Research Article

Synthesis, Characterization and Application of Silver nanoparticles for Synthetic Fabrics

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ABSTRACT

Eco-friendly synthesis of silver nanoparticles in situ onto synthetic fabrics [polyester (PET), polyamide6 (PA6) and polypropylene (PP)] is carried out by reduction of silver nitrate salt into silver nanoparticles. The presence of nanosilver onto fabrics was confirmed by Scanning electron microscope (SEM) and Energy dispersive X- ray spectroscopy (EDX) patterns. SEM images indicated the well distribution of silver nanoparticles onto PET and PP fabrics surface and the formation of a nanolayer onto PA6fabric. The treated fabrics acquired outstanding antibacterial and antifungal properties against *Staphylococcus aureus*, *Klebsiella. pneumonia* and *Candida albicans*. Furthermore, surface electrical conductivity and color changes are measured for the treated fabrics. Nano Ag coated PA6 fabric displayed electrical conductivity better than the untreated one.

Keywords: Silver nanoparticles, Synthetic fabrics, Green synthesis, SEM, EDX, Antibacterial activity, Electrical conductivity.

INTRODUCTION

Nanocomposites are used in many applications such as medical textiles, cosmetics, agriculture, optics, food packaging, semiconductor devices, aerospace, construction and catalysis. Nanoparticles can be incorporated into polymeric composites to improve thermal, mechanical, electrical, catalytic and optical properties.[1] New properties are imparted to textiles using nanotechnology such as anti-bacteria, UV-protection, anti-static, improvement of dye ability, flame retardency, water repellency, soil resistance and wrinkle resistance.[2-4] Preparation of silver nanoparticles (Ag NPs) by green synthesis has many advantages compared to the conventional method which involve chemical agents having environmental toxicity.[5]

The green synthesis of Ag nanoparticles consists of three steps as follows: starting with selection of solvent medium, a reducing agent and finally an eco-friendly stabilizing agent for Ag NPs.[6] Green synthesis include polysaccharide, Tollens, irradiation and biological methods. Solutions of $AgNO_3$ containing glucose and starch in water protect Ag NPs, which were used for medical applications. Tollens process involves the reduction of $[Ag(NH^3)_2{}^+]$complex forming nano Ag films, hydrosols and colloid particles of different shapes and particle sizes. Formation of Ag NPs by irradiation of Ag+ ions does not involve a reducing agent, which is an important procedure. Bio-organisms in plant extracts containing proteins, which act as both reducing and capping agents, form stable and shape-controlled Ag NPs. For example, the extract of unicellular green algae *Chlorella vulgaris* was used to synthesize single-crystalline Ag nanoplates at room temperature.[5]

Surface modification of natural and synthetic fabrics by plasma and/or nano silver highly improved hydrophilicity, antibacterial properties, dyeing and fastness properties.[7-10]

Antibacterial properties were produced by coating fabrics with silver nanoparticles via ultrasound radiation in a one-step reaction forming a uniform coating of silver nanoparticles on fabrics surfaces with different functional groups.[11]

Microorganisms play an important role in reduction of metal ions. It was demonstrated that stable silver nanoparticles can be generated by reduction of silver ions extracellular using fungi of *Fusarium oxysporum*. These fabrics exhibited antibacterial activity against *Staphylococcus aureus.*[12]

Green synthesis of silver nanoparticles using plant extract is an interesting method. Natural products have antibacterial, antioxidant and antitumor activity.[13-15] *Parthenium hysterophorus* was used for the removal of heavy metals and dyes from the environment. So, synthesis of silver nanoparticles from bio reduction of *Parthenium hysterophorus* leaf extracts showed effective antibacterial, and antioxidant activity.[16]

Antimicrobial polypropylene / nano Ag (PP/Ag) of nonwoven dressings was manufactured by melt extrusion and spinning technique.[17-19] Application of PP/Ag dressings to wounds enhanced complete wound healing.[20-23]

Conductive nylon6 fabric was prepared using nano silver through in situ, green and low cost method based on Tollens' reagent (silver/ammonia complex $[Ag(NH_3)_2]^+$). The functional groups of polyamide chains reduced silver complex without using any additional chemical reducing agents.[24, 25]

In the present investigation, an eco-friendly synthesis of silver nanoparticles onto synthetic fabrics through in situ reduction of silver nitrate salt into silver nanoparticles is presented. Characterization of nano silver onto fabrics was confirmed by SEM and EDX patterns. Antibacterial and antifungal activity was determined by optical density measurement. Furthermore, surface electrical properties and color changes of treated fabrics were measured.

MATERIALS AND METHODS

Materials

Fabrics

Nonwoven polypropylene (PP, 70 g/m^2, thickness 0.58 mm), woven polyester fabrics (PET, 155g/m^2, thickness 0.33 mm) and woven polyamide 6 (PA6, 103 g/m^2, thickness 0.31 mm).

Chemicals

Silver nitrate and sodium hydroxide were purchased from local market and have been used as received. Nonionic wetting agent (Triton X-100) was obtained from Aldrich Co.

Methods

Scouring of fabrics

Woven PET and woven PA6 fabrics were washed with 2g/L nonionic detergent (Triton X-100), at 50°C for 1h, liquor ratio 1:50 with continuous stirring. Nonwoven PP fabric was extracted with acetone for 30 min to remove lubricants.

Synthesis of silver nanoparticles from silver salt

Adjust 80 ml distilled water to pH 12 using sodium hydroxide (0.1 M) then 1 g of fabric was added and heated at 85°C with continuous shaking. 10 ml of silver nitrate (0.058M and 0.015 M) was added drop wise and the solution was continuously heated at 85°C for 2 hours where silver nitrate was reduced to Ag nanoparticles and adhered onto the fabric. Finally, the fabric was thoroughly washed using 2 g/L nonionic detergent at 50°C for 20 min, well rinsed in a sonicator and air dried.

Analysis methods

SEM and EDX

The fabric containing nano silver was well washed with distilled water in a sonicator and then dried before SEM and EDX determination. The fabric was tested on SEM Quanta FEG 250 (FEI Co.) working at 20 KV. The fabric was coated with Carbon double face and fixed with stubs of Quanta holder and examined in low vacuum.

Antimicrobial activity test

The antibacterial and antifungal studies of treated fabrics with Ag nanoparticles were accomplished in triplicates using standard methods (AATCC TM100).

The treated fabric was introduced into 20 ml nutrient broth and inoculated with the respective bacterial strains followed by overnight (24 hrs) incubation at 37°C. Growth of the bacterial strains was determined spectrophotometrically by measuring the optical density at 660 nm (OD_{660}) in presence of the treated fabric against a blank of un-inoculated sterile medium. Similarly, the fungal strains inoculated into potato dextrose broth and incubated for 48 hours at 28°C in a shaker incubator followed by measurement of optical density at 450 nm (OD_{450}) against a blank of un-inoculated sterile medium. Before recording the OD of the respective media after incubation, the culture tubes were shaken thoroughly in order to bring microorganisms into suspension.

Optical density is directly proportional to the number of microorganisms (bacteria or fungi) in the medium. The percentage of reduction of the microorganisms was expressed as follows.

R = (B - A)/B x100 (Eq.1)

R is the percentage of reduction of microbial population; B is the absorbance of the media inoculated with microbes and A is the absorbance of the media inoculated with microbes and treated fabric.[26]

Surface electrical conductivity measurement

Surface electrical conductivity measurement was performed using Computerized LRC-bridge (Hioki model 3531 zHi Tester). The electrical conductivity (σ) for the investigated treated and untreated PA6 fabrics were studied at room temperature and frequency ranging from (0.1 to 100 KHz). The PA6 fabrics used in the electrical conductivity measurement were in disc form, having 10 mm in diameter and pressed using a pressure of 10 tons at room temperature. Then, silver paste was coated to form electrodes on both sides of the sintered ceramic specimens in order to ensure good contact. The electric measurements were carried out by inserting the sample between two parallel plate conductors forming cell capacitor. The electrical conductivity was calculated from the relation:

$\sigma = 2\pi f d\, c \tan\delta / A$ (Eq. 2)

Where σ is the A.C. conductivity, f is the operating frequency, d is the thickness of the sample, tanδ is the loss tangent, c is the capacitance and A is the area of the electrode.

Color measurements

The three coordinates (L*, a* and b*) of CIELAB color system as a common method for color measurement of textile were obtained using Ultra scan Pro Hunter Lab. L* indicates the lightness and a* and b* show the redness–greenness and yellowness–blueness values, respectively. The color difference between two fabrics is determined by ΔE using Eq. (3)[27]

$$\Delta E = [(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{0.5} \quad \text{(Eq. 3)}$$

ΔE: the total difference between the untreated and treated fabrics, L*: represent white-black axis, a*: represent the red- green axis, b*: represent the yellow-blue axis

RESULTS AND DISCUSSION

Eco friendly, low cost syntheses of silver nanoparticles onto synthetic fabrics (PET, PP and PA6) were done to acquire new functionality to these fabrics such as antimicrobial properties. Silver nanoparticles are formed in situ within the polymeric chains of fabrics. The polymeric chains reduced and stabilized Ag nanoparticles.

SEM and EDX

Figures 1-3 illustrated SEM images and EDX patterns of the untreated and treated PET, PP and PA6 fabrics coated with silver nanoparticles respectively.

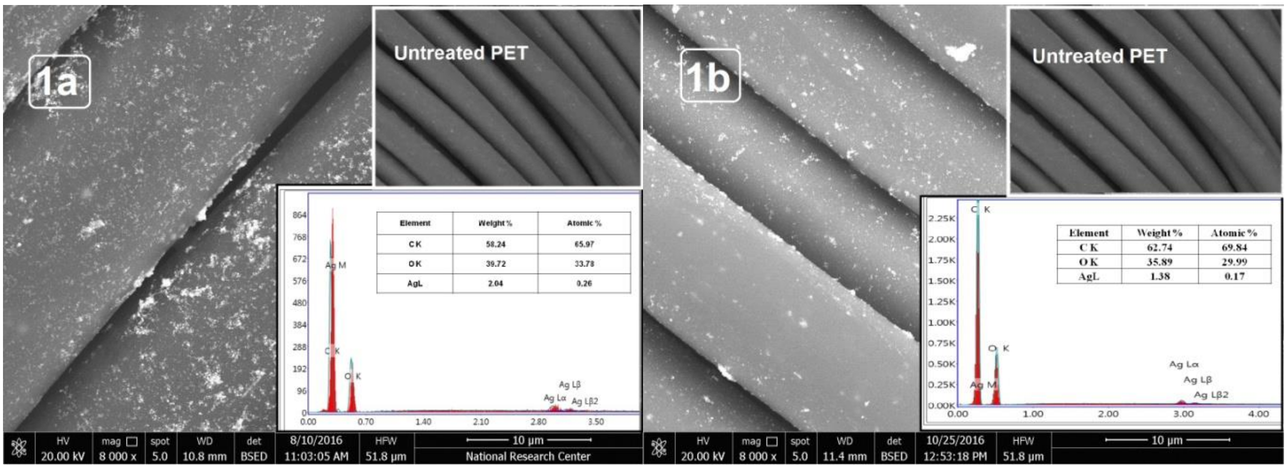

Figure 1: SEM and EDX of PET fabric coated with Ag nanoparticles, $AgNO_3$ concentration (1a: 0.058 M) and (1b: 0.015 M)

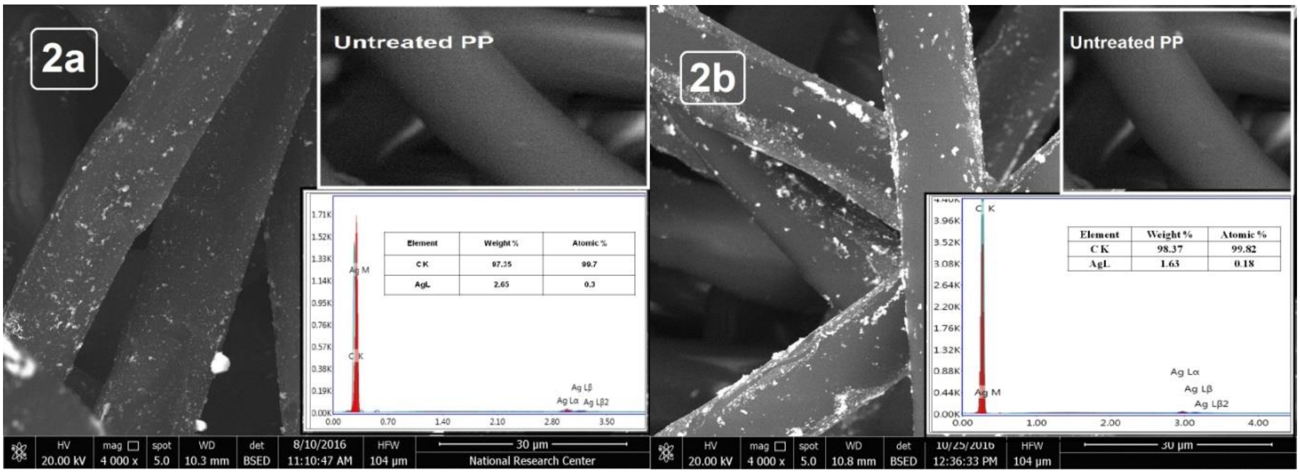

Figure 2: SEM and EDX of PP fabric coated with Ag nanoparticles, $AgNO_3$ concentration (2a: 0.058 M) and (2b: 0.015 M)

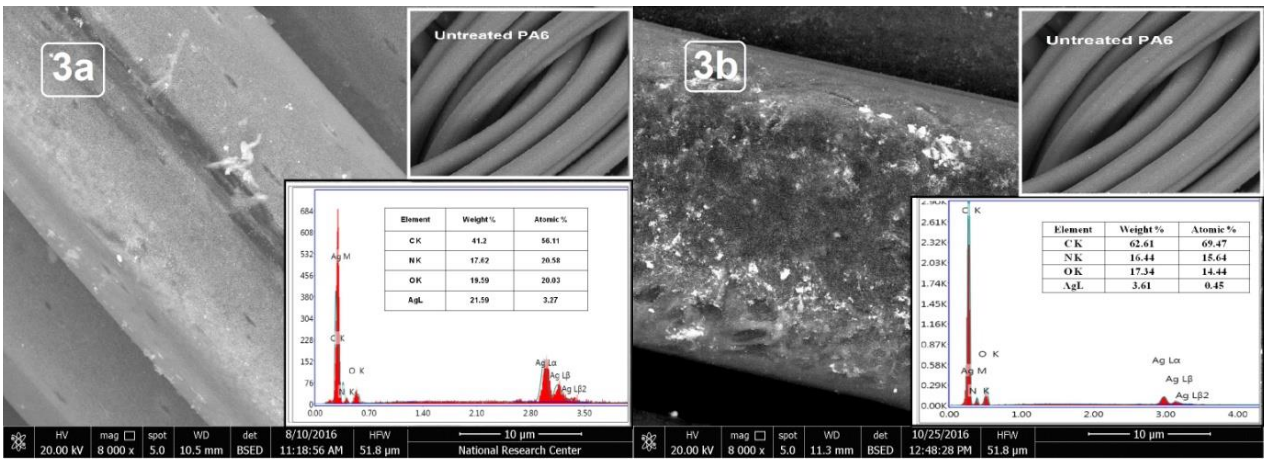

Figure 3: SEM and EDX of PA6 fabric coated with Ag nanoparticles, $AgNO_3$ concentration (3a: 0.058 M) and (3b: 0.015 M)

SEM showed the homogeneous deposition of silver nanoparticles onto the treated PET and PP fabrics and the formation of a nanolayer onto PA6 fabric surface.

Also, EDX spectrum of the treated PET, PP and PA6 fabrics indicated the percentages of the elements based on weight percentage and atomic percentage. It also confirms the presence of Ag nanoparticles on the treated fabric.

Antimicrobial activity test

Silver nanoparticles have high antimicrobial efficacy against a wide range of bacteria, fungi, algae, etc.[5] The treated fabric of PP and PET containing 2% nano silver and PA6 containing 21% nano silver were tested against gram-positive bacteria (*Staphylococcus aureus*), gram-negative bacteria (*Klebsiella pneumonia*) and fungi (*Candida albicans*). An efficient antimicrobial effect is achieved on the treated fabrics due to the presence of silver nanoparticles (Table 1). The treated PET fabric showed the best bactericidal activity on the tested bacteria and fungi.

Table 1: Antimicrobial activity of PP, PA6 and PET fabrics loaded with silver nanoparticles

Fabric	Percentage reduction of microbial count (as compared to control growth)		
	Staphylococcus aureus	*Klebsiella pneumonia*	*Candida albicans*
PP treated with $AgNO_3$ (0.058 M)	92%	99%	95%
PA6treated with $AgNO_3$ (0.058 M)	95%	100%	100%
PET treated with $AgNO_3$ (0.058 M)	100%	99%	100%

Surface electrical conductivity measurement

PA6 fabric loaded with nano silver displayed enhanced electrical conductivity compared with the untreated fabric showed in Table 2. Fabric electrical conductivity increased with increasing the operating frequency. The conduction occurs when the distances between the conductive particles are close enough; roughly less than 10 nm.[24] These results approved the formation of a continuous silver nanoparticles network producing a nanolayer coated the fiber surface.

Color measurements

In case of PA6 fabric, from Table 3, L* value of the treated PA6 fabric is lower than the untreated fabric. Which means the color becomes darker.

Table 2: Conductivity measurements for the treated and untreated PA6 fabrics

Frequency (Hz)	Conductivity measurements ($\Omega^{-1}cm^{-1}$)	
	Untreated PA6 fabric	PA6 treated with $AgNO_3$(0.058 M)
100	0.003	0.006985
1000	0.000314	0.000855
10000	0.000991	0.001911
100000	0.000989	0.003791

At low concentration of $AgNO_3$ (0.015 M), a* and b* values for the treated fabric are higher than the untreated one. This means that a little shift of color towards red yellowish area is happened. By increasing the concentration of $AgNO_3$ (0.058 M), Ag nanoparticles deposition was increased onto the fabric and the hue of color is shifted towards green yellowish area.

Also, for the polyester fabric, a little shift of color (L*, a* and b*) occurred in the same manner as PA6 fabrics.

Table 3: The color difference for the untreated and nano Ag treated PA6 and PET fabrics

Fabric	L*	a*	b*	ΔE
Untreated PA6	71.74	-0.82	-0.68	0
PA6 treated with $AgNO_3$(0.015 M)	27.77	4.55	11.77	46.20
PA6treated with $AgNO_3$(0.058 M)	25.59	-1.54	6.12	46.84
Untreated PET	81.37	-0.55	-1.31	0
PET treated with $AgNO_3$(0.015 M)	61.62	3.01	15.09	25.73
PET treated with $AgNO_3$(0.058 M)	50.27	2.06	14.16	34.59

CONCLUSION

Green synthesis of silver nanoparticles in situ onto synthetic fabrics [polyester (PET), polyamide 6 (PA6) and polypropylene (PP)] was carried out by reduction of silver nitrate into silver nanoparticles. Characterization and confirmation for the presence of nano silver onto treated fabrics were performed by Scanning electron microscope (SEM) and Energy dispersive X- ray spectroscopy (EDX) patterns. SEM images indicated the well distribution of silver nanoparticles onto PET and PP fabrics surface. Moreover, the formation of a nanolayer onto PA6 fabric was illustrated. The treated synthetic fabrics exhibited excellent antibacterial and antifungal activities against *Staphylococcus aureus*, *K. pneumonia* and *Candida albicans*. PET treated fabric inhibited 100% the growth of all tested micro-organisms. Surface electrical conductivity and color changes were measured for the treated fabrics. The electrical conductivity for the treated PA6 fabric increased relative to the untreated fabric due to coating of fabric with silver nanoparticles.

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