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A green deposition method of silver nanoparticles on textiles and their antifungal activity

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ABSTRACT

This study aims to propose a new green method for the deposition of silver nanoparticles (AgNPs) on textiles without the use of chemical compounds as binders. The deposition of AgNPs on textiles was achieved by immersing textiles in silver nitrate solution before adding with a natural reducing agent obtained from the extraction of *Mikania micrantha*. Plasmonic properties of the synthesized AgNPs were characterized using Ultraviolet-visible (UV–vis) spectroscopy and surface morphology of textiles was identified using the field-emission scanning electron microscopy (FESEM). In addition, energy-dispersive X-ray spectroscopy was also employed for the characterization. Inhibition zone measurement was performed for evaluating the antifungal capability of textiles attached with AgNPs. This study showed that the attachment of AgNPs to several textile types (cotton, cotton-polyester, silk, and fiber) without the use of binders or other chemical compounds had been successfully achieved. Moreover, all textiles attached with AgNP exhibited effective antifungal activity.

Keywords: *Silver nanoparticles; textiles; green method; antifungal materials.*

1. INTRODUCTION

The development of nanoparticle for various applications, especially for silver nanoparticles (AgNPs) has been increasing in recent years. AgNPs are a class of materials with sizes ranging from 1 to 100 nm. AgNPs are currently widely studied due to their attractive specific and genuine chemical, biological, and physical properties [1-2]. Several studies have been conducted to investigate their properties and potential applications as antimicrobial agent, antifungal agent, water treatment, semiconductor devices, and biomedical devices [3-6].

It is noted that AgNPs can be synthesized using chemical, physical, and biological methods [7]. However, chemical and physical processes are not promising because of their potential as pollutant sources to the environmental and high energy consumption, respectively. Therefore, research concerning the topic of AgNPs synthesis by employing biological methods (green synthesis) has been intensively developed [2]. In nanomaterial synthesis, it requires three essential ingredients, namely precursors, reducing agents, and stabilizing agents. In the green synthesis of AgNPs, silver nitrate ($AgNO_3$) is commonly used as a silver source while bacteria, fungi, algae, plants, and yeast can be used as natural reducing agents as well as stabilizing agents. $AgNO_3$ can be present as Ag^+ and NO_3^- when dissolving in water. The reducing agent has a role in converting Ag^+ ions to Ag^0 particles. In addition, the stabilizing agent is employed to stabilize the produced AgNPs to avoid agglomeration [8]. This method is considered as low cost and simple because reducing and stabilizing agents can be obtained freely, and the synthesis can be carried out quickly at room temperature [2].

Today, many researchers have interested in developing antimicrobial and antifungal textiles functionalized with AgNPs. Various textiles such as cotton, wool, polyester, silk, cotton/polyester blend, regenerated cellulose, and polyamide have been utilized for the development. One of the challenges in the fabrication of AgNPs-coated textiles is the deposition method. The previous study reported that the deposition of AgNPs on textiles was controlled by electrostatic interactions between AgNPs and textiles constituent. There are three proposed interactions between AgNPs and textiles, such as (a) the absorption of silver ions having a positive charge on textiles having a negative charge, (b) diffusion of AgNPs into textile fibers, and (c) interaction of AgNPs with textile fibers. For instance, the interaction between AgNPs and sulfur atoms present on wool fibers has been verified [9]. In addition, AgNPs were deposited on the fabric due to the electrostatic interaction between AgNPs and vat dyed cotton fabrics [10].

In the deposition process, chemicals are often used to increase the stabilization of AgNPs on textiles by employing binders, cross-linkable polymers, and crosslinking agents [11-12]. The chemicals mentioned above have been mostly used to suspend AgNPs in an aqueous phase [3, 9, 13-17]. However, the use of chemicals can threaten the health of the skin and the environment due to their toxic characteristics [18]. In the application of AgNPs as wound healing, physically or chemically induced cutaneous wounds may significantly disturb skin structural and functional integrity at different stages, leading to permanent disability or even death, depending on the severity of the injury [19]. It indicates a need to propose a new green deposition method of AgNPs on textiles without the use of binders or other chemical compounds for the stabilization.

Aligning the aforementioned research necessity, the present study proposed and demonstrated a new green method for the deposition of AgNPs on textiles without the use of chemicals as binders, cross-linkable polymers, and crosslinking agents. AgNPs were synthesized using *Mikania micrantha* extract that contains phenolic compounds [20]. Several textile types, which are cotton, cotton-polyester, silk, and fiber were used in this work as textile models. Moreover, the antifungal activity of the textiles enhanced using AgNPs against *Aspergillus* sp was also investigated.

2. MATERIALS AND METHODS

2.1. Materials.

Mikania micrantha was obtained from the surrounding area of the Universiti Teknologi Malaysia (UTM), Johor, Malaysia. $AgNO_3$ and malt extract agar were purchased from the Sigma-Aldrich. *Aspergillus* sp., was provided by the Department of Biosciences, UTM. The employed textiles for this study are cotton, cotton-polyester, silk, and fiber obtained from a local textile shop in Johor Bahru.

2.2. Extraction of *Mikania micrantha.*

In the preparation, 10 g of *Mikania micrantha* leaves were washed using tap water three times and then by the ultrapure water three times. Subsequently, the glass beaker containing the washed leaves was added with the 200 mL of the ultrapure water and then boiled on a hot plate for 30 mins. Furthermore, it was cooled at room temperature and then filtered using 0.45 µm of filter paper to obtain pure extracts. The obtained leaf extract was stored at a temperature of 7 ^{o}C for further use.

2.3. Preparation of textiles.

The textiles were cut in a circle shape with a diameter of 8 mm and then boiled with 25 mL of the ultrapure water in a glass beaker at a temperature of 80 ^{o}C for 30 mins to remove impurities. Subsequently, it was dried at 40 ^{o}C for 10 min before further use.

2.4. Deposition of AgNPs on textiles.

The cleaned cotton was firstly immersed into 0.1 M $AgNO_3$ solution (5 mL) and stirred at a speed of 100 rpm for 30 min. Furthermore, 5 mL of the leaf extract was slowly added into the mixture (the cleaned textiles and $AgNO_3$ solution) and continuously stirred at a speed of 100 rpm for 30 min with the temperature maintained at 50 ^{o}C. The deposition process of AgNPs on textiles was initiated since the leaf extract was added into the mixture. The leaf extract acts as a reducing agent in converting Ag+ ions present on textiles to Ag^0 particles. After completing the process, the treated textiles were taken and dried at 40 ^{o}C for 15 min. The same procedure was applied for all textiles and they abbreviated as AgNPs-coated cotton-polyester (PAg), AgNPs-coated silk (SAg), and AgNPs-coated fiber (FAg).

2.5. Preparation of agar plates.

In this work, 27.6 g of Malt extract agar (Merck) was dissolved using 575 mL ultrapure water in a 1 L glass bottle. Furthermore, a glass bottle containing Malt extract agar solution was sterilized using an autoclave (Hiclave HVE-50 HIRAYAMA). The sterilized agar was poured into Petri dishes carried out in a laminar airflow cabinet and then cooled to room temperature. All dishes were then sealed with parafilm tape and then stored at 7 ^{o}C for further use.

2.6. Antifungal investigation.

Three pieces of AgNPs-coated cotton (with a diameter of 8 mm) were placed onto the agar plate with the triangle position. Subsequently, a small portion of the *Aspergillus* sp. was placed onto the middle of the surface of the agar plate containing three pieces of AgNPs-coated cotton and then sealed with parafilm tape as a treatment sample. The agar plate containing three pieces of cotton (without treatment with AgNPs) with a small portion of the *Aspergillus* sp. was used as a control sample. The same procedure was applied to all AgNPs-coated textiles.

2.7. Plasmonic investigation.

The plasmonic properties of AgNPs were analyzed using UV–vis spectrometer (Perkin–Elmer, No. 101N4110104). In the preparation, 2 mL of AgNP solution was observed in UV–vis spectrometer in the wavelength ranging from 350-700 nm with a resolution of 1 nm and a scan speed of 960 nm/min. All measured data were processed and analyzed using the Lambda 25 software.

2.8. Field-emission scanning electron microscopy and energy-dispersive X-ray spectroscopy characterization.

Morphology of the synthesized AgNPs-coated textiles was analyzed and characterized using a field-emission scanning electron microscopy (FESEM, Zeiss Supra 35VP). It was operated at 5 kV acceleration voltage with a magnification of ×50,000. The composition of the produced nanoparticles from synthesized AgNPs-coated textiles was also characterized using energy-dispersive X-ray spectroscopy (EDX, Hitachi S-34000N) at a voltage operating of 15 kV and equipped with the Bruker Quantax software.

3. RESULTS

3.1. UV–vis characteristics.

In this study, *M. micrantha* extract mixed with $AgNO_3$ showed a change in color from light green to dark grey (Figure 1) due to the excitation of surface plasmon resonance (SPR) [21]. Figure 2 shows the UV–vis spectra of the synthesized AgNPs using *M. micrantha* extract. It can be analyzed that the AgNPs synthesized using *M. micrantha* extract leaves have an absorbance peak at ~374 nm. The plasmonic properties of AgNPs are typically affected by their size and the surrounding media [22]. The results obtained of this study are similar to the previous studies that also investigated the UV–vis spectra of AgNPs in the range 350-400 nm [23]. Several studies have confirmed the correlation between the UV–vis spectrum and properties of AgNPs [24-27]. A single peak in the UV–vis spectrum can be correlated with uniform spherical AgNPs [28-29].

3.2. Stability of AgNPs.

The synthesized AgNPs were also evaluated under UV–visible spectroscopy with variations reaction time duration (1–4 days) in order to analyze the stability of the formed nanoparticles. The samples were stored in plastic tubes under ambient temperature in the absence of light, and UV/vis spectra were recorded regularly at one-day intervals until four days. It can be

seen that with increased reaction time, the absorbance decreased while the peak (λ_{max}) was constant approximately at 374 nm from 1 until 4 days (Table 1).

The estimation of production of AgNPs during synthesis can also be analyzed using UV–vis spectra because the absorbance is the amount of light absorbed by the particles. A decrease in absorbance indicates a lower production of AgNPs. The results showed that there is no alteration in the peak, thus indicated the higher stability of biosynthesized AgNPs using *M. micrantha* extract [30]. This result was similar with obtained by [31], who reported a decrease in absorbance indicating aggregated nanoparticles.

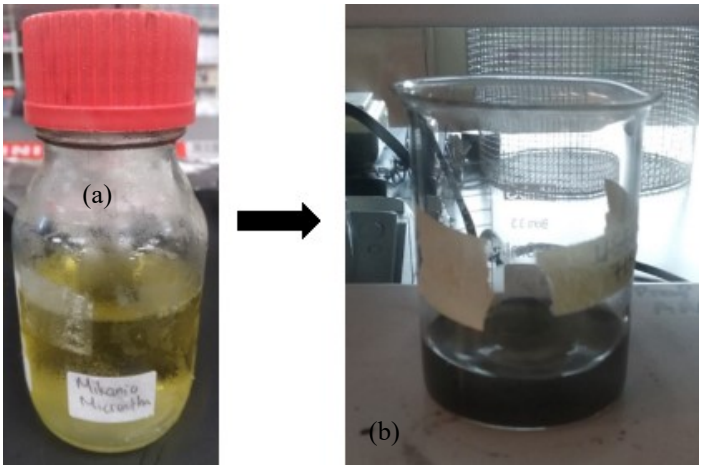

Figure 1. Visual inspection of (a) *M. Micrantha* and (b) the synthesized AgNPs

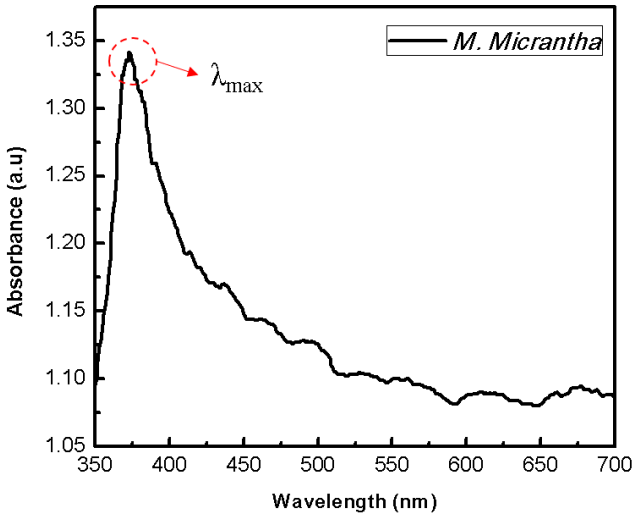

Figure 2. UV–vis spectrum of AgNPs synthesized using *M. Micrantha.* extracts

Table 1. Stability of AgNPs

No.	Duration (Day)	Absorbance (a.u)	λ_{max} (nm)
1	1	1.342	374
2	2	1.109	374
3	3	0.850	374
4	4	0.499	374

3.3. Surface Morphology.

The FESEM images of the treated textiles are shown in Figure 3. AgNPs synthesized using the extract of *M. micrantha* were successfully attached to textiles. The results showed that they have various shapes and sizes range depending on the type of textiles. It can be analyzed that both PAg and FAg have spherical nanoparticles with sizes of 83.6 ±18.22 nm and 60.7 ±16.55 nm, respectively. In addition, SAg has a mixture of nanoparticle shapes, namely spherical and rod with a size of 51.25 ±14.43 nm. AgNPs with rod shapes were also observed for AgNPs-coated cotton (CAg) with particle size 35.81 ±10.42 nm. PAg has the widest and evenly distributed particle distribution range (60-110 nm) compared to others (SAg: 30-70 nm, FAg: 40-70 nm, and CAg: 25-40 nm).

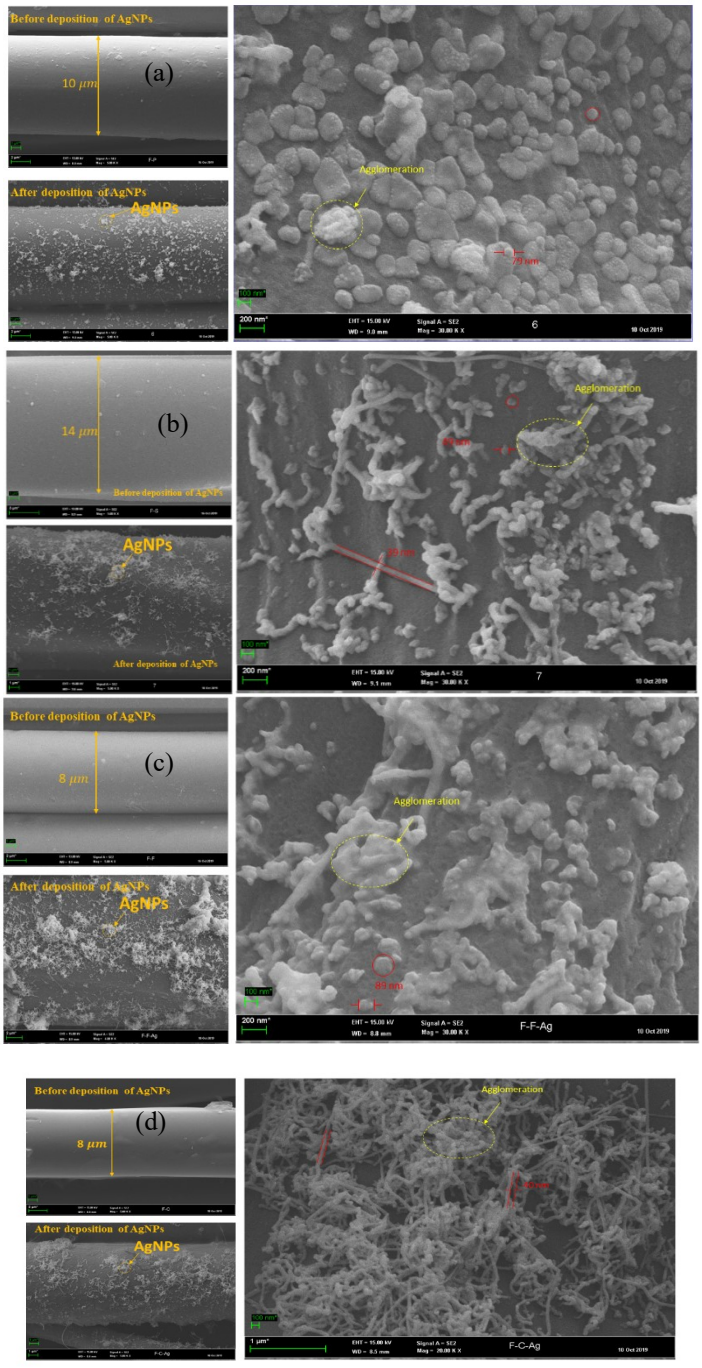

Figure 3. FESEM images of (a) PAg (b) SAg (c) FAg, (d) CAg

Several studies have reported that AgNPs in the range 10–100 nm can be attached to textiles [37-39]. For instance, AgNPs of ~50 nm could be produced using pomegranate peel extract and attached to cotton fabric using sodium hydroxide (NaOH) [33]. In addition, the spherical shape of AgNPs with a size ranging from 12 to 16 nm could be produced as listed in Table 2. AgNPs were synthesized using sugar cane and successfully

deposited in cotton fabric using a binder of butane tetracarboxylic acid (BTCA) and sodium hypophosphite (SHP) [34]. As a comparison, the present study obtained that AgNPs can be deposited on textiles without the addition of binders. For a comprehensive overview, Table 2 summarizes methods for the deposition of AgNPs on textiles without or with chemical compounds as binders.

Table 2. Existing methods for the deposition of AgNPs on textiles

Size(nm)	Textiles	Deposition method	Ref
~50	cotton	The synthesized AgNPs were added and maintained on textiles in an acid condition.	[33]
12 to 16	cotton	Deposition of AgNPs on cotton fabric was carried out using a binder, butane tetracarboxylic acid (BTCA) and sodium hypophosphite (SHP).	[34]
N/A	cotton	In the proposed method, cetyl trimethyl ammonium bromide (CTAB) was added as a binder.	[35]
~50	cotton	Cotton fabric was padded with AgNPs solutions after treatment with warm water	[36]
~35	cotton	Textiles were dipped into $AgNO_3$ solution before adding with leaf extract combined with stirring and heating treatment.	This study

3.4. EDX of the treated textiles.

EDX analysis was conducted to confirm the presence of AgNPs on the textiles. Figure 4 shows the EDX spectra of the synthesized AgNPs-coated textiles. The presence of AgNPs on textiles was proven by peak at approximately 3 eV, which is typical for absorption of metallic AgNPs due to their SPR properties [40]. The difference of intensity from samples can be correlated to the concentration of the element in the sample [41].

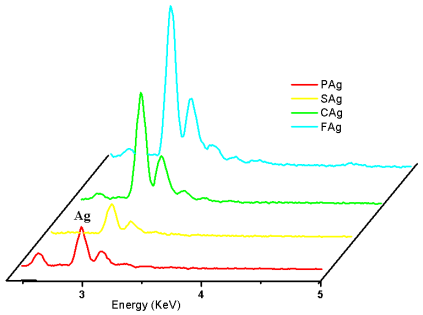

Figure 4. EDX spectra of all treated textiles

Table 3. Elemental analysis of textiles treated with AgNPs

Samples	Element	Mass (%)
PAg	**Silver**	21.0
	Carbon	48.9
	Oxygen	19.3
	Nitrogen	10.8
SAg	**Silver**	9.7
	Carbon	59.1
	Oxygen	20.3
	Nitrogen	10.9
FAg	**Silver**	14.7
	Carbon	52.8
	Oxygen	22.1
	Nitrogen	10.4
CAg	**Silver**	36.4
	Carbon	42.7
	Oxygen	14.0
	Nitrogen	6.9

The present study found that the silver atom contents of 21%, 9.7%, 14.7%, and 36.4% can be observed for PAg, SAg, FAg, and CAg, respectively, as listed in Table 3. It was indicated that cotton was the most effective fabric to hold AgNPs compared to others. Other elements detected in the EDX characterization possibly came from the basic textile elements. In addition, EDX characteristics also depend highly on the synthesis procedures, reducing agents, and stabilizing agents [42].

3.6. Antifungal investigation.

All AgNPs-coated textiles were tested against *Aspergillus sp.,* and the results are presented in Table 4. It can be observed that AgNPs have the antifungal ability with the formation of the inhibition zone. Zone of inhibition of the treated textiles was 11.66±2.08 mm, 13.33±3.51 mm, 13.33±3.05 mm, and 14.33±3.51 mm for PAg, SAg, FAg, and Cag, respectively. In this study, the most effective AgNPs-coated textile as antifungal against *Aspergillus* sp. was CAg. This result was also confirmed by the previous study [43], where the highest antifungal activity of treated textiles was observed when AgNPs were coated on cotton against *Aspergillus* sp. due to the effective distribution of AgNPs in surface area. High antifungal activity of AgNPs-coated cotton fabric was also reported in another study [44], which found that AgNPs-coated cotton fabric has high stability due to the release of silver ion. In general, all synthesized AgNPs-coated textiles show antifungal ability against *Aspergillus* sp.

The finding has also been confirmed from several studies. For instance, the synthesized AgNPs by green tea and black tea leaves extracts inhibited approximately 80% of *Aspergillus sp.* [45] Moreover, inhibition zones of 12 mm were found when AgNPs were synthesized using *Aspergillus* niger filtrate [46]. As a comparison, Table 4 lists the inhibition zone of AgNPs against *Aspergillus sp*. It is well established that the antifungal capability of AgNPs depends highly on the size, shape, distribution of particle, species, and concentration [47].

Table 4. Antifungal activity of AgNPs against *Aspergillus* sp.

Extract	Textiles	Inhibition zone (mm)	Ref
Green tea leaves	N/A	±30	[45]
Black tea leaves	N/A	±30	
Aspergillus niger	cotton	12	[46]
H. muciformis	N/A	20	[47]
M. micrantha	Polyester-Cotton	11.6±2.08	This study
	Silk	13.3±3.51	
	Fiber	13.33±3.05	
	Cotton	14.33±3.51	

4. CONCLUSIONS

The deposition of AgNPs on textiles has been succesfully prepared by a green method. All AgNPs-coated textiles demonstrated antifungal ability against *Aspergillus* sp. AgNPs coated cotton (CAg) has the highest inhibition zone against

Aspergillus sp. due to the effective distribution of AgNPs in surface area. FESEM and EDX analysis confirmed the successful deposition of AgNPs on textiles without using chemical compounds as binders. These results are beneficial for the development of textiles functionalized with AgNPs for medical purposes in the future.

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