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A Green Method for the Enhancement of Antifungal Properties of Various Textiles Functionalized with Silver Nanoparticles

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Abstract: Development of textiles functionalized with nanoparticles has received growing interest. A wide range of nanoparticles can be deposited on textile fibers, which brings new properties to the final product. Although many methods for the deposition of silver nanoparticles (AgNPs) on textiles are possible, the current trend focuses on how this can be carried out in a cheaper and greener manner. Therefore, the present study aims to propose a green method for the enhancement of antifungal of textiles using AgNPs. Textile properties such as surface morphology, elemental contents, density, water absorption, and antifungal capability were comprehensively characterized. This study found that the deposition of AgNPs on the textiles can be successfully carried out using the proposed method, confirmed by the field emission scanning electron microscopy (FESEM) and energy-dispersive X-ray spectroscopy (EDX) inspections. The water absorption capability of the treated textiles was lower compared to untreated textiles. In addition, the effective antifungal capability of the treated textiles has been approved, although after 5 washing cycles.

Keywords: Silver nanoparticles; antifungal properties; functionalized textiles; green method.

1. Introduction

Silver nanoparticles (AgNPs) have earned enormous popularity in scientific literature due to their exclusive and desirable physical, chemical, and biological properties [1-7]. Several industries employed AgNPs for their final products, such as cosmetics, detergents, paint industries, and textiles. For medical and textile industries, AgNPs are used to inhibit the growth of bacteria and fungi.

Several methods have been proposed for the synthesis of AgNPs, such as by chemical, physical, and biological methods [8]. Chemical and physical methods have limitations such as the use of toxic chemicals that are potential to be released into the environment and high energy consumption, respectively. Therefore, the biological or green method seems to be a safe procedure since it provides several advantages such as simple procedures for preparation, the use of non-toxic materials, and environmentally friendly [9,10]. In green synthesis, natural reducing and stabilizing agents are used to reduce Ag^+ to Ag^0 and to prevent agglomeration, respectively [11,12].

Recently, there has been tremendous research interest in the functionalization of textiles using AgNPs with the aim to produce fabrics with diverse practical performance. Due to their remarkable surface plasmon resonance feature, AgNPs have also been applied as novel and new generation colorants for various textile products. Numerous procedures for the deposition of AgNPs on textiles have been proposed and still continuously revised. They include solution-immersion, layer-by-layer deposition, and sonochemical procedures. For instance, AgNPs were formed on the cotton fabric surface using the solution-immersion by pre-activation by potassium hydroxide and in situ reductions of silver nitrate ($AgNO_3$) [13]. The treated cotton showed high antibacterial activity against Gram-positive *Staphylococcus aureus* and Gram-negative *Escherichia coli*. In addition, nylon and silk fibers were employed to fabricate antimicrobial textiles enhanced using AgNPs prepared by the layer-by-layer deposition method [14]. The study successfully achieved 80% and 50% bacteria (*Staphylococcus aureus*) reduction for the silk and the nylon fibers, respectively. Moreover, the sonochemical coating of AgNPs on various textile fabrics, namely, nylon, polyester, and cotton, can also be an alternative method [15]. The successful method exhibited excellent antibacterial activity against *Staphylococcus aureus* and *Escherichia coli*, depending on the textiles type.

It is noted that the solution-immersion method provides a simple procedure for the deposition of AgNPs on textiles compared to others. In addition, the solution-immersion method can potentially diminish the use of high energy consumption, which is difficult to achieve when the layer-by-layer deposition and sonochemical are used. Currently, a green method based on plant biomolecule-mediated in-situ synthesis seems a viable option for the deposition of AgNPs onto textile surfaces. In this method, biomolecules present in plants were used as natural reducing agents. It has been well known that the different plant compounds such as tannins, flavonoids, anthraquinonoids, terpenoids, and chalcones are have been widely used as reducing and stabilizing agents for the green synthesis of AgNPs with several beneficial properties for textiles.

In this contribution, the aim of the present study was to propose a simple and reliable green method based on plant biomolecule-mediated in situ synthesis to fabricate antifungal textiles enhanced by AgNPs. AgNPs were produced on various textiles fibers by in situ reductions of $AgNO_3$ with the *Ageratum conyzoides* extract. Next, the treated textiles were tested for the potential application as an antifungal textile. *Ageratum conyzoides* is categorized as a weed and is commonly found in Indonesia and Malaysia. It basically contains flavonoids and terpenoids that can be potential as the natural reducing agents to reduce Ag^+ to Ag^0 [16].

2. Materials and Methods

2.1. Materials.

Ageratum conyzoides leaves were collected from the surrounding area of Universiti Teknologi Malaysia, Johor, Malaysia. Whatman filter paper (Whatman, Sigma-Aldrich, USA) was used as a filter for preparing pure leave extracts. Ultrapure water was used for this process. *Aspergillus* sp. was used as a fungus model obtained from the Faculty of Sciences, Universiti Teknologi Malaysia. $AgNO_3$ (QReC, Auckland, New Zealand) was used as a silver source. A stock solution of $AgNO_3$ 0.1 M was prepared for this study. Four types of textiles, which are cotton (C), poly-cotton (PC), fiber (F), and silk (S) were used in this study and were obtained from a local shop in Johor, Malaysia.

2.2. Preparation of Ageratum conyzoides extracts.

In this study, fresh leaves (10 g) were used. To remove any impurities, the fresh leaves were washed three times, each using the tap water and ultrapure water. Next, the cleaned leaves were mixed with the 200 mL ultrapure water in a beaker glass. The mixture was boiled up to 80 ^{o}C for 40 min before cooling at room temperature. To obtain a pure leaves extract, the mixture was filtered using the filter paper. The pure extracts were then stored at a fridge at a temperature of 7 ^{o}C before further use.

2.3. Preparation of textiles.

The textiles were cut in a circle shape, having a diameter of 8 mm. The textiles were rinsed using tap water and ultrapure water three times each. It was then followed by boiling the cleaned textiles for 30 min for the complete removal of any impurities. After boiling, the textiles were dried in the Memmert oven at 50 ^{o}C for 10 min. To attach AgNPs on textiles, the single textile was soaked in 5 mL solution of 0.1 M $AgNO_3$ for 30 min in a beaker glass with stirring at a speed of 100 rpm. Then, 5 mL of *Ageratum conyzoides* extracts were poured carefully into the mixture. The mixture was then heated at 50 ^{o}C for 30 min. To dry the treated textile, the textile was dried in an oven at 50 ^{o}C for 15 min. The same procedure was carried out for all employed textiles. The four types of antifungal textiles are SA (silk-AgNPs), CA (cotton-AgNPs), FA (fiber-AgNPs), and PCA (polycotton-AgNPs).

2.4. Surface morphology and elemental composition.

The surface morphology of the treated textiles was studied using the field emission scanning electron microscopy (FESEM, Zeiss Supra 35VP). The equipment was operated at 5 kV acceleration voltage with a magnification of ×50,000. The treated textiles were also characterized using scanning electron microscopy (SEM) with an energy-dispersive X-ray spectroscopy attachment (SEM-EDX, Hitachi S-34000N) to determine their elemental composition. It was operated at a voltage of 15 kV and equipped with the Bruker Quantax software.

2.5. Density test and water absorption investigation.

For the density test, the diameter of the textiles was measured. The mass of dry textiles before attaching with AgNPs was weighed. Next, the treated textiles were also weighed. The density was estimated by dividing the mass weight with the area of the textiles. The density is presented in the unit of g/m^2. For water absorption investigation, it was estimated from the dry and wet mass of the textiles. For this test, all treated and untreated textiles were used. The treated textiles were first soaked in the ultrapure water for 30 min. Water absorption characteristics for all treated textiles were estimated using the following equation [17]:

$$WA = \frac{W_w - W_d}{W_d} \times 100\% \quad (1)$$

where WA is the water absorption (%), W_d is the initial weight of the textiles before absorption (g), and W_w is the weight of the textiles after absorption (g).

2.6. Antifungal examination and washing durability test.

In the present study, Aspergillus sp. was used for antifungal examination. In the preparation, *Aspergillus* sp. was placed on the center of the agar surface. The petri dish containing the fungal was incubated for 24 h to assess the inhibition zone. As a comparison, the inhibition zone of the textiles without the AgNPs treatment was also measured as a control. For the investigation of washing durability, the treated textiles were washed for 1, 3, and 5 cycles rpm for 15 min. After the washing treatment, the inhibition zone was measured. In this investigation, the zone of inhibition is presented in the unit of mm.

3. Results and Discussion

3.1 Surface morphology.

The surface morphology of the textiles before attaching with AgNPs is shown in Figure 1. It is noted that that the surface of the untreated demonstrated smooth longitudinal fibril structure of the fibers without any contaminating particles on the textile surfaces. The deposition of AgNPs on textiles changes the surface morphology of textiles, as shown in Figure 2. It is established that the properties of AgNPs on textiles are influenced by reducing agents, stabilizers, and textiles types.

In general, the aforementioned observation indicated that AgNPs could be successfully deposited on the textiles. Rough fiber surface can be observed after binding with AgNPs because the deposition of AgNPs may make a modification of the surface morphology of the textile fabrics. The formation of particles and agglomerates in different sizes can be observed because every fabric surface has a unique interaction between AgNPs and textile fiber [18].

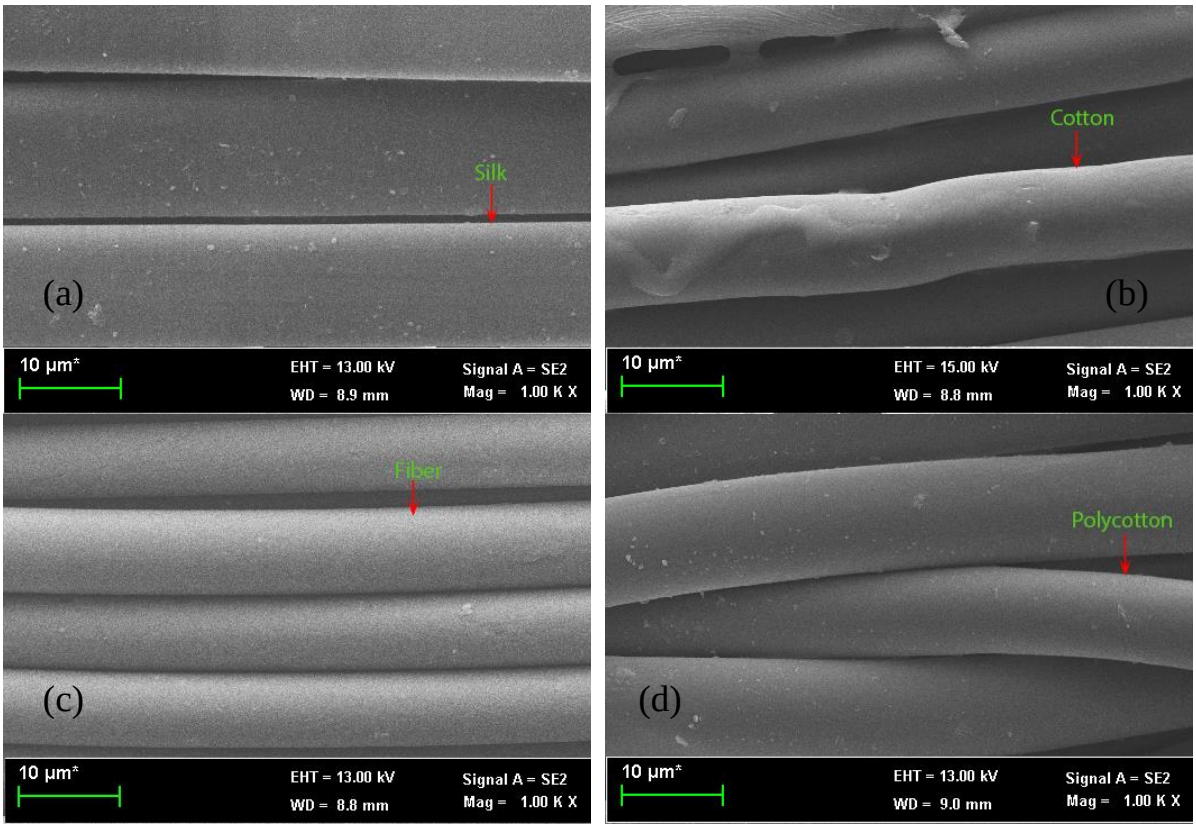

Figure 1. FESEM images for (a) silk, (b) cotton, (c) fiber, and (d) poly-cotton.

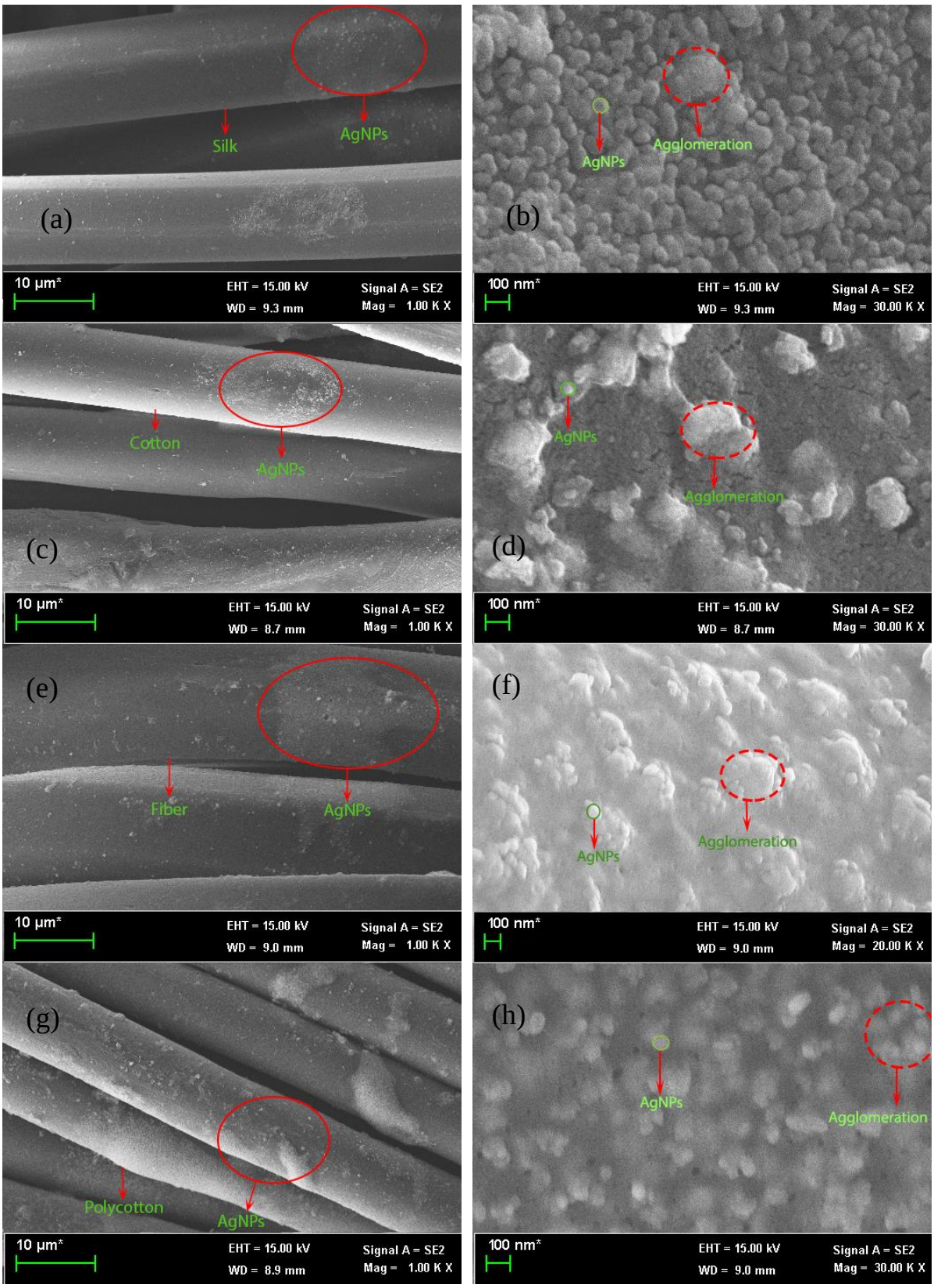

Figure 2. FESEM images of (a-b) SA, (c-d) CA, (e-f) FA, and (g-h) PCA

3.2. EDX characterization.

EDX examination was used to determine the elemental composition of textiles before and after attaching with AgNPs. Figure 3 shows the EDX spectra of treated textiles. AgNPs attached to textiles is possibly caused by physical bonding between AgNPs and the textile surface. The physical bonding indicates that AgNPs have temporarily bonding on the textile

surface. There are some elements detected on the treated textiles, as listed in Table 1. The elements are carbon (C), oxygen (O), nitrogen (N), and silver (Ag). It is noted that C and O elements detected possibly came from textile elements. In addition, the N element detected possibly came from *Ageratum conyzoides* extracts.

EDX spectra showed the highest peak, approximately 3 keV for all treated textiles, as shown in Figure 3. Similar results were obtained from previous studies with the highest peak observed at 3 keV when AgNPs were synthesized using Ocimum sanctum, Hydrangea paniculate, and Tamarind fruit [19-21]. The peak of approximately 3 keV is attributed as the characteristic of the silver atom, which has an energy level of around 3 keV.

Table 1. Elements analysis of all untreated and treated textiles

Textiles	Elements	Before treated (%)	After treated (%)
S	C	71.4	68.2
	O	28.6	18.3
	N	-	11.3
	Ag	-	2.3
C	C	72.4	65.9
	O	27.6	20.5
	N	-	11.7
	Ag	-	1.8
F	C	72.6	45.2
	O	27.4	20.9
	N	-	17.1
	Ag	-	16.9
PC	C	73.8	60.5
	O	26.2	19.3
	N	-	13.4
	Ag	-	6.8

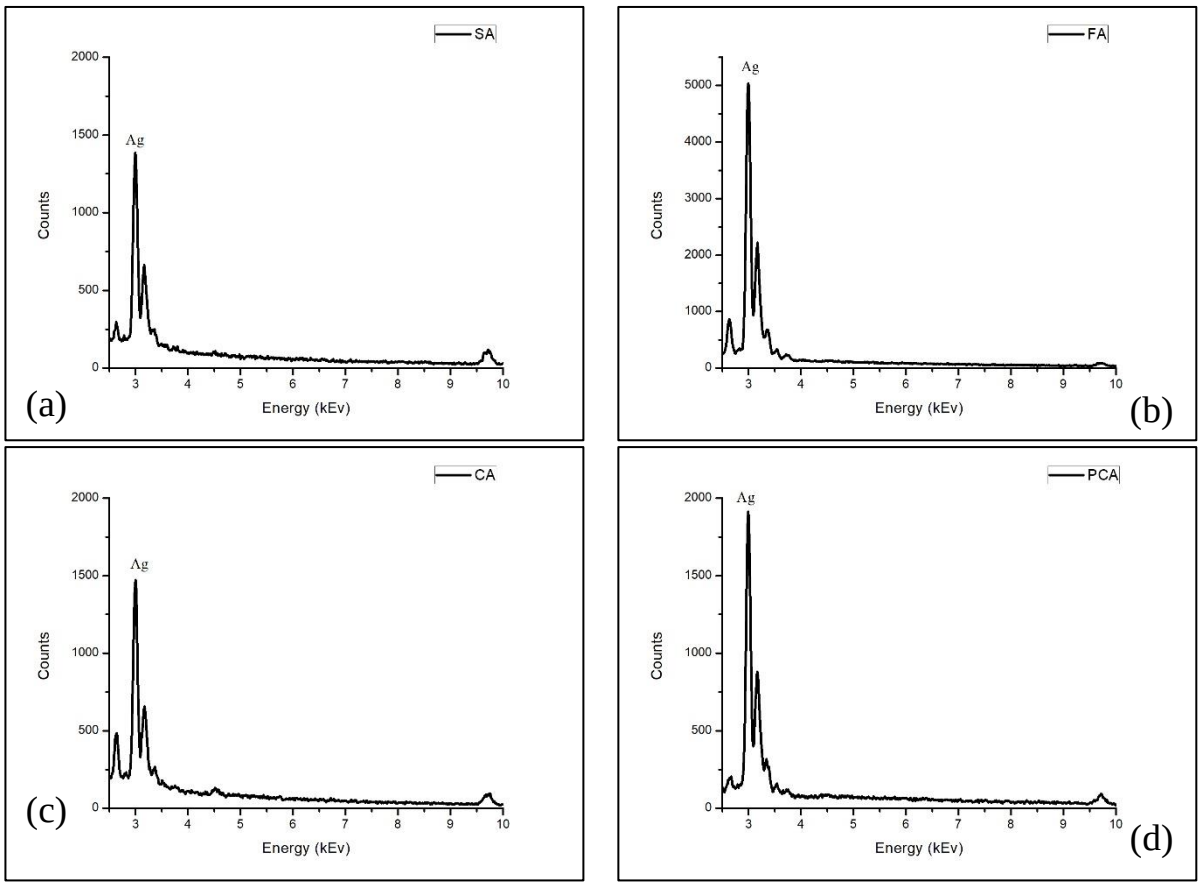

Figure 3. EDX spectra of textiles of (a) SA, (b) CA, (c) FA, and (d) PCA

3.3. Density and water absorption.

Table 2 list the density of untreated and treated textiles. Before treating with AgNPs, cotton, poly-cotton, fiber, and silk had densities by 278.41 g/m^2, 119.32 g/m^2, 258.52 g/m^2, and 99.43 g/m^2, respectively. After treating with AgNPs, the corresponding values increase up to 556.81 g/m^2, 139.20 g/m^2, 357.95 g/m^2, and 119.32 g/m^2. It is noted that the deposition of AgNPs on cotton, poly-cotton, fiber, and silk can increase their density by 2.0, 1.2, 1.4, and 1.2 times higher compared to untreated textiles.

Table 2. Density of all untreated and treated textiles

Textiles	Density (g/m^2)
S	99.43
C	278.41
F	258.52
PC	119.32
SA	119.32
CA	556.81
FA	357.95
PCA	139.20

Generally, textile density values increase after treating with AgNPs. An increase in density is due to an increase in textile mass after attaching with AgNPs. Table 2 indicates that cotton performed the highest increase in density compared to others. This shows that the mass of cotton increased significantly. An increase in the mass of the cotton indicated that AgNPs could be well attached to cotton. The water absorption test determines the ability of textiles to absorb the water. Table 3 lists the water absorption of untreated and treated textiles. Textiles are made from cellulose, which is easy to absorb water. The textiles have many -OH groups that can bind to water through hydrogen bonds [22]. In general, the percentage of water absorption decreases after the presence of AgNPs.

Table 3. Water absorption of all untreated and treated textiles

Textiles	Water Absorption (%)
S	92.31
C	69.57
F	71.74
PC	68.42
SA	89.47
CA	37.78
FA	41.94
PCA	56.25

Findings of the present work are in agreement with the previous study, which observed that the treated textiles performed better water resistance compared to the untreated textiles [23]. After dropping water to the treated and untreated textiles, the bulb of water can be formed on their treated textiles surface. In contrast, the water can be perfectly absorbed in untreated textiles. A similar result was also obtained by the previous work, which examined the water absorption of polyester and viscose [17]. Their study reported that the deposition of AgNPs on the textiles could form a barrier between textile fibers and water, which can affect the water absorption characteristics. It is well known that the textile's fiber has a large surface area enabling it to maintain its moisture. Therefore, the deposition of AgNPs on the textile surface can reduce their moisture properties.

3.4. Antifungal investigation.

Table 4 presents the zone of inhibition of the treated textiles against *Aspergilus* sp. It was found that the treated cotton had a zone of inhibition by 14.67 ± 3.05 mm. In addition, silk, fiber, and poly-cotton performed zone of inhibition by 12.00 ± 1.00 mm, 15.33 ± 6.11 mm, and 15.00 ± 7.81 mm, respectively. In general, the highest zone of inhibition was performed by the treated fiber and followed by poly-cotton, cotton, and silk. The findings of the present study are in line with those of previous studies. AgNPs had a zone of inhibitions by 16.00 ± 2.00 mm, 20.60 ± 1.52 mm, 19.30 ± 1.52 mm, 11.0 ± 1.60 mm when they were tested against *Aspergillus niger*, *Aspergillus fumigatus*, and *Aspergillus flavus*, respectively [24,25]. In addition, photochemical deposition of AgNPs conventional cotton gauzes performed antifungal properties against *Candida albicans* by up to 5 mm [26]. By using a simple pad-dry-cure method, cotton fabrics treated with AgNPs also has antifungal properties against different fungal strains *Aspergillus fumigatus*, *Aspergillus niger*, *Penicillium* sp., and *Rhizoctonia oryzae* [27].

Table 4. zone of inhibition of the treated textiles against *Aspergilus* sp

Textiles	Inhibition zone (mm)
SA	12.00 ± 1.00
CA	14.67 ± 3.05
FA	15.33 ± 6.11
PCA	15.00 ± 7.81

It is well established that the antifungal capability of AgNPs depends on their particle properties, procedures, and types of fungi [24,25,28-30]. The current study found that the treated textiles have antifungal properties. These results are likely related to antifungal mechanisms such as by (a) enzyme inactivation, (b) damage DNA replication, (c) disruption of the cell wall, (d) disruption of the cytoplasmic membrane, and (e) preventing the fungal to growth [31,32].

3.5. Antifungal durability.

Table 5 presents the inhibition zones of treated textiles after washing the durability test. After washing, the inhibitory zone of the treated textiles decreased. The slight decrease in inhibition zone value is possibly caused by the loss of AgNPs present in textiles after washing treatment. This decrease in inhibition zone indicates that the washing causes reduced antifungal activity on textiles. A previous study showed that antimicrobial reduction was smaller after washing [33-35]. The loss of AgNPS in textiles indicates that there is a physical bonding between AgNPs and textiles. The physical bonding is possibly caused by van der Wals force [36]. The van der Waals bonding has a weak bonding force. The weak bonding force possibility allows AgNPs to be easily released after washing. In general, findings from this study exhibited that the AgNPs showed antimicrobial properties, which supported previous findings reported elsewhere [37-41].

Table 5. Inhibition zones of all treated textiles

Textiles	Inhibition zone (mm)			
	Washing cycles			
	Without washing	1 time	3 times	5 times
SA	12.00	11.00	10.00	9.00
CA	15.00	12.00	11.00	10.00
FA	15.33	12.00	11.00	10.00
PCA	15.00	12.00	11.00	10.00

4. Conclusions

The aim of this study was to propose a green method for the enhancement of antifungal of textiles using AgNPs. This study found that AgNPs can be successfully deposited on the various textiles. Generally, the current study established that the treated textiles with AgNPs had antifungal ability depending on textile types. The surface bonding AgNPs on textile was possibly caused by van der Waals force. Moreover, all treated textiles demonstrated antifungal activity, although after 5 washing cycles.

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Conflicts of Interest

The authors declare no conflict of interest

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