

EXPERIMENTAL STUDY ON DYEING PERFORMANCE AND ANTIBACTERIAL ACTIVITY OF SILVER NANOPARTICLE-IMMOBILIZED COTTON WOVEN FABRIC

Imana Shahrin Tania^{1,*}, Mohammad Ali², Riyadh Hossen Bhuiyan³

1 Department of Wet Process Engineering, Bangladesh University of Textiles (BUTEX), Tejgaon, Dhaka 1208, Bangladesh

2 Department of Mechanical Engineering, Bangladesh University of Engineering & Technology (BUET), Dhaka 1000, Bangladesh

3 Fibre and Polymer Research Division, Bangladesh Council of Scientific & Industrial Research (BCSIR), Dhaka-1205, Bangladesh

*Corresponding author. E-mail: imanat008@gmail.com

Abstract:

The purposes of the current research were to deposit the silver nanoparticles on the surface of a textile woven fabric and evaluate their dyeing performance and antibacterial activity. The synthesis of silver nanoparticle (Ag^0) is done by the in situ method. Strong alkali is used to improve functionality of cellulose before the application of silver nitrate salt ($AgNO_3$). The silver nanoparticle is formed by reduction of ascorbic acid. Various instrumental analyses are done to prove the formation of nanoparticles on the fabric surface. The morphology of nanodeposited fabric is characterized by using scanning electron microscope (SEM), elemental composition is done by energy dispersive spectroscopy, and crystallinity of nanoparticles is obtained by X-ray diffraction (XRD). Nanodeposited fabric is then dyed with direct dyestuff (Direct Red-89). Fourier transform infrared spectroscopy analysis is done to explore the bonding phenomena of un-dyed and dyed fabrics. The dyeing performance and antibacterial activity are examined on the colored fabric to investigate the dyed fabric quality after nanoparticle deposition. Results demonstrate the improvement of 54% of color strength and 11% of dye exhaustion with excellent antibacterial activity.

Keywords:

Nanoparticle, dyestuff, cotton, silver, color strength

1. Introduction

Cotton is the most plentifully used cellulosic fibers in the clothing industry due to its biodegradability, comfortability, hydrophilicity, smoothness, and notable coloration. Three hydroxyl groups per repeating unit of this fiber are responsible for the chemical reactivity, such as chemical modification, coloration, and chemical finishing [1]. In recent times, several studies have been carried out on cotton-based textiles to improve the existing properties and impart additional and innovative functional properties, such as antibacterial; UV protection; self-cleaning; anti-pilling; and softness [2-4]. By this way, the textile clothing sector can cope with the ever-growing consumer demands for hygienic, protective, and active wear clothing as well as it increases potential applications and value addition. On the other hand, nanotechnology is one of the upcoming sectors where lots of research works are being done with the goal of improving/altering properties of the textile materials [5]. It is an art and science of manipulating matter in a nanoscale to create new and unique materials and products. Nanoparticles are clusters of atoms with a size range of 1–100 nm [6] and show unique physical and chemical properties that would not be exposed by the bulk material [7-9]. The prime use of nanoscience in textile finishing is coating with various metallic nanoparticles in different methods for obtaining some additional properties that cause value addition on cloth. Silver is the popular nanoparticles used in cotton as it has some inherent antimicrobial properties. So many research works are done on nanofinishing for the goal to achieve antimicrobial property and ensure health protection [10,11]. However, the

dyeing properties of nano-Ag-immobilized fabric are still very little defined. Therefore, this research work is carried out to impart some essential properties on cotton fabric by using silver nanoparticles. There are many methods for treatment of cotton fabric with nanoparticles, such as padding, spraying, screen printing, and in situ.

Among these methods, the in situ generation of silver nanoparticles in textile fabric is a predominantly competent approach on the work of other researchers [12-14]. Haji et al. in 2013 reported better antibacterial property of plasma-treated cotton fabric via in situ synthesis of Ag nanoparticle [15]. Silver (Ag) nanoparticle is also synthesized from $AgNO_3$ salt and aloe vera by in situ reduction process to obtain ultraviolet protection and antibacterial activity on cotton fabric [16]. In situ generation of Ag nanoparticles causes sufficient deposition of particle by avoiding the separate reaction time [17,18].

In view of all information, the in situ method has been followed in this work for direct formation of nanoparticles on cotton surface. Here, at first, silver ions (Ag^+) get deposited on cotton via weak hydrogen bonds and Van der Waals bonds, and finally reduce to nanoform by the action of the reducing agent. Subsequently, the nanoparticles get cotton fabric as a template for instant immobilization. However, the nanosilver deposition has some significant effects on dyeing performances of the treated fabric. The current study explores the color performance of nano-treated and dyed cotton woven fabric including antibacterial activity.

2. Experimental

2.1. Materials

2.1.1. Fabric

A gray fabric sample of 100% cotton (count: 24, GSM: 140, ends per inch: 75, picks per inch: 72) with plain weave structure is collected from Nomam Textile Mills Ltd., Bangladesh. The fabric is then subjected to single-stage scouring before application of nanoparticles. For one bath scouring, 1.0 g/l of nonionic detergent and 2.0 g/l of sodium carbonate are mixed in water and boiled at 90°C for 50 min maintaining 11.5 pH.

2.1.2. Chemicals

The chemicals required for the purpose of nanoparticle synthesis are silver nitrate (AgNO_3), sodium hydroxide pallets (NaOH), ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$), and acetic acid (CH_3COOH). These are purchased from Merck, Germany. Additionally, for dyeing, CI Direct Red 89 (Megadirect Scarlet BNLE), leveling agent, and Glauber's salt ($\text{Na}_2\text{SO}_4 \times 10\text{H}_2\text{O}$) of analytical grade are collected from Orient Chem., Bangladesh. The chemical structure of CI Direct Red 89 is shown in Figure 1. It shows the

anionic nature in dye solution, which participates in bonding with nanoparticles.

2.2. Methods

2.2.1. Preparation of nanosilver-deposited fabric

Nanoparticles are deposited on the fabric surface directly by the in situ process. Silver nanoparticles are synthesized by reduction of AgNO_3 salt with ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) as shown in Figure 2. Initially, the fabric is subjected to alkali treatment in slack condition for surface activation. This enables the cotton fabric to absorb and formation of silver nanoparticles. This treatment is done on 3.0 g fabric dipping in 100 ml of 1.0M aqueous solutions of NaOH for 5 min at room temperature. The fabric is then taken away from bath and washed extensively. The alkali-treated fabric is instantly immersed into the 0.01M AgNO_3 aqueous solution for 1.0 h. Then, it is rinsed well with water. Silver nanoparticles are then synthesized by immersing fabrics into the 150 ml aqueous solution of $\text{C}_6\text{H}_8\text{O}_6$ (0.01M) for 45 min. The sample is afterward removed from the bath, rinsed with water, and then immersed in aqueous acetic acid at room temperature and dried at 60°C.

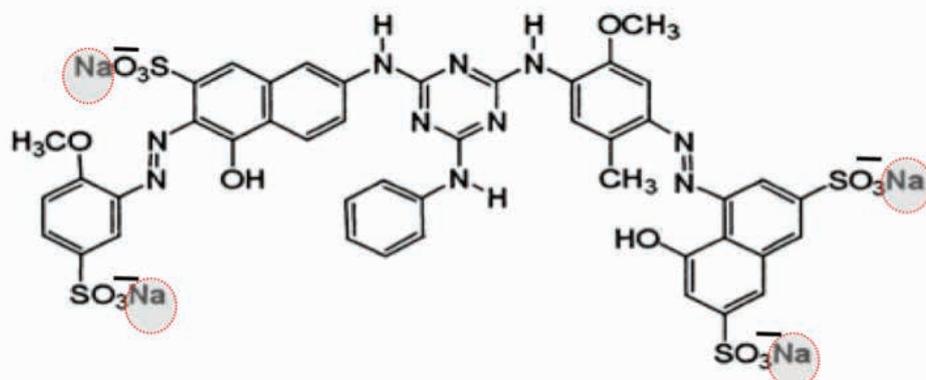


Figure 1. CI Direct Red 89.

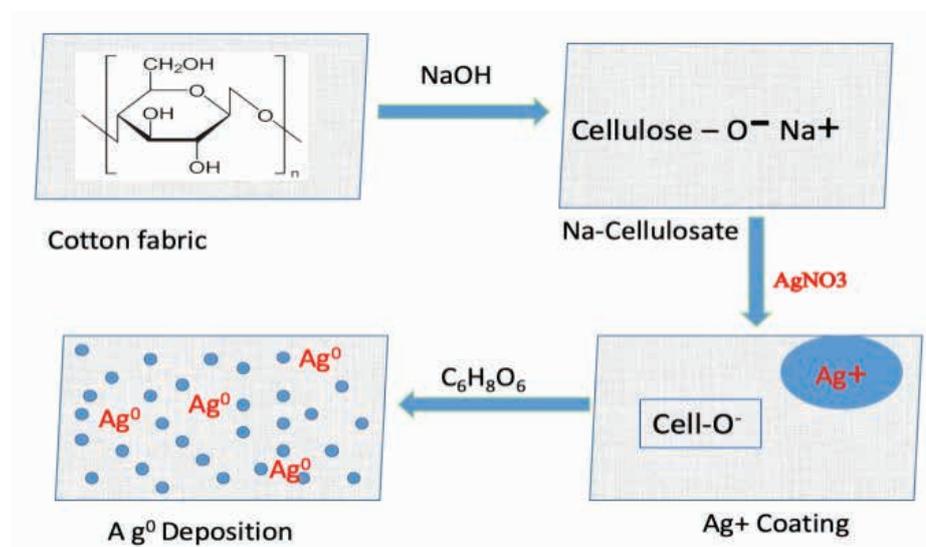


Figure 2. In situ synthesis and deposition process of Ag nanoparticles on cotton fabric.

2.3. Analysis and measurement

2.3.1. Scanning electron microscopy (SEM)

The surface morphology of the silver-deposited fabric is observed by a scanning electron microscope (SEM) that produces images of a sample by scanning the surface with a focused beam of electrons. Field emission electron microscopy (JSM-6700F, Tokyo, Japan) is used for this purpose. The fabric of nanosilver is well-washed with distilled water before the SEM test. It mainly reflects the morphology, size, and distribution of silver nanoparticles on the fabric surface. The presence of nanosilver is confirmed by elemental analysis of energy dispersive spectroscopy (EDS) using the same instrument as SEM.

2.3.2. X-ray dispersive (XRD) spectroscopy

The XRD pattern is determined by using X-ray diffractometer (Phillips, X'Pert Pro, Holland). It is used to find out the shape and size of nano-Ag form in the treated fabric surface.

2.3.3. Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) is used to check the chemical changes and bonding interaction of cotton fabric with nanosilver after dyeing. Both the untreated and treated dyed cotton fabrics are characterized by ATR/FTIR spectrometer (Model:Frontier, PerkinElmer, USA). The scanning area is in the range of 1000–4000 cm⁻¹.

2.3.4. Dyeing of untreated and nano-Ag-deposited fabrics

The dyeing behavior of Ag^o-deposited fabric is studied and compared with the dyeing behavior of the untreated fabric. Dyeing is performed with the instrument of laboratory scale applying the exhausted method. The bath maintains 1:10 material to liquor ratio, neutral to slightly alkaline pH, and 50 min time and 60°C for dyeing of 2.0% (weight basis) shade. Wetting agent and sequestering agent each of 1 g/l of dye solution are used, and Glauber's salt and soda ashes are used in the required amount.

2.3.5. Fabric performance test

For investigating the performances of nanosilver deposition in the fabric, dye exhaustion, color fastness, color strength, and antibacterial activity are measured. The antibacterial activity of Ag^o-deposited cotton fabric is tested by using the quantitative method (plate count agar method) following the test method of AATCC 100:2004 [19]. The exhaustion, color strength, and color fastness are determined for color characteristic analysis. The formula of exhaustion is shown in Equation (1) as

$$\text{Exhaustion \%} = [(C_0 - C_s) / C_0] \times 100 \quad (1)$$

where C₀ is the initial concentration of dye in the dye bath and C_s is the concentration during the dyeing process. The color strength of the fabric is calculated by Equation (2) as described in the Kubelka-Munk theory [20].

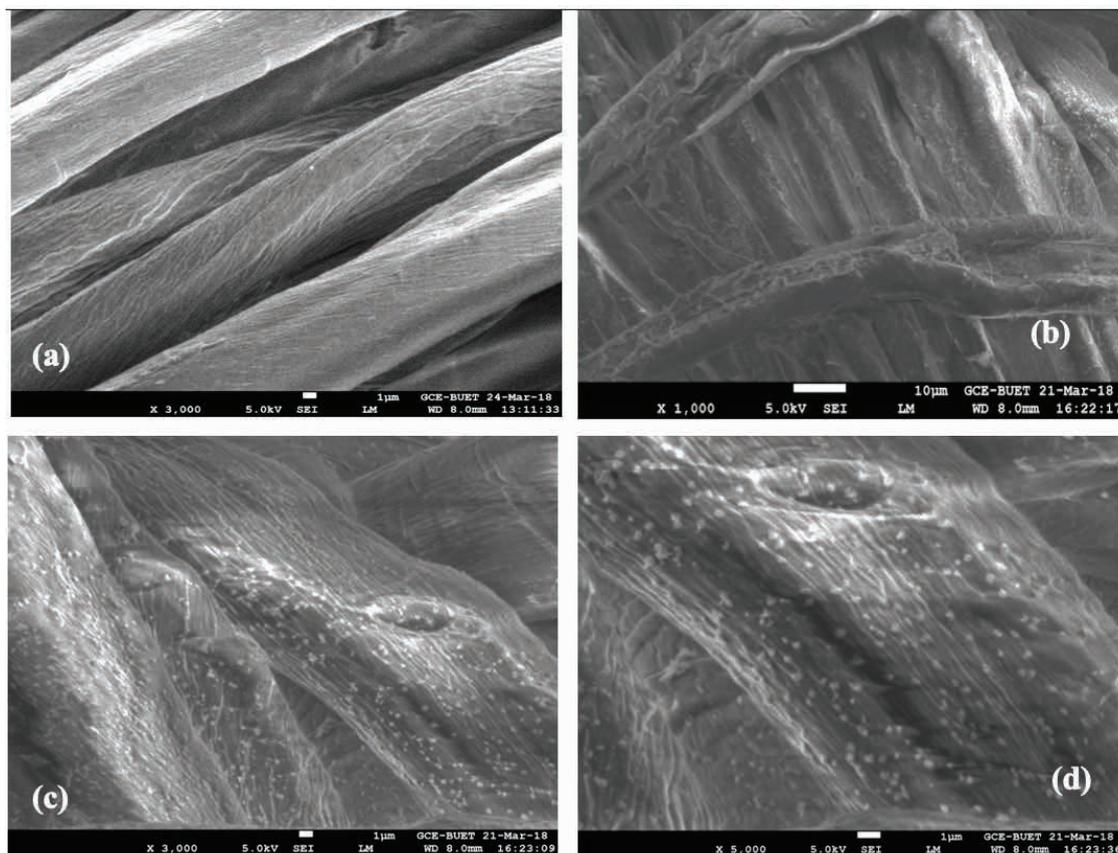


Figure 3. FE SEM image of (a) untreated and (b–d) Ag^o-deposited fabric on x1000, x3000, and x5000 magnifications. FE, field emission; SEM, scanning electron microscopy.

$$K / S = [(1 - R)^2 / 2R] \tag{2}$$

where *R* is the reflectance, *K* is the absorbance coefficient, and *S* is the scattering coefficient. The color fastness to wash, rubbing, and light is measured by the test method called ISO:105-CO4, ISO:105-X12, and ISO:105-B02, respectively.

3. Results and discussion

3.1. Surface morphology by SEM analysis

The existence and intensity of nanodeposition on the treated fabric are investigated by taking the SEM images in different magnifications and placed in Figure 3(b)–(d). The SEM image of the untreated fabric is also taken and placed in Figure 3(a) for better understanding and comparison. These images represent the larger view of the fiber and enable to show the deposition of nanoparticles. The images show that nanoparticles are distributed evenly through the whole surface area and no agglomeration of silver nanoparticles is found.

3.2. Energy dispersive spectroscopy

The EDS results of untreated and treated samples are shown in Figure 4. The untreated fabric shows C (carbon) and O (oxygen) as characteristics peaks of cellulose as found in Figure 4(a). After nanosilver deposition, a new peak appears at 3 keV, which attributes to the signal of silver as shown in Figure 4(b). Both EDS and the SEM images have strong evidence of the formation and deposition of Ag° on the surface of cotton fabric. The elemental mass percentage of silver obtained from EDS is represented in Table 1. The table shows that the elements

for untreated fabric are carbon and oxygen, which are the basic elements of cellulose. The treated fabric shows carbon, oxygen, and silver as its elements. The elemental mass of Ag in the treated fabric is obtained as 1.58%. The effect of this nanodeposition is analyzed in the subsequent section.

3.3. X-ray diffraction analysis

The XRD pattern of Ag°-deposited fabric is shown in Figure 5. The figure shows that there are four diffraction peaks of Ag° deposition exhibited at 38°, 44.32°, 64.8°, and 78°. These sharp peaks confirm the formation of Ag° crystal on the fabric surface. It also confirms that silver ions have been reduced to nanometallic state inside the fabrics network. From the XRD data, the average crystalline sizes are determined by the Debye–Scherrer Equation (3) as follows:

$$D = \frac{K\lambda}{\beta \cos \theta} \tag{3}$$

where *D* is the average crystalline size, *K* is the crystalline shape factor (0.94), *λ* is the wave length of X-ray (0.15406 nm), *β* is the full width half maximum (FWHM), and *θ* is the Bragg angle in degree. The average crystalline size of Ag° in the fabric is about 26 nm, which is obtained from the peak analysis of Figure 5 using Equation (3).

3.4. Dyeing performance evaluation

The color strength (*K/S*) and the reflectance (*R*%) curve of untreated and nano-Ag-treated fabric obtained from the spectrophotometer are shown in Figure 6(a) and (b). The *K/S* value of the untreated fabric is 14.38, which lies in between wavelengths 450 and 550 nm as found in Figure 6(a), where as for the Ag°-treated fabric, the value of color strength

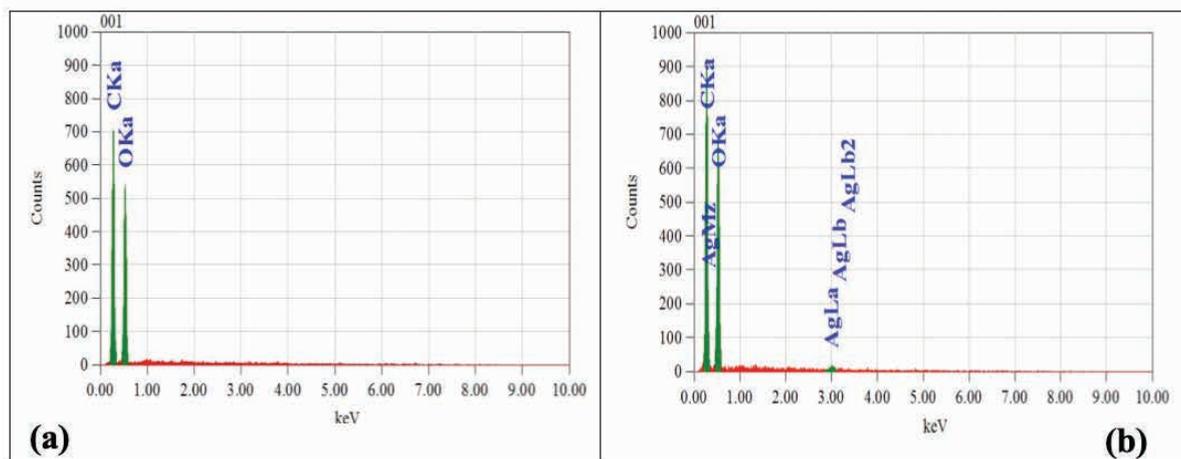


Figure 4. EDS spectrum of (a) untreated and (b) Ag°-treated fabric. EDS, energy dispersive spectroscopy.

Table 1. Elemental mass percentage of untreated and treated cotton obtained by EDS analysis

Sample	Elemental mass (%)		
	C (carbon)	O (oxygen)	Ag (silver)
Untreated	53.37	48.63	0
Ag° deposited	51.13	47.29	1.58

EDS, energy dispersive spectroscopy.

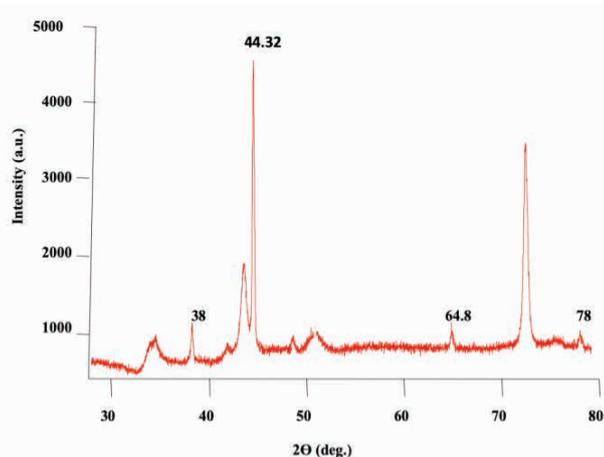


Figure 5. XRD pattern of Ag⁰-deposited fabric. XRD, X-ray diffraction.

rises to 22.13 and lies at the same wavelength as for the untreated fabric. The obtained results of color (*K/S*) strength and dye exhaustion are given in Table 2. The improvement of color strength and dye exhaustion of Ag⁰-treated fabric are determined by taking the value of untreated and treated fabrics. The Ag⁰-deposited fabric shows 54% increment in color strength. The dye exhaustion percentage of the treated fabric increases about 11%. The color fastness property of untreated and treated fabrics is given in Table 3. The table indicates that the Ag nanodeposition causes improvement in the fastness property. The treated fabric shows better fastness rating as found in washing fastness (shade change), dry rubbing, and light fastness. The other fastness grades and color staining are quite same with the untreated fabric.

In the present investigation for the improvement of dyeing performance, it can be pointed out that the direct dye is an

Table 2. Exhaustion and color strength of Ag⁰-deposited and dyed cotton fabric

Sample	Color strength (K/S)	Dye exhaustion (%)	Improvement of color strength	Improvement of dye exhaustion
Untreated fabric	14.38	80	–	–
Ag ⁰ -deposited fabric	22.13	89	54%	11%

Table 3. Color fastness of untreated and Ag⁰-deposited fabric

Sample	Wash fastness							Rubbing fastness		Light fastness
	Shade change	Staining						Dry rubbing	Wet rubbing	
		Acetate	Cotton	Nylon	Polyester	Acrylic	Wool			
Untreated fabric	2	5	2–3	4–5	4–5	4	3	2–3	2	3
Ag ⁰ -deposited fabric	3	5	3	4–5	4–5	4–5	3	3	2	3–4

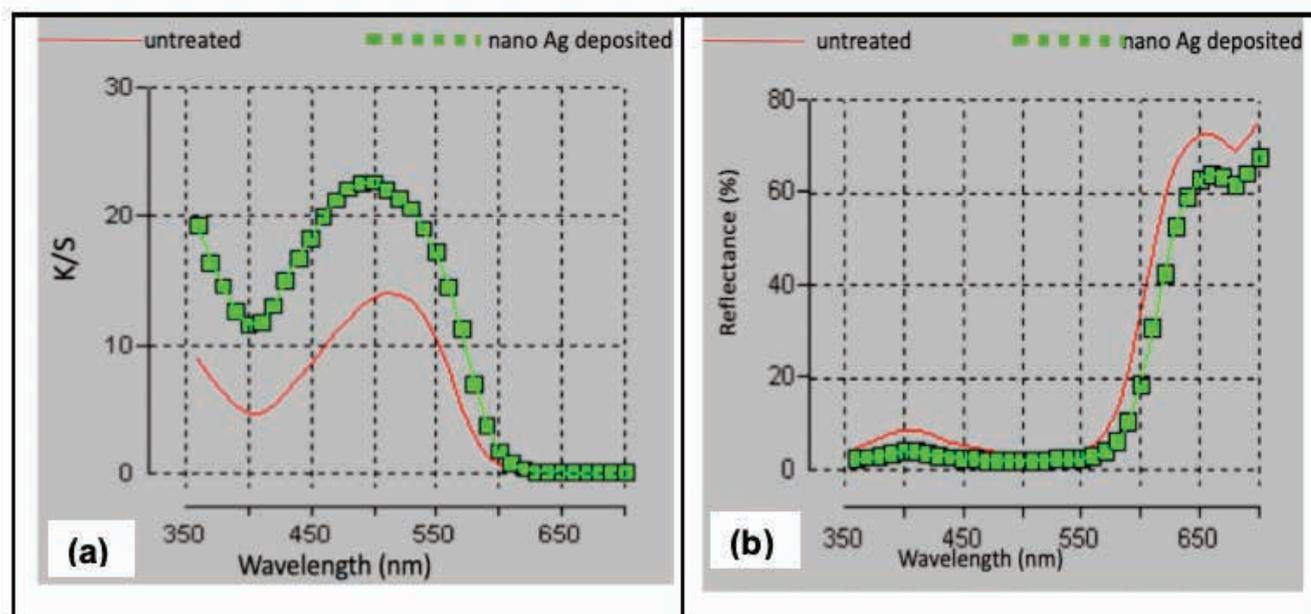


Figure 6. (a) Color strength (K/S) and (b) reflectance (%) curve of untreated and Ag⁰-treated fabric.

anionic dyestuff with low fastness property. Metallic salt is used for the improvement of the fastness property of the direct dye where the metallic molecules make a complex bond with the dyestuff and improve color fastness quality [21]. In case of Direct Red 89, several anionic sulfonate groups (SO_3^-) are presented as shown in Figure 1. The negatively charged dye anions get attracted toward the fiber due to the polarity formed by the Ag^+ on the fabric surface [22]. As a result, the dyestuff makes a strong bond with nanoparticles and causes the improvement of color strength, better fastness, and higher exhaustion for Ag^+ -immobilized fabric. The higher dye exhaustion indicates that Ag^+ has a significant role to reduce the dye wastage.

3.5. FTIR analysis of fabric

Internal bondings of untreated, nano- Ag -treated, and dyed both fabrics are investigated by taking the FTIR pattern as shown in Figure 7. This study reflects the bonding phenomenon of nano- Ag with cotton fiber and dyestuff. The wavelength $3000\text{--}4000\text{ cm}^{-1}$ is magnified for showing clearly the newly formed small peak on the dyed fabric. The graph indicates mainly the general characteristic peaks of cellulose fibers 1036 cm^{-1} (C–O–C stretching), 1317 cm^{-1} (C–O stretching), 1656 cm^{-1} (C=O stretching), 2906 cm^{-1} (–CH stretching), and 3338 cm^{-1} (O–H stretching). So nanodeposition does not change the chemical structure of the cellulose fiber. It only causes the physical deposition of nano- Ag on the fabric surface. On the other hand, the pattern of untreated dyed fabric is quite different where a new dyestuff is added with the cellulose fiber. The pattern of nano- Ag -treated and dyed fabric shows some new shapes of wave and the peak intensity of curve is also different. A clear peak is found at 1646 cm^{-1} , which is for the

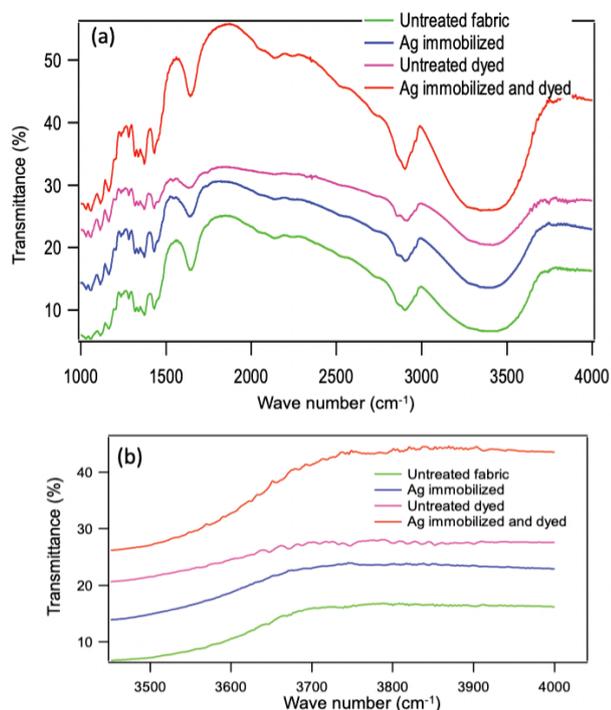


Figure 7. FTIR spectra of untreated fabric, Ag^+ -immobilized fabric, untreated and dyed fabric, dyed fabric with nano- Ag . (a) Wave number $1000\text{--}4000\text{ cm}^{-1}$ and (b) wave number $3000\text{--}4000\text{ cm}^{-1}$ after magnification. FTIR, Fourier transform infrared spectroscopy.

C=C stretching, and 2200 cm^{-1} , which is for the C≡N stretching. The dyed and Ag^+ -treated fabrics show many small peaks from $3500\text{ to }4000\text{ cm}^{-1}$ that is visible after magnification in Figure 7(b). This is assigned for S=O bond from SO_3^- function of Direct Red [23]. The displacement of peaks is observed in the dyed fabric for precipitation of the dyestuff.

3.6. Antibacterial properties

The silver nanoparticle is a potential and popular antimicrobial agent. The activity of nanoparticles depends on the size, shape, form, and application methods. In this study, the nanosilver is deposited by the in situ method and the Ag^+ -deposited fabric shows excellent bacterial reduction against both Gram-positive and Gram-negative bacteria as shown in Figure 8. The bacterial reduction is given with laundering durability, where the treated sample is subjected to 15 home laundering for the longevity test of modified fabric. The performance of cotton woven fabric against both bacteria is excellent on washing and shows 88% reduction of bacteria for *Staphylococcus aureus* and 80% for *Escherichia coli*. The activity reduces gradually after washing with the increase in the washing cycle. The results of antimicrobial tests shown in Figure 8 indicate that the silver nanodeposited fabric is more effective against Gram-positive bacteria compared with Gram-negative bacteria. Additionally, it can be found that the antibacterial activity of present Ag^+ -deposited fabric is better compared with the results of our previous work on mechanical deposition of Ag^+ in fabric by the pad-dry-cure method [24].

4. Conclusions

An experimental investigation is performed on the performance of nanoparticles for improvement of color strength, dye exhaustion, color fastness, and antibacterial property. The well deposition of nanoparticles through the in situ method is ensured by the surface characterization test, SEM, and elemental composition analysis EDS. Well distribution and nanoparticles of size 26 nm are confirmed by the XRD spectrum. The FTIR analysis shows no significant change in cellulosic fiber bonding

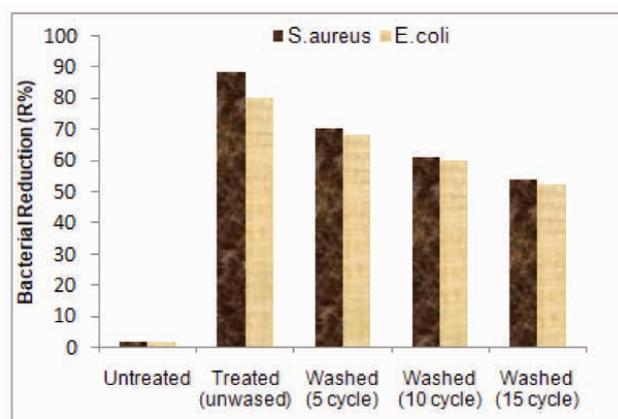


Figure 8. Bacterial reduction (R%) with wash durability of Ag^+ -deposited fabric.

due to nano-Ag deposition. In results, the silver nanodeposited fabric shows 54% improvement of color strength and 11% of dye exhaustion and slightly improved color fastness. The treated fabric also shows excellent bacterial reduction ability such as 89% for *S. aureus* and 80% for *E. coli*. Since direct dye is cheaper than other cellulosic dyestuff, the present process of silver nanoparticle deposition might be more useful for direct dyed fabric. Thus, a cheap cellulosic dyestuff can be popular on nanosilver-deposited fabric. Besides, Ag^o deposition saves dyestuff cost by improving dye exhaustion and color fastness.

Abbreviations

SEM, scanning electron microscopy; EDS, energy dispersive spectroscopy; XRD, X-ray dispersive spectroscopy; FTIR, Fourier transform infrared spectroscopy; *R*%, reduction percentage of bacteria; *K/S*, color strength; %*T*, transmittance percentage.

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