)**. This temperature increase occurs due to the presence of rGO, a material with excellent thermal conductivity [37]. According to Balandin *et al.* [38], the thermal conductivity of single-layer graphene at room temperature was reported to be 5,300 W/mK. Meanwhile, cotton fabrics have a low conductive temperature, around 0.026 - 0.065 W/mK [39]. However, when the test was carried out on rGO-PMS-coated cotton fabric in **Figure 6(c)**, the heating temperature for 5 min dropped to 56 °C. At this temperature, World Health Organization (WHO) reported that almost all SARS-CoV-2 viruses experienced a reduction to 99.99 % at 56 °C (133 °F) with consistent 15-minute exposure and temperature, which can kill other infectious substances [39]. The temperature of the fabric can also continue to increase to the maximum temperature of 77 °C. According to published research, a temperature of 70 °C or more is sufficient to inactivate the majority of viruses and bacteria [40]. Kumar *et al.* [41] also observed the photothermal activity of fabrics at similar temperatures, showing that *Escherichia coli* and *Staphylococcus aureus* were 100 % dead. Thus, this material has the potential to sterilize pathogens that may stick to the surface of the fabric.

Conclusions

FTIR and SEM investigations revealed that PMS (polymethysiloxane) and rGO (reduced graphene oxide) layers were successfully applied to cotton fabric using a simple dip-pad-dry procedure. When exposed to IR lamps, rGO has photothermal properties. This photothermal ability has the potential to sterilize microbes that may adhere to clothes. By enhancing the roughness of cotton fabric fibers, PMS coating can increase the hydrophobicity of clothes. This feature assists in self-cleaning by preventing debris and pathogen-containing liquids or droplets from permeating the cloth. The combination of these 2 substances is also antibacterially effective. rGO-PMS-coated cotton fabric with these properties can be utilized as medical textiles or personal protective equipment to prevent disease transmission. Using coconut shells as a source of rGO is environmentally friendly, inexpensive and convenient. These qualities and benefits encourage the development of low-cost personal protective equipment that has self-cleaning features and can protect health professionals and patients from hospital-acquired infections.

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