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Durable Anti-bacterial Nylon Carpet Using Colloidal Nano Silver

Abstract
Silver nanoparticles (Ag-NPs) are increasingly being incorporated in a variety of products, textiles, and in healthcare, mainly due to their antibacterial properties. The present study investigated the antimicrobial efficiency and colour changes of floor covering loaded with colloidal silver nanoparticles via a simple and cost-effective method. The influences of colloidal concentration on antimicrobial activity against Escherichia coli and Staphylococcus aureus as well as laundering durability were studied. The results of the silver-treated floor covering (nylon 6 piles) with 50 - 100 ppm dilution exhibited outstanding antimicrobial efficiency and indicated a 99.42% reduction in Staphylococcus aureus and a 79.25% reduction in Escherichia coli. Furthermore, the bioactivity of Ag-NPs was maintained even after ten washings. The removal of silver nanoparticles in wastewater was investigated by UV–visible spectroscopy. Scanning electron microscopy (SEM) and EDX were employed to confirm the presence of nano silver on the surface. The results indicated that Gram positive bacteria are more tolerant to silver than Gram negative bacteria. An appropriate antimicrobial agent deposited on floor covering can prevent unpleasant odours and growth of pathogenic microorganisms.

Key words: *antimicrobial, nano silver, floor covering, nylon, durability.*

Introduction

In the past few decades many efforts have been made in nanotechnology and nanoparticles due to their unique properties and potential application in healthcare, medicine, domestic textiles, and hygienic as well as protective textiles [1 - 4]. Nowadays there has been constantly increasing concern about the health hazard arising during medical and other treatment from microbial infections [5]. The means of shielding the human body against such threats need to be developed, with one of the most popular being the production of antimicrobial textiles for usage inside protective clothing, medical gauzes and sheets [6]. Due to the significant increase in using bactericide, antiviral and fungicide products, there is great demand for the antimicrobial finishes of textiles, as an excellent culture to control the growth of microorganisms and prevent textiles from the deterioration of odours, which is a health concern caused by microorganisms [7-9]. Silver based antimicrobials have captured much attention not only because of the non-toxicity of active Ag^+ to human cells but also due to their novelty in being a long lasting biocide with high temperature stability and low volatility [10, 11].

Silver nanoparticles show an efficient and strong antimicrobial property compared to other agents due to their large surface area to volume ratio, which provides better contact with microorganisms [12 - 15]. Ag-NPs penetrate inside the cell membrane and react with thiol groups, thereby preventing protein synthesis [16, 17].

Multifunctional domestic implements, especially floor covering with antimicrobial properties, have been actively developed to prevent human contamination and the exposure of any bacterium in the human life environment [18]. The large surface area and ability to retain the moisture of the floor covering also assist the growth of microorganisms on the fabric [19, 20]. Using silver nanoparticles leads to an increase in the number of particles per unit area, thereby maximising antimicrobial effects [21].

The purpose of this study was to determine the antibacterial activity of silver nanoparticles deposited on nylon carpet against *Staphylococcus* aureus and *Escherichia coli* bacteria. As one of the challenges in the development of textile applications is to keep the process simple and inexpensive, special attention was paid to using an easy and applicable method. Therefore this paper presents a simple and effective method for the antibacterial treatment of nylon carpet that contains silver nanoparticles via spraying.

Experimental

Material

The solution used was colloidal nano silver in alcohol media with an average particle size of 5 nm (0.8%) 8000 ppm, supplied by Narminchemie Co. Iran. A carpet of pile loop nylon was produced by Palaz Moquette Co. Iran. Carpet samples

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were in a nylon (polyamide-6) pile-loop form. The carpet is formed from three layers, with the first layer made of nylon yarn, the second layer - polypropylene, and the third layer was non-woven polyester attached to the second layer with a synthetic resin.

Method

First, silver nano particle solutions of different concentration (50, 75, 100 ppm) were prepared. The volumes of the solutions were chosen based on the appropriate concentration of nano silver in the ppm scale; the primary concentration was 80 ppm, selected within the range 50 - 100 ppm. Then nylon carpet samples were cut to the size of 5 × 10 cm^2.

Here a spraying method was used as being convenient for the application of an antibacterial compound to the carpet due to the latter's construction. Thus 10 mL of different percentages of silver nano particle solution were sprayed on the surface of the carpet, and then the samples were put in an oven at 100 °C for 30 min to 90 min. Then the samples treated were cut to the small size of 0.5 × 1 cm^2 and prepared for antibacterial testing

The AATCC 100-2004 test method was applied to evaluate bacterial reduction via immersing the treated samples in bacteria solution. The two main types of bacteria, a Gram positive (*Staphylococcus aureus*, AATCC 6538) and a Gram negative (*Escherichia coli*, AATCC 11303), were prepared for testing. All samples were tested in two cultured bacteria based on the McFarland standard, 1.5 × 10^8 CFU/mL and 1 × 10^{-3} (0.5 McFarland). The sterile samples prepared were immersed in 1 mL of an appropriate dilution in a bacterium test tube (according to the McFarland standard). The test tubes contained carpet and bacteria solution and were transferred to an incubator with a temperature of 37 °C for 24 h. The tubes were then removed from the incubator and the solution was dripped onto a plate to assess antibacterial properties. From each test tube, 0.1 mL of bacterial suspension mixed with melted trypticase soy agar (45 °C) was cultured and placed in the incubator at 37 °C for 24 h, and then the bacterial colony was counted. The bacterial reduction was calculated with the use of ***Equation 1***.

Table 1. *Assessment of antibacterial finishes on nylon 6 carpet treated with nano silver after one to ten washings with a dilution factor of 1×10-3 (0.5 McFarland).*

Bacteria		Silver concentration, ppm					
		50		75		100	
		1st washing	10th washing	1st washing	10th washing	1st washing	10th washing
Staphylococcus aureus, CFU/mL	Start	1.9×10^4		1.9×10^4		1.9×10^4	
	After 24 h	0	6×10^1	0	1×10^1	0	0
	Reduction %	99.99	99.69	99.99	99.95	99.99	99.99
Escherichia coli, CFU/mL	Start	1.9×10^4		1.9×10^4		1.9×10^4	
	After 24 h	1.9×10^3	3.1×10^3	1.1×10^2	2.6×10^2	2×10^1	4.1×10^2
	Reduction %	92.11	83.68	99.42	86.32	99.89	97.84

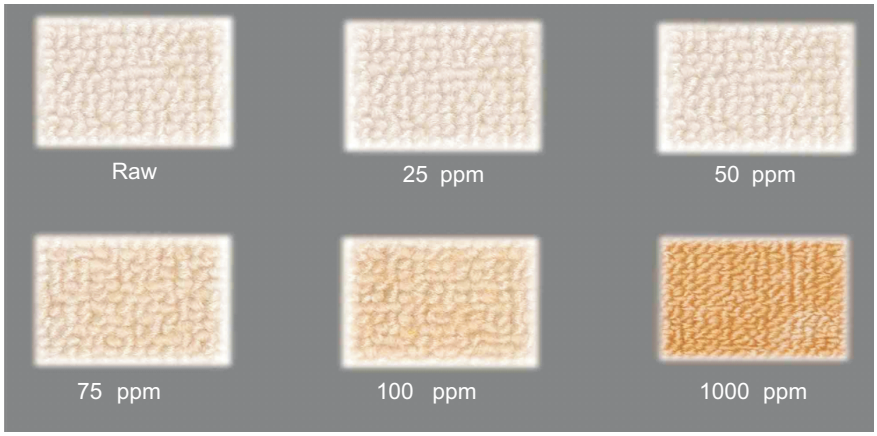

Figure 1. *Colour changes of raw and treated carpets with different Ag-NPs concentrations.*

$$C = \frac{A - B}{A} \times 100 \qquad (1)$$

where C is the bacterial reduction ratio in percentage CFU/ml, A the number of bacterial colonies from untreated fabrics, and B is the numbers of bacterial colonies form treated fabrics.

Moreover the colour change was considerably more prominent on the carpet loaded with Ag-NPs in high dilution colloid. The colour changed from yellow to brown, which occurs during an oxidation reaction, being a common disadvantage of Ag particles. This experiment attempted to find an appropriate dilution not only with an excellent antimicrobial property but also one producing carpet without yellowing. To assess colour changes in the carpet, color indexes L*a*b* were measured and reported. Also the colour changes were reported based on the calculation of ΔE (***Equation 2***).

$$\text{Contrast} = \Delta E = E_2 - E_1 = \qquad (2)$$
$$= \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2}$$

With regards to the importance of durable finishing treatment and exploring the durability of silver on a product against repeated washing, samples were standard washed 1 to 10 times with 1% (w/v) detergent solution at 60 °C for 20 min, and finally the antibacterial characteristics were obtained and compared.

Moreover the spectra absorbed were measured with a Cary 300 UV-vis spectrophotometer to show the existence of nano silver in the washing effluent.To have a precise study of the treated carpet surface and silver nano particles, scanning electron microscopy (SEM: model LEO 440i, England) at 300 - 30000 magnification was used. Energy-dispersive X-ray analysis (EDX) was also used to confirm the presence of silver particles as well [22].

Results and discussion

By counting the number of bacteria in the control and treated sample, the reduction in the bacterial percentage was determined. The results of the reduction in all concentrations with two bacteria are reported in ***Tables 1*** and ***2. Table 1*** indicates that the minimum dilution of silver nano particle solution (50 ppm = 0.05%) has a bacteriostatic property of 99.99% in 1.9 × 10^4 CFU/mL of *S. aureus* bacterium, and by increasing the dilution up to 100 ppm a 99.99% bacterial reduction was obtained.

Table 2. *Antibacterial properties of carpets treated with nano silver after one and ten washings against different bacterium with 0.5 McFarland.*

Bacteria		Silver concentration, ppm					
		50		75		100	
		1st washing	10th washing	1st washing	10th washing	1st washing	10th washing
Staphylococcus aureus, CFU/mL	Start	6.5×10^5		6.5×10^5		6.5×10^5	
	After 24 h	2.2×10^4	7.9×10^4	1.5×10^4	1.8×10^4	3.8×10^2	3.8×10^3
	Reduction %	96.62	87.85	97.69	97.23	99.94	99.42
Escherichia coli, CFU/mL	Start	5.3×10^5		5.3×10^5		5.3×10^5	
	After 24 h	2.2×10^5	2.7×10^5	3.4×10^4	1.9×10^5	<10	1.1×10^5
	Reduction %	58.49	49.06	93.58	64.15	99.99	79.25

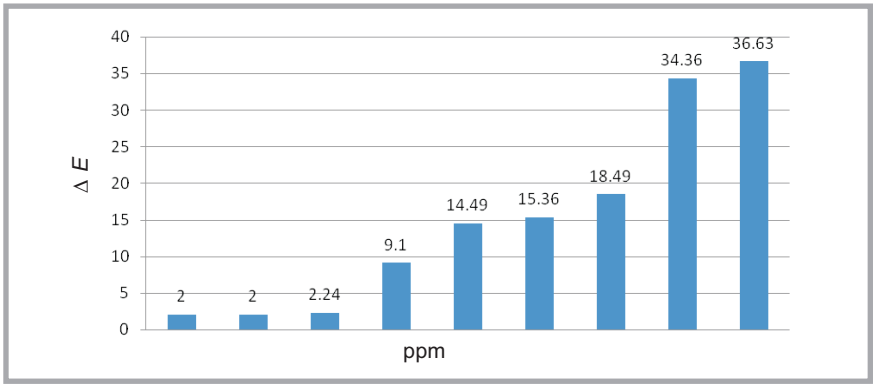

Figure 2. *ΔE alteration for different carpets treated with various Ag-NPs concentrations.*

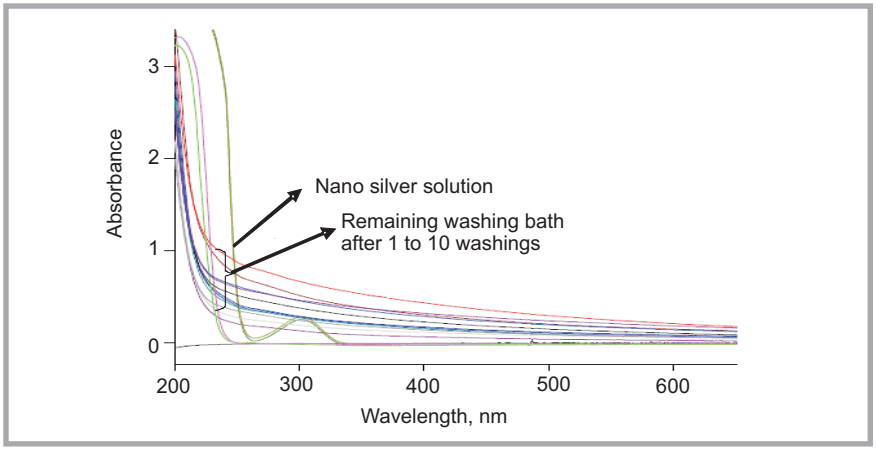

Figure 3. *UV–vis spectrum of the silver nanoparticle solution and remaining washing bath after 1 to 10 washings.*

Lee and Jeong observed a bacteriostatic of 99.99% against *S. aureus* and *K. pneumonia,*while the concentrations of the colloidal silver bath were 10, 20, and 30 ppm on nonwoven polyester fabrics treated with 2 - 3 nm silver particles [23].

Table 1 (see page 97) shows the silver not only preserved its durability after repeated washings but also its antibacterial property. The durability of the antibacterial property against *S. aureus* was more than for *E. coli*. However, after 10 washings, the percentage of bacterial reduction decreased due to unabsorbed silver nanoparticles on the surface of the fibre, which had been removed after primary rinsing. The reduction percentages were low in comparison with the increase in concentration of nano silver but were within the acceptable range of antibacterial activity.

The highest antibacterial properties against two different kinds of bacteria were obtained at 1.9×10^4 CFU/mL (lower bacteria) with 100 ppm of nano silver. ***Table 1*** (see page 97) indicates that by decreasing the dilution of bacteria to 1.9×10^4 CFU/mL, an appropriate antibacterial is obtained.

Using a colloidal solution of nano silver with a 50 - 100 ppm concentration produced the best antibacterial properties without any significant changes in colour. ***Figure 1*** (see page 97) presents the colour produced on the samples treated with different concentrations, and ***Figure 2*** shows the colour changes based on ΔE.

Furthermore, to confirm the presence of silver nanoparticles in the effluent and its durability during repeated washings, UV-vis spectroscopy was employed. The curves indicate that there is a peak at around 300 nm for nano silver solution, whereas there is no peak to confirm the presence of nano silver in the effluent. The results are shown in ***Figure 3***.

SEM images and EDX analyses obtained from the samples confirmed the presence of silver on the surface of the carpet. SEM images of the nanoparticles are illustrated in ***Figure 4***. In all SEM images, Ag-NPs showed in white colours after 10 repeated washings, confirming reasonable durability for antibacterial properties. The EDX patterns indicated a small peak related to Ag, due to the very low silver concentration (100 ppm) (***Figure 5***).

According to the results presented in ***Table 2***, Ag-NPs showed very good antibacterial properties even at a very low concentration in relation to the maximum bacteria dilution 1.5×10^8 (0.5 McFarland).

The bacterial reduction of carpets with 75, 100 ppm of Ag-NPs against *S. aureus* was about 98% after the first washing due to the high durability of the silver nanoparticle solutions. The resistance of nano silver gradually decreased particularly against *S. aureus,* showing very low resistance after ten washings. There is also a slight difference by increasing the dilution from 50 to 100.

E. coli was more resistant than *S. aureus* against the antibacterial compound after 10 washings; however, by increasing the silver concentration from 50 ppm to 100 ppm, the antibacterial property was about 79.25% after 10 washings.

The aim of this study was to provide strict conditions on a product to determine the maximum viability of silver nanoparticles against two kinds of bacteria. ***Figures 6*** and ***7*** (see page 100) show the number of bacterial colonies in the con-

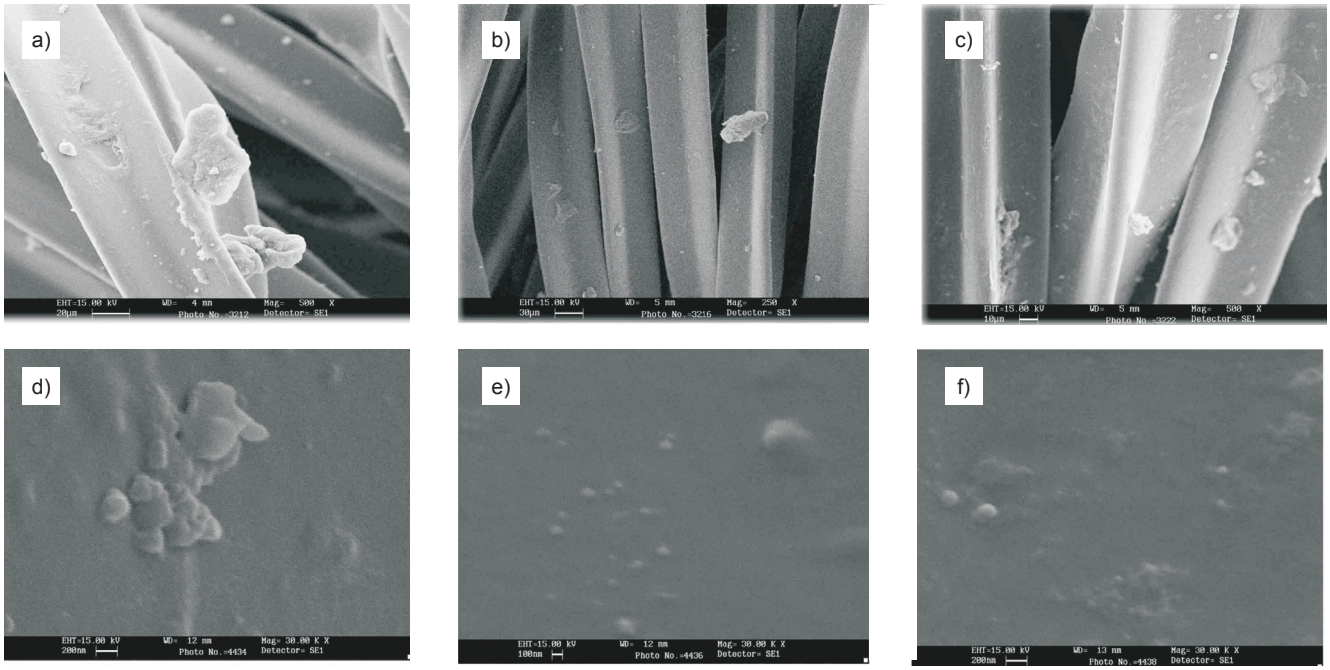

Figure 4. *Scanning electron microscopy (SEM) of nylon 6 fibres at different magnifications in 100 ppm nano silver: a) without washing (500×), b) without washing (30000×), c) after one washing (500×), d) after one washing (30000×), e) after ten washings (500×), f) after ten washings (30000×).*

trol and treated samples after 10 washings against both bacteria.

Zheng et al. reported that silver treated cotton fabrics showed an excellent and durable antibacterial effect against both *S. aureus* and E. *coli* with over 98.77% bacterial reduction even after 20 consecutive home launderings [24]. Vesna et.al indicated that cotton fabrics loaded with silver nanoparticles from 10 ppm and 50 ppm colloid exhibit excellent antibacterial activity against *E. coli*, *S. aureus* and *C. albicans*. On the other hand, in another research it was reported that cotton fabrics loaded with silver nanoparticles with 10 ppm colloid showed poor laundering durability. However, the desirable antibacterial efficiency of cotton fabrics loaded with silver nanoparticles from 50 ppm colloid was preserved after five washings [25]. Jantas indicated that antibacterial textile showed an excellent antibacterial effect against *E. coli* and could withstand 50 washings [26].

Suk-Woo Park et al. revealed that nylon 6/silver possessed excellent antibacterial properties and an inhibitory effect on the growth of *S. aureus* and *K. pneumoniae* [27].

In the present study, the most susceptible bacteria were *S. aureus* and *E. coli* . Tests performed on carpets indicated that the antibacterial properties were greater against the Gram negative (*E. coli*) than with the Gram positive (*S. aureus*). In general, Gram positive bacteria appeared to be more tolerant to silver than Gram negative cells. It has previously been reported that Gram positive bacteria are less susceptible to the antibacterial activity of silver. This resistance may be attributed to Gram negative bacteria with complicated cell walls. The cell wall of Gram negative consists of lipids, proteins and lipopolysaccharides (LPS), providing effective protection against biocides, whereas Gram positive is without LPS. On the other hand, Gram positive has a simple cell wall structure in which the cytoplasm membrane has a rigid peptidoglycan layer composed of networks with plenty of pores, which allow foreign molecules to enter the cell without any difficulty [28-30].

Our intention in this work was to assemble silver nanoparticles on carpets using a simple method applicable in industry in terms of safety and with the least impact on the environment. Nylon was chosen as a biodegradable and biocompatible polymer, widely used in many industrial fields due to its low cost, superior fibre forming ability (resiliency), good mechanical strength, and strong chemical and thermal stability.

Conclusions

The purpose of this paper was to find an easy way to apply nano size silver colloi-

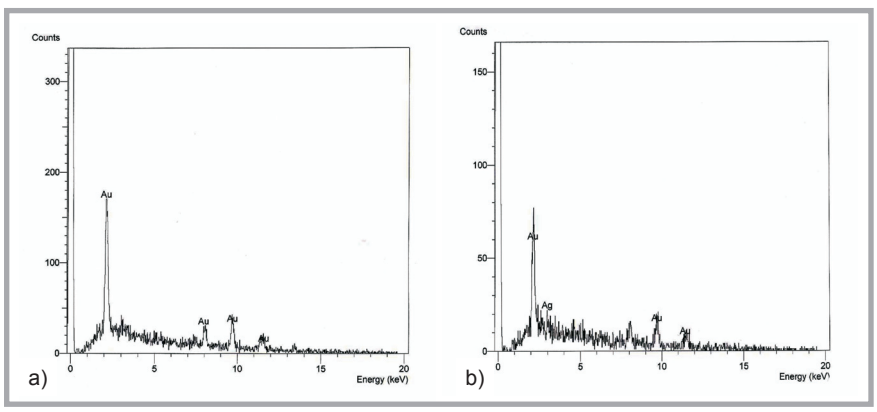

Figure 5. *a) EDX patterns of a) raw sample b) sample treated with 100 ppm nano silver after 10 washings.*

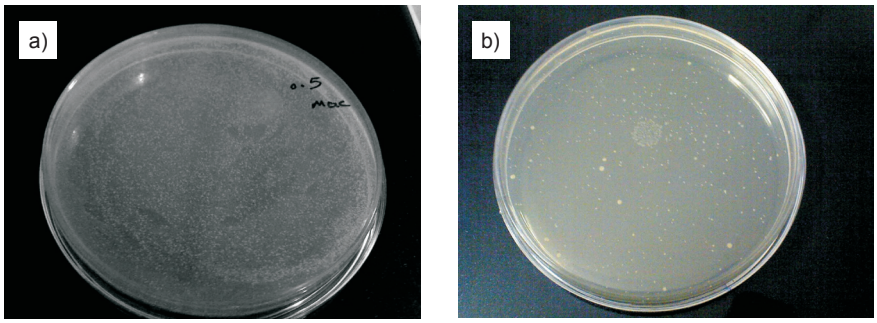

Figure 6. *Growth of S. aureus on a) control sample (6.5×10^5 CFU/mL), b) sample treated with 100 ppm nano silver after 10 washings (6.5×10^5 CFU/mL).*

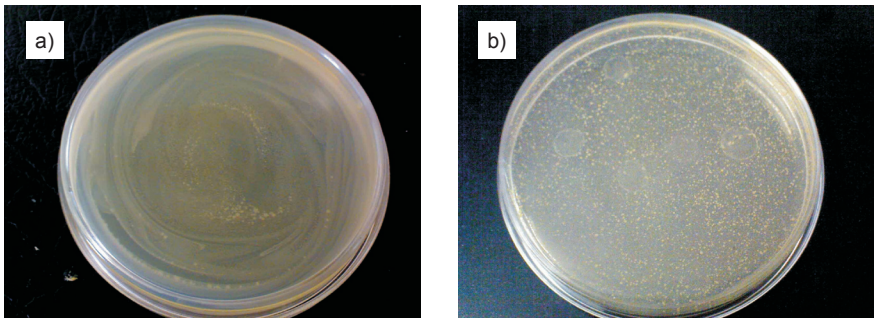

Figure 7. *Growth of E. coli on a) control sample (5.3×10^5 CFU/mL), b) sample treated with 100 ppm nano silver after 10 washings (5.3(105 CFU/mL).*

dal solution on carpet to obtain an antibacterial effect without colour changes. Utilising nano silver solution to remove bacteria, due to its economical consumption and competent performance of its applications, is widely employed in comparison with other finishing agents. By controlling the activity of the pathogenic factor, this technology is important for everyday applications. Therefore it has become preferred to other improvement and manufacturing methods because of its high efficiency, applicability, environmental compatibility and durability . With regard to the operations carried out on nylon carpet with very low rate of nano silver (50-100 ppm), the highest degree of removing bacterial against the most common bacteria was achieved without significant colour change. In addition, the spraying method could be applied to the last stage of finishing on the carpets or even used domestically by spraying during usage. UV-vis spectra confirmed that there is no silver in washing effluent. This high aspect ratio of the antibacterial property on carpet without any side effects of silver on the environment suggests potential application in other textile areas.

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