Investigation of antibacterial activity of cotton fabric incorporating nano silver colloid

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Abstract. In this work, silver nanoparticles were prepared by polyol process with microwave heating and incorporated on cotton fabric surfaces. The antibacterial performance of the antibacterial cotton fabric was tested for different concentration of nano-sized silver colloid, contact time germs, and washing times. It was found that antibacterial activity increased with the increasing concentration of nano-sized silver colloid. The antibacterial fabric with 758 mg/kg of silver nanoparticles on surface cotton was highly effective in killing test bacteria and had excellent water resisting property.

Keywords: Silver nanoparticles, antibacterial activity, cotton fabric, microwave radiation.

1. Introduction
The application of nanoparticles to textile materials has attracted considerable interest due to their novel physicochemical properties and their potential applications. However, some of these particles are toxic or poorly effective, which makes them not suitable for applications in medicine, filters, and textiles and for the exclusion of pollution. For example, nano-TiO$_2$ for self-cleaning properties and applying in hygienic textiles [1-3] and ZnO nanoparticles for UV-blocking, antibacterial properties and applying in medical clothes, protective [4, 5]. For a long time, silver nanoparticles are non-toxic, no tolerant disinfectant which can remove more than 650 bacteria, virus, and fungi species. Therefore, silver nanoparticles attract an increasing amount of scientific and industrial interest from fields such as textile science [5, 6], medicine [7], agriculture [8], sensors and detectors [7], and catalysis [9]. Antibacterial activity of silver nanoparticles containing materials as diverse as polyurethane foam can be used for water treatment [10].

Cotton is a common material for the production of textiles for sport and leisure. It has excellent moisture absorption ability. However, the moist cotton can be easily attacked by bacteria. Decomposed products of body secretions have a characteristic odor [11].

In recent research, a good antibacterial effect of nano-sized silver colloidal solution on polymer and textile fabrics was shown [6, 12]. The objective of this study was not only the synthesis of silver nanoparticles used microwave radiation as a heating source but also investigated the surface morphologies of antibacterial cotton fabrics and the relationship between the antibacterial property and the content of nano-sized silver on cotton fabric was discussed. Moreover, the laundering durability of the bacteriostasis of the silver nanoparticles-treated cotton fabric was also examined.
2. Experimental

2.1. Material
Plain weave cotton fabric (107 g/m²) made by Phuoc Thinh Textile Company, Ho Chi Minh, Vietnam, was used. AgNO₃, Polyvinylpyrrolidone (PVA) and Ethylenlycol (C₂H₄(OH)₂) were obtained from MERCH-Germany was purchased from Hoa Nam Company-Vietnam. *Escherichia coli* (*E. coli*) (ATTC 25922) and *Staphylococcus aureus* (*S. aureus*) (ATCC 290408) were provided by Pasteur Institute-Ho Chi Minh, Vietnam. Nutrient agar was provided by Laboratory for Microbiology – Natural Science University, Ho Chi Minh city, Vietnam.

2.2. Synthesis of nano-sized silver colloid solution
An aliquot of 0.70 g of PVP and 50 ml C₂H₄(OH)₂ was mixed by magnetic stirrer at 80 – 90°C for 1 h. After that, amount of 0.05 g AgNO₃ was added into PVP solution. This solution was placed into microwave oven chamber for 4 minutes at 160 W (figure 1). Finally, nano-sized silver colloid solution which has yellow color was obtained.

The UV-visible absorption behaviors for silver nanoparticles suspensions were recorded by using UV-VIS spectrophotometer (Varian, model 100, Australia). The size and shape of silver nanoparticles were measured using a Transmission Electron Microscopy (TEM) JEM model 1400, 100 kV. The size distribution of the particles was determined by using UTHSCSA Image Tool 3.00 software.

2.3. Preparation of antibacterial cotton fabric
The concentration of colloidal silver solutions was varied - 20, 50, 80 and 100 ppm - by diluting each nano–sized silver colloid solution with distilled water. The cotton fabric (d = 9 cm) were padded with each concentration of the colloid solutions about 5 minutes and squeezed to 100% wet pick-up using a laboratory pad at constant pressure. The samples were immediately dried at 80°C for 15 minutes. After that, the samples were laundered in water for 15 minutes and dried at 80°C. The antibacterial cotton fabrics were obtained.

The dispersibility of the silver nanoparticles on the surface of the cotton fabric were estimated by using a field-emission scanning electronic microscopy (FESEM, S-4800) was operated at 5kV and used at 20000 magnifications.

Inductively coupled plasma Atomic Absorption Spectroscopy (ICP-AAS) was used to measure the remained quantity of silver particles on cotton fabric surface. We compared the concentration of silver particles on fabrics before washing to the particles on them after 5, 10, and 15 times washing.

2.4. Antibacterial test
We used Colony Count Method to study the antibacterial properties of the fabrics finished with the nano-sized silver colloid solution. The effect of bacterial strains is determined by the relation

\[ \eta = \frac{N_1 - N_2}{N_1} \times 100\% \]

where \( \eta \) is the percentage of bacterial reduction, \( N_1 \) is the number of surviving bacterial colonies from the control sample, and \( N_2 \) is the number of surviving colonies from test sample.

3. Result and discussion

3.1. Characterization of nano-sized silver colloid solution
The reduction of silver nanoparticles was visibly evident from the color changes associated with it (from white to yellow). Figure 2 shows the UV-VIS absorption spectra and image of nano-sized silver colloid after the process of reduction. As shown in figure 2, absorbance of nano silver synthesized by microwave heating was very high. From the Bee’s law, a UV-visible absorbance is proportional to the path length and the concentration of the suspension.
Hence, it can be concluded that the suspension of microwave heating has a high concentration of Ag nanoparticles. The sharp peak at 407 nm can be proved a narrow size distribution of silver nanoparticles formed in the solution, because the maximum absorbance peak shifts to a longer wavelength when the particles becomes larger [13] and heating by microwave is more uniform [14]. As evident in figure 3, the UV-visible spectra of silver nanoparticles do not change after a month of ageing time. It can be concluded that the stable nature of nano-sized silver in solution is very high. TEM image and the size Ag nanoparticles distribution results showed that the silver nanoparticles were found to be spherical in shape, having a mean size about 7-11 nm (figure 4).
3.2. Morphologies of antibacterial cotton fabrics

Figure 4. Transmission electron micrograph and particle size distribution of Ag nanoparticles.

Figure 5. SEM images of antibacterial cotton fabrics treated by immersed in silver colloids: a) control; b) 20 ppm; c) 50 ppm; d) 80 ppm; e) 100 ppm.
Figure 5 is the FESEM images of the antibacterial cotton fabrics with different content of nano Ag colloid. As predicted, the silver nanoparticles were generally well-dispersed on fiber surfaces in each fabric. On the other hand, the surface morphology and the content of Ag nanoparticles on cotton fabrics (table 1) varied with the concentration of the nano-sized silver colloid solution. In figure 5b, the suspension concentration of nano silver colloid solution was low, silver nanoparticles were only observed at the cotton fiber surface, suggesting a low grafting content. As the concentration increased, Ag nanoparticles were well dispersed on cotton surface (figures 5c and 5d) with a few agglomerates, and the content of nano silver on cotton fabric was also increased. With a further increased suspension concentration, more Ag nanoparticles are aggregated to one another on the fibers because of their high surface free energy, so bigger agglomerates were observed on cotton fiber surface (figure 5e). However, Hoon Joo Lee and Sung Hoon Jeong [6] concluded that the agglomerated silver particles do not seem to have any ill effect on the antibacterial activity of nano-sized silver colloidal on the cotton fabric samples.

| Table 1. The content of silver nanoparticles on cotton fabrics. |
|---------------------------------------------------------------|
| Concentration of nano silver colloid solution | 20 ppm | 50 ppm | 80 ppm | 100 ppm |
| Content of nano silver on cotton fabrics (mg/kg) | 276     | 698     | 702     | 758     |

3.3. Antibacterial property of antibacterial cotton fabrics

3.3.1. Effect of nano-sized silver colloidal concentration on the percentage reduction of bacteria

The antibacterial activity of cotton fabrics was resulted from the presence of nano silver particles grafted on their surface. The effect of the suspension concentration of nano silver colloidal on the antibacterial activity of cotton fabrics was shown in table 2. It has also been seen that the percentage reduction of bacteria increased with increasing concentration of nano-sized silver colloid and correspondingly with the content of nano silver on cotton fabrics. When the concentration was lower 50 ppm, the percentage reduction of *E. coli* bacteria was higher than and *S. aureus*. It has been known that the structures of gram-positive and negative cell walls have different compositions. The layer of peptidoglycan (about 20-30 nm) in gram-positive bacteria is thicker than gram-negative bacteria [7]. However, when the concentration was over 50 ppm, the percentage reduction of bacteria showed a slow increase. Above results can be found that silver nanoparticles on cotton surface are necessary for inhibition of the bacterial growth.

3.3.2. Effect of contact time on the reduction of bacteria

| Table 2. The antibacterial activity of cotton fabrics treated in different nano-sized silver colloidal concentration. |
|---------------------------------------------------------------|
| Concentration of nano-sized silver colloid (ppm) | Content of nano-sized silver on cotton fabric (mg/kg) | *E. coli* | | *S. aureus* | |
| | | Bacterial reduction (%) | Standard deviation | Bacterial reduction (%) | Standard deviation |
| 20 | 276 | 44.88 | 4.18 | 39.62 | 2.70 |
| 50 | 698 | 96.12 | 1.00 | 94.08 | 0.96 |
| 80 | 702 | 99.91 | 1.11 | 99.31 | 2.40 |
| 100 | 758 | 99.97 | 2.14 | 99.96 | 1.30 |

The effect of the contact time on percentage reduction of bacteria with antibacterial cotton fabrics (cotton fabrics were padded with concentration of nano-sized silver colloid 100 ppm) against *E. coli* and *S. aureus* is shown in figure 6. It can be found that the reduction of bacteria cotton fabric
increased to 65% after 10 h in contact with *E. coli* or *S. aureus*. It can be seen clearly that the percentage reduction of *S. aureus* bacteria is lower than *E. coli* bacteria at 10 h, because the cell wall for gram-positive consists of linear polysaccharide chains cross-linked by short peptides to form a three dimensional rigid structure [15]. From figure 6, it can be seen that the percentage reduction of nano silver on cotton fabrics was to 99.99% and 99.96% after 15 h in contact with *E. coli* and *S. aureus*. Hence, it can suggest that the antibacterial activity of cotton fabrics immersed by nano silver colloid solution was excellent.

![Figure 6. Antibacterial cotton fabrics with against *E. coli* and *S. aureus*.](image)

### 3.3.3. Effect to washing times on the reduction of bacteria

The silver nanoparticles on cotton surface may be washed away when immersed in water. Therefore, the adhesion of silver nanoparticles on cotton fabrics was evaluated by immersing the sample (the specimens were padded with concentration of nano-sized silver colloid 100 ppm) in water and keeping stirred for a given time. Antibacterial activity was tested after the immersion. Table 3 shows the results for the washing 5, 10, 15 times. It can be seen that the antibacterial percentage of the sample washed for 15 times was lower than that of one’s washed for 5 times and that of the content of silver nanoparticles on cotton fabrics decreased with the increase of washing times. This phenomenon is associated with the weak physical bonding between the silver nanoparticles and cotton surface. However, even after washed for 10 times, the content of silver nanoparticles on cotton fabrics about 370 mg/kg gave an antibacterial percentage of around 50%. Therefore, the durability of the antibacterial cotton fabrics was satisfied.

| Washing times | Content of nano-sized silver on cotton fabric (mg/kg) | Bacterial reduction (%) |     |           |
|---------------|-----------------------------------------------------|-------------------------|-----|-----------|
| 5             | 570                                                 | E. coli                 | 97.42 | 95.87     |
| 10            | 370                                                 | S. aureus               | 55.66 | 41.67     |
| 15            | 230                                                 |                         | 36.85 | 26.42     |
4. Conclusions
Silver nanoparticles were synthesized by a reduction method where microwave radiation was used as heating source, the as-prepared silver nanoparticles were nearly spherical particle with the particle size distribution about 7-11 nm. The structure of nano Ag had no change after adhesiving cotton surface.

The silver nanoparticles were grafted and well dispersed on the surface of the cotton fabrics. With the increased concentration of nano-sized silver colloid during immersing, especially for concentration over 80ppm, the particles of nano Ag became agglomerated on the cotton surface.

The antibacterial percentage of cotton fabrics showed excellent antibacterial activity against the tested germs. Conten of nano silver on cotton fabric and the antibacterial percentage were adjustable by controlling the concentration of nano-sized silver colloid during immersing. When the concentration was 80 ppm, the antibacterial percentage was 99.91% against *E. coli* and 99.31% against *S. aureus*. In addition, a good laundering durability of the antibacterial cotton fabrics was obtained, after washed for 10 times.

References
[1] Burnision N, Bygott C and Stratton J 2004 *J. Surface Coating International Part A* 179
[2] Fei B, Z Deng, Xin J H, Xhang Y, Pang G 2006 *Nanotechnology J.* 17 1927
[3] Shi Z L, Neoh K G, Kang E T 2005 *Biomaterials* 26 501
[4] Choi S H, Zhang Y P, Gopalan A, Lee K P, Kang H D 2005 *Colloids and surface A: Physicochem. Eng. Aspects* 165
[5] Satio M 1993 *J. Coated Fabrics* 23 150
[6] Lee H J and Jeong S H 2005 *Textile Res. J.* 75 551
[7] Silver S, Phung L T, Silver G 2006 *J. Ind. Microbiol. Biotechno.* 33 627
[8] Park H P, Kim S H, Kim H J, Choi S H 2006 *Plant Pathol. J.* 22 295
[9] Vaseashta A, Malinvoska D D 2005 *Sic. Technol. Adv. Mater.* 6 312
[10] Jain P, Pradee T 2005 *Biotecnol. Bioeng* 90 59
[11] Goresek M and Recel P 2007 *Textile Res. J.* 77 138
[12] Yeo S Y, Lee H J and Jeong S H 2003 *J. Mater. Sci.* 38 2199
[13] Heard S M, Grieser F, Barraclough C G and Sanders V J 1983 *Colloid Interface Sci.* 93 545
[14] Jiang H, Moon K, Zhang Z, Pothukuchi S and Wong C P 2006 *J. Nanoparticles Research* 8 117
[15] Shrivastava S, T Bera, Roy A, Singh G, Ramachandrarao P and Dash D 2007 *Nanotechnology J.* 18 225103

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