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Simultaneous Dyeing and Antibacterial Finishing of Nylon Fabric Using Acid Dyes and Colloidal Nanosilver

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Abstract
The simultaneous finishing and dyeing of nylon fabric were investigated through exhaustion using acid dye and colloidal silver nanoparticles to obtain colored nylon with antimicrobial properties. The antimicrobial property of the fabrics was evaluated against two pathogenic bacteria including E. coli and S. aureus. The durability of antibacterial properties and the colour coordination of the fabrics treated against washing were investigated. In addition, some properties of the fabrics treated, including the surface morphology, and XRD and EDX diagrams were reported and discussed.

Key words: *silver nanoparticles, nylon fabric, antimicrobial, dyeing.*

Introduction

Silver nanoparticles can be used in extensive applications, such as catalytic, electrical, optical and antibacterial [1, 2]. Various methods have been employed to synthesise silver nanoparticles including chemical, irradiation, biochemical, and water/oil microemulsion. Physical and chemical properties of silver nanoparticles such as the size, morphology and stability are influenced by experimental conditions [1, 3].

Silver nanoparticles, with their unique chemical and physical properties, are proving to be an alternative for the development of new antibacterial agents. They have also found various applications in the form of wound dressings, coatings for medical devices and impregnated textile fabrics, among others [4, 5]. Additionally silver nanoparticles have been applied in textile by various methods e.g. silver nanoparticles can be added to polymers through spinning, used as a master batch [6, 7, 9] or coated on fabrics [8, 10]. Different methods have been used for the coating of silver nanoparticles on various fabrics [8, 11]. On the other hand, the wash fastness of silver nanoparticles is poor and needs to be solved using a cross-linking agent [12]. Chitosan was co-applied on cotton fabric to improve the laundry durability of antimicrobial treatment by the formation of a covalent bond between the cross-linking agent, antimicrobial agent and cellulosic chains [13, 15]. Furthermore the dyeing of cotton and cotton/polyester fabrics in the presence of silver nanoparticles with a binder exhibited excellent antibacterial activity against *E. coli*, *S. aureus* and the fungus *C. albicans* [14, 18]. Also the laundering durability and colour change of cotton fabrics have been investigated [16, 17, 19, 20]. Wool fabric was treated with sulfur nano silver colloidal solution (SNSE) with Ag/S complex to obtain various functionalities such as moth-proofing, as well as antibiotic and antistatic properties [21, 22]. Moreover silk applied with silver nanoparticles through exhaustion showed 100% antimicrobial activity against Gram-positive bacterium [23]. Furthermore PA6 dyed with reactive dye produced an antimicrobial effect with nano silver, showing high antimicrobial effectiveness; but it disappeared after 10 washings, with colour change as a result of the nano silver. Cotton fabric dyed with vat dye compared with treatment with vat dye/silver and wool fabric dyed along with nanosilver showed antibacterial properties and colour difference [24 - 28].

In this study nylon fabrics were dyed with acid dyes in the presence of various silver nanoparticle concentrations. The influence of colloid nanosilver concentration on antibacterial properties, colour change, and laundering durability of nylon fabrics in the presence of different acid dyes was studied.

Materials and methods

100% knitted nylon fabric of 85 g/m^2 was used. A colloid solution of nano silver with a concentration of 10,000 ppm and 60 nm average particle size was supplied by Nano Group Co. (USA). Red (C.I.Acid Red 88), blue (C.I.Acid Blue 7) and yellow (C.I.Acid Yellow 11) dyes were prepared (***Figure 1***) by the Guangdong Company (China).

Apparatuses

A Philips x-ray diffractometer, model X PERT MPD (PANALYTICAL Co., the Netherlands), was used to assess the crystalinity of silver nanoparticles on the nylon fabrics. A spectrophotometer - Varian Carry 5000 (VARIAN Co., Australia) was employed to obtain the UV-VIS absorbance spectrum of the fabrics treated. An XL30 Philips scanning electron microscope (FEI Co., the Netherlands) was applied to study the surface morphology of samples and obtain EDS spectra.

Colour coordinates

Three indices of the CIELAB colour system were taken using a ColorEye 7000A calorimeter (X-Rite Gretag Macbeth Co., USA). The CIE LAB color system is widely used in the colour measurement of textiles. In this system L* shows the lightness of the fabric, and a* and b* indicate red-green (redder if positive, greener if negative) and yellow-blue colors (yellower if positive, bluer if negative), respectively. The whole of the colour difference between untreated and treated nylon samples can be indicated by the term ΔE, which was calculated based on ***Equation 1*** [25].

$$\Delta E=[(AL^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{0.5} \quad (1)$$

Methods

Scouring

The nylon samples were washed in a bath containing 1 g/l of detergent with L:G = 50:1 (liquor to good result) at 60 °C for 20 min, then scoured with water and dried at room temperature.

Dyeing

The nylon samples were dyed with acid dye (2% o.w.f), acetic acid (2% o.w.f),

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and different concentrations of nanosilver (100, 200, 400, 500 ppm). The liquor to goods ratio (L.R.) was 50:1 for all specimens. The nylon samples were added to a dye bath at 40 °C and left for 5 min. Then acetic acid and nanosilver were added and the nylon samples treated for 10 min. Acid dye was added to the dye bath and temperature was increased to boiling within 15 min and maintained thereat for 45 – 60 min. The samples were finally washed and dried at room temperature. The pH of the dye bath was dependant on the concentration of nanosilver, which was 5 – 5.5 for the bath (shown on ***Figure 2***). In the samples treated that they were dyed only, we did as above without using nano silver, and in the undyed samples we used only nano silver for disinfecting them.

Nano Ag treatment

In this procedure, two different methods were applied for preparing dyed and undyed fabric containing silver nano particles. First a bath containing silver nanoparticles was prepared and then nylon fabric was added to it, with the process temperature initially set at 40 °C and then gradually raised to boiling point after 20 min. Secondly dyed fabric was achieved by preparing a dye bath and then silver nanoparticles were added to it. Nylon fabric was soaked in the bath, with the process temperature initially set at 40 °C and then gradually raised to boiling point after 20 min. The baths were then boiled for 30 min along with the gradual adding of acetic acid to the bath. The samples were thoroughly rinsed with water, squeezed and dried at room temperature. The fabric was treated with various concentrations of silver nanoparticle (100, 200, 400, 500 ppm).

Antibacterial test

There were two antibacterial organisms tested: *S. aureus,* American type culture collection NO.6538, as a Gram positive bacterium, and *E.coli*, American type culture collection NO.11303, as a Gram negative bacterium. Stocks of these bacteria freeze dried were suspended in tryptis soy broth (TSB) (Antec diagnostics) and incubated at 37 °C for 24 h and then transferred to plates of nutrient Agar to be preserved for examination.

Bactericidal test: the antibacterial activity of the treated nylon colonies of each bacterium was suspended in a physiologic saline solution (NaCl 0.9% in distilled water at pH 6.5) with a concentration of

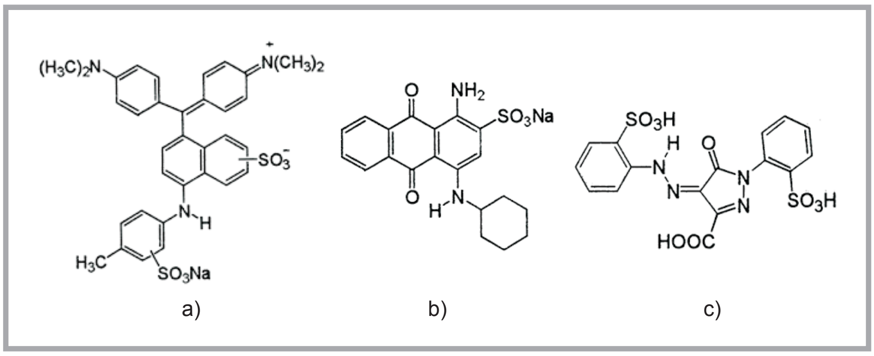


Figure 1. *Molecular structure of dyes: a) red (Red A (804): C.I.Acid Red 88, $C_{20}H_{13}N_2NaO_4S$), b) sky blue (Sky blue A: C.I.Acid Blue 7, $C_{37}H_{35}N_2NaO_4S_2$), c) light yellow (Light yellow 2G: C.I.Acid Yellow 11, $C_{16}N_{13}N_4NaO_4S$).*

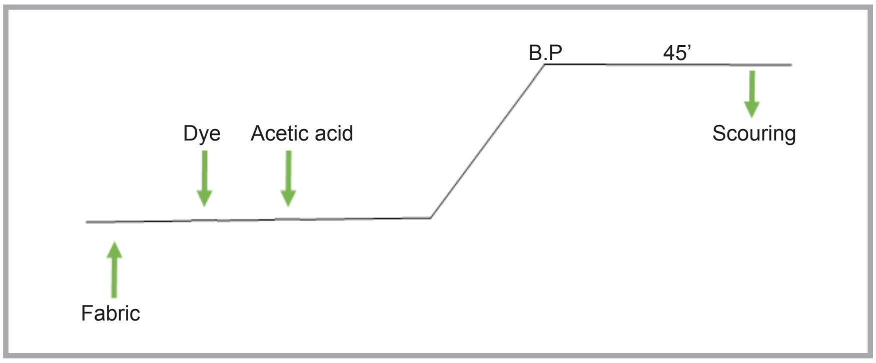


Figure 2. *Diagram of nylon dyed with acid dye.*

0.5 McFarland. The bacterial suspensions were then incubated with agitation at 37 ± 2 °C, 220 r.p.m. for 2 h, and a homogenous suspension of bacteria was prepared. Then serial dilution was done in 5 steps (Dilution of 1:100,000) and a concentration of about 1.5 - 2×10^3 CFU/ml was applied for antibacterial testing. Bacteriological culture tubes (125 × 17 mm glass tubes) containing one piece of nylon fabric (10 ×10 mm) were sterilised by an autoclave device in moisturized heat (121 °C, 15 psi) for 15 - 20 min. An aliquot of 1 ml bacterial suspension and 2 ml TSB broth was then added to every tube and 3 ml in each tube was detected to ensure that any decrease in the bacterial count was due to exposure to nylon fabrics. One control of the saline solution with TSB broth, and one control of aliquot with untreated fabrics including the tubes containing treated nylon fabrics with bacterial suspensions and control tubes were incubated at 37 °C for 24 h. Then samples of 10 µl were taken from each tube and counted by the pour plate method. In this method, samples are mixed with melted agar that decreases in temperature at 45 °C and is then poured. The plates were incubated at 37 °C for 24 h and the colonies of each plate were counted by a colony counting device to compare and determine the bacteria reduction of the suspensions. The results of the numbers before and after treatment with nylon fabrics were used to determine the bacteria using ***Equation 2***:

$$\text{Reduction rate} = 100(A – B)/A \text{ in } \% \quad (2)$$

where, R is the bacterial reduction ratio, A the number of bacterial colonies from untreated fabrics, and B is the number of bacterial colonies from the treated fabrics.

Washing fastness

Washing fastness was conducted according to the AATCC 61(2A)-1996 test method, in which washing is equivalent to five launderings at the medium or warm setting in the temperature range of 38 ± 3 °C. This test was used to investigate the stability of nanoparticles on the surface of the polyamide after 5 washes.

Results and discussions

Antimicrobial properties of samples treated

Nylon is produced from diamine and dicarboxylic acid. Polyamide 6 is the most important polyamide for the commercial

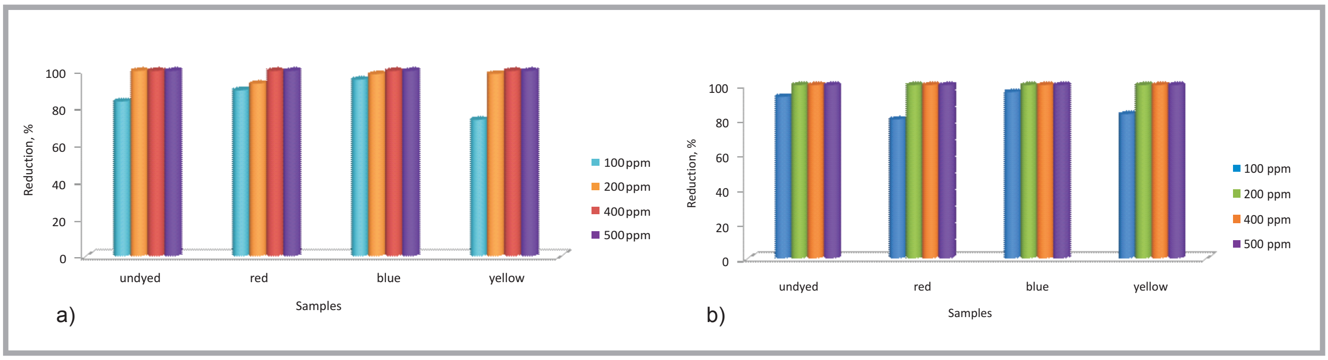


Figure 3. *Antibacterial reduction percentages for raw sample and samples treated with diverse concentration against: a) E. coli and b) S. aureus.*

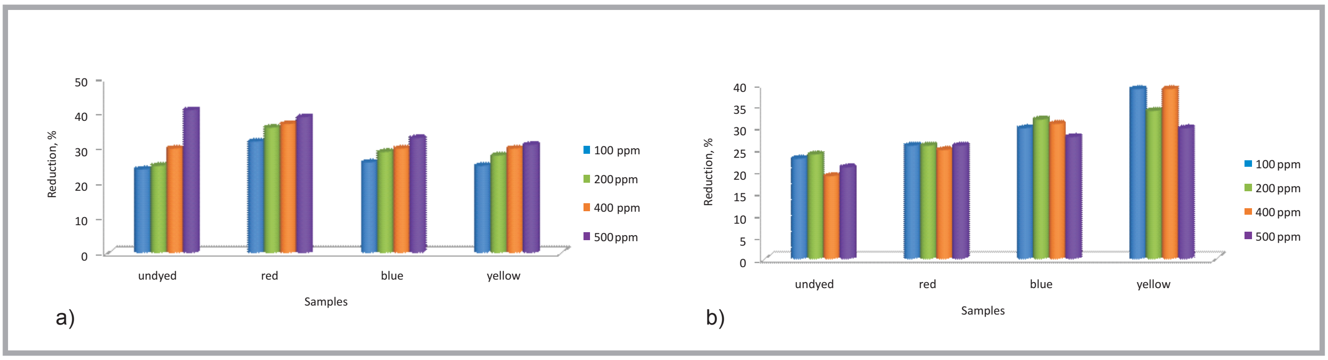


Figure 4. *Antibacterial reduction percentages for raw sample and samples treated with diverse concentrations after 5 laundering cycles against: a) S. aureus and b) S. aureus.*

production of fibres and resins. In this procedure, Nylon samples were treated with four diverse concentrations of silver nanoparticles in the dye bath and the antimicrobial activity of the treated samples against two well-known bacteria was evaluated. *E. coli* (Gram-negative) which is one of the general micro organism that can be selected for antimicrobial tests and is resistant to common antimicrobial agents as well as *S. aureus* (Gram positive) bacteria, which is the major cause of disease in a hygienic environment.

Antibactericidal effects of the samples treated with silver nanoparticles were observed against *S. aureus* and *E. col*i (***Figure 3***), and efficient antibacterial properties were achieved due to the presence of nanoparticles on the surface of the fabric. This could be related to the attraction of silver to the protein sites of wool. Subsequently the antibacterial properties of each sample were assessed after five cycles of washing (***Figure 4***), and the antibacterial properties of the fabric reduced with repeated washings. The stability of nanoparticles was not efficient after the washing process, and a dramatic decrease was observed. Even higher amounts of nanoparticles did not lead to significant improvements in the antibacterial properties of samples. It seems a high amount of silver was released from the surface of the samples treated. Therefore it was supposed that the after-treatment process is essential after simultaneous dyeing and finishing with silver nanoparticles, and several methods have been suggested for this matter.

Both bacteria showed different resistance against silver nanoparticles. The differential response/susceptibility of bacteria towards the fabric treated is higher for *S. aureus* as a Gram-positive than that of *E. coli* as a Gram-negative, which corresponds to the thick peptidoglycan layer of the Gram-positive cell wall.

Table 1. *Colour indices of untreated and treated samples.*

Samples		L*	a*	b*	ΔE
Undyed	Audience	76.73	2.37	-6.30	-----
	1	76.72	2.33	-5.26	0.906
	2	75.27	1.91	-5.29	1.83
	3	78.77	2.21	-5.25	2.29
	4	74.53	0.72	-5.26	2.94
Red	Audience	34.32	51.76	26.14	-----
	1	34.14	52.55	27.35	11.64
	2	35.73	53.81	27.14	10.28
	3	39.05	53.95	21.77	4.10
	4	39.53	52.23	19.41	3.48
Blue	Audience	44.78	-34.70	-28.80	-----
	1	47.24	-34.70	-27.90	2.60
	2	49.10	-35.40	-26.90	4.75
	3	51.17	-36.40	-26.20	7.05
	4	51.90	-36.70	-26.60	7.63
Yellow	Audience	63.03	20.09	78.49	-----
	1	62.96	20.06	79.64	1.15
	2	63.98	21.98	79.85	2.51
	3	64.87	21.59	81.91	4.15
	4	66.64	22.20	81.81	4.25

Colour coordinates

In order to evaluate the colour coordinates of the samples treated, three indices of the CIE Lab colour system were measured (***Table 1***). The concentration of the dye was constant, hence the colour differences can be related to the concentration of nano silver. It seems that increasing the amount of silver nano particles leads to an increase in ΔE. On the other hand, the deposition of silver nanoparticles on white samples reduces the colour change, which cannot be visually detected; the ΔE of white samples dyed with nanoparticles were below 2.3, and an ΔE value of around 2.3 corresponds to a just noticeable difference (JND). This correlation is, however, quite approximate, and there are significant variations in the visual JND over colour space. As expected, the deposition of silver nanoparticles led to a color change irrespective of the order of silver loading and dyeing, which could be visually detected since in both cases the colour difference (ΔE) was higher than in silver loading after dyeing, with an almost doubled colour difference compared to the results obtained for the opposite order of operations. Thus according to the results of colorimetric measurements, it is suggested that the loading of silver nanoparticles after dyeing should be avoided because of the considerable colour change.

XRD pattern

XRD patterns of the samples containing nano silver at a 500 ppm concentration are indicated in ***Figure 5*** (a - undyed, b - red, c - blue, d - yellow). All evident peaks on the 2θ scale are about 44.56, 51.9, 76.48 and 93.21, showing the (1,1,1), (2,0,0), (2,2,0) and (3,1,1) i.e. Bragg`s reflections face centered cubic (fcc) phase structure of silver nano particles. These results are in good agreement with earlier findings [27]. The X-ray diffractometer (XRD) pattern shows a peak of Ag for the sample treated with 500 ppm nanosilver, which is good enough to prove the crystalline formation of nanosilver on the nylon surface in ***Figure 5*** (a - undyed, b - red, c - blue, d - yellow).

SEM micrographs

The SEM images of nylon fibres before and after the deposition of silver nanoparticles are shown in ***Figure 6***. The images in ***Figure 6.a*** demonstrate the smooth structure of the nylon fabric before being treated with silver nanoparticles. The silver nano particles absorbed were observed on the surface of fibres.

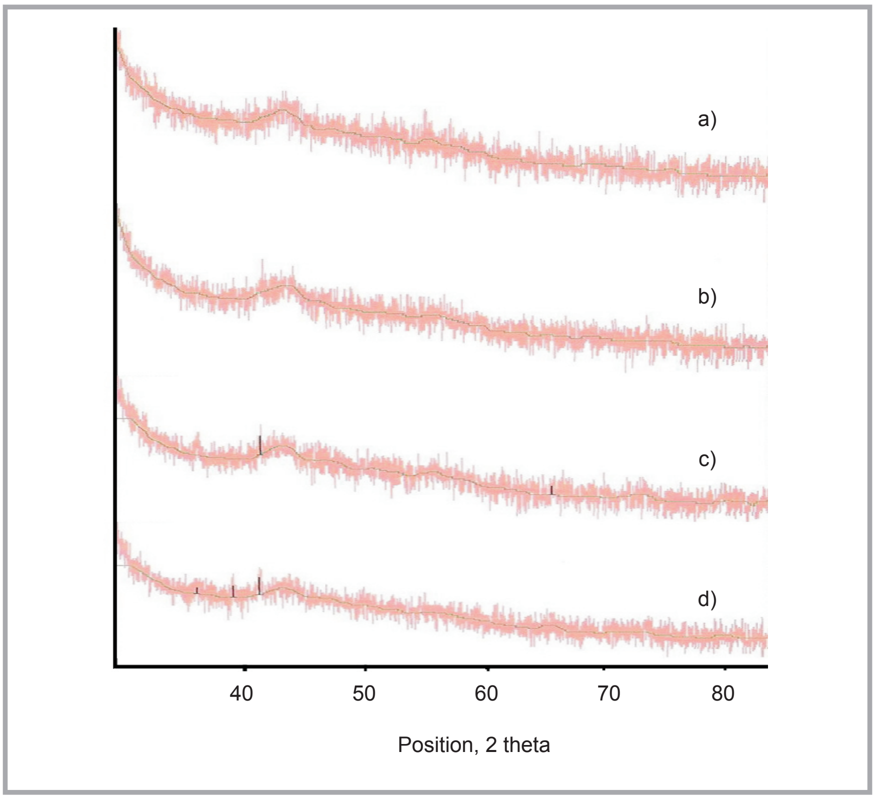


Figure 5. *XRD spectrum of samples containing silver nanoparticles on the top; a) undyed, b) red, c) blue, d) yellow.*

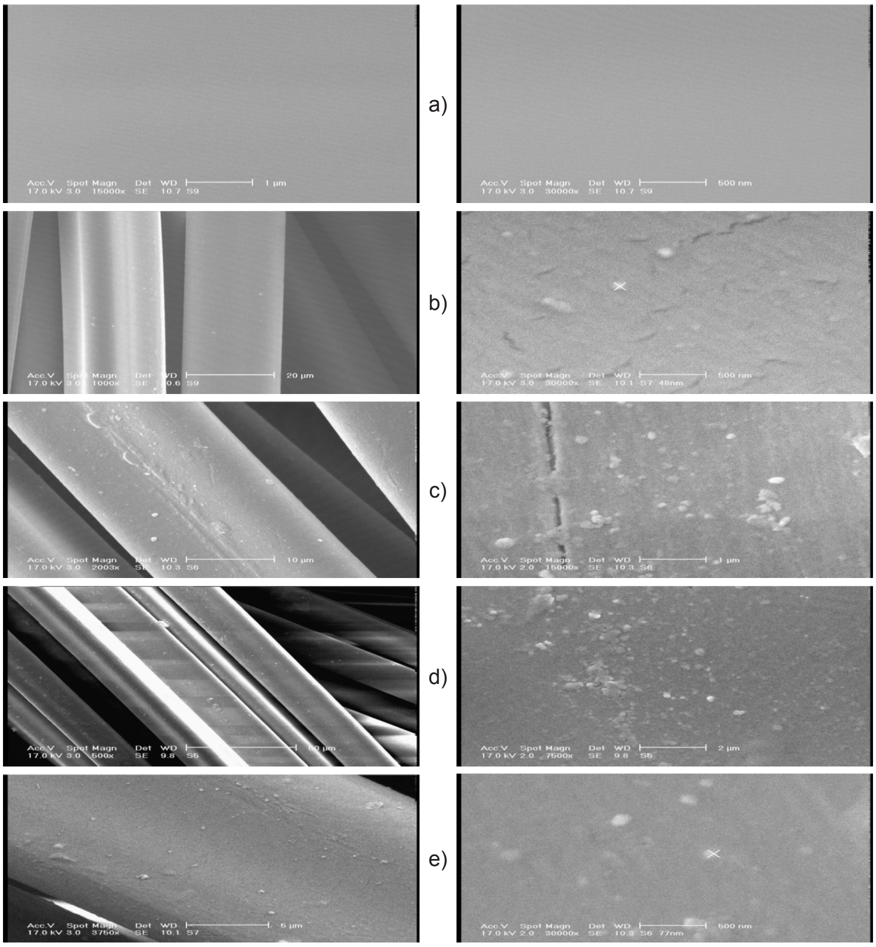
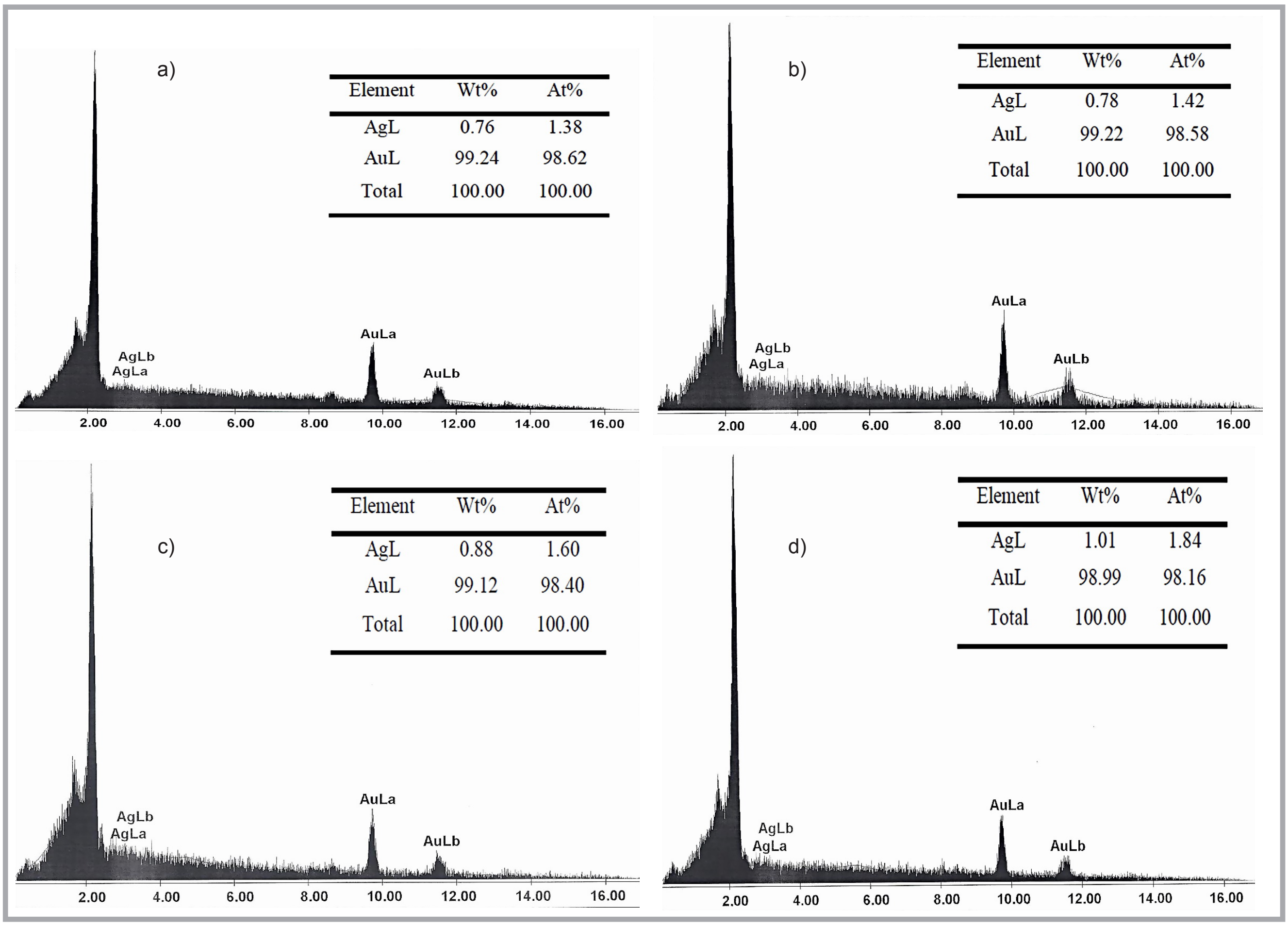


Figure 6. *SEM images of samples: a) untreated, b) undyed, dyed fabrics contain silver nanooparticles, c) red, d) blue, e) yellow.*



a)

Element	Wt%	At%
AgL	0.76	1.38
AuL	99.24	98.62
Total	100.00	100.00

b)

Element	Wt%	At%
AgL	0.78	1.42
AuL	99.22	98.58
Total	100.00	100.00

c)

Element	Wt%	At%
AgL	0.88	1.60
AuL	99.12	98.40
Total	100.00	100.00

d)

Element	Wt%	At%
AgL	1.01	1.84
AuL	98.99	98.16
Total	100.00	100.00

Figure 7. *EDAX images of samples: a) undyed, b) red, c) blue, d) yellow.*

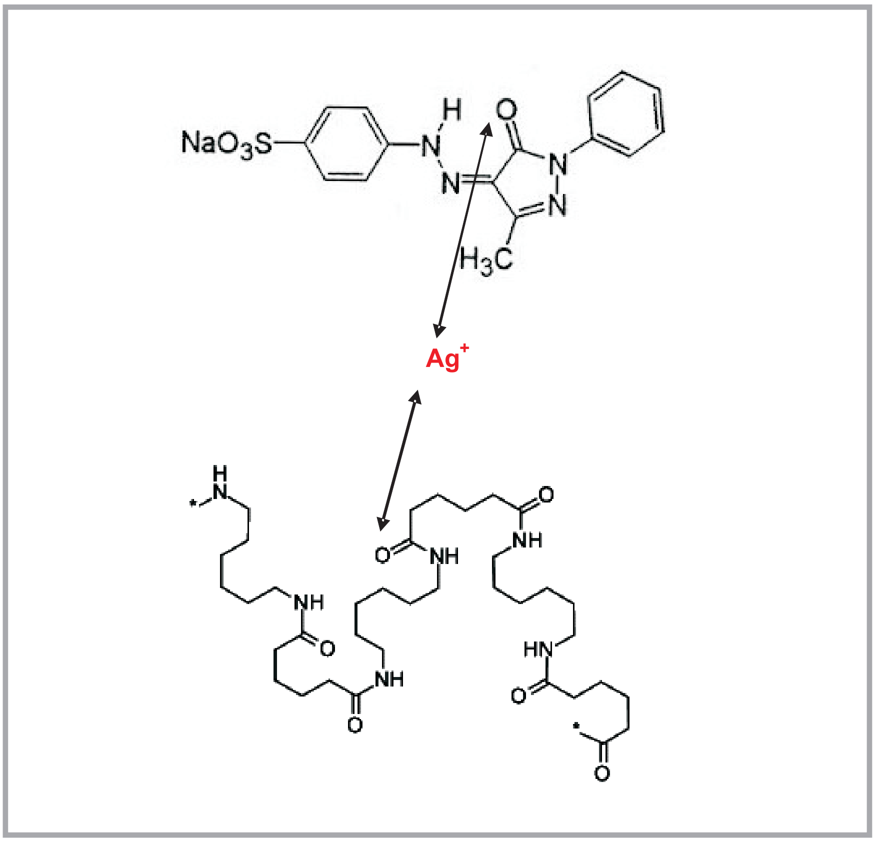


Figure 8. *Formation of complex by adding silver ion.*

The aggregation of particles can also be seen that occurred during dyeing at boiling point. The agglomeration of nano particles may be attributed to the thermo-migration of the nano-sized silvers that happened during boiling. The undyed fabric showed lower aggregation of nanoparticles on the fabric surface, while the dyed fabrics indicated more aggregation of nanoparticles due to the materials added to the dyeing bath (see ***Figure 8***).

EDS spectrum

The presense of silver nanoparticles on the surface of nylon samples was confirmed using the EDS spectrum. ***Figure 7*** indicates the EDS spectra of treated samples, confirming the existence of the Ag element on the surface of the fabric treated at a 500 ppm concentration of Ag in each sample (a - undyed, b - red, c - blue, d - yellow). Also the existence of the Au element in the EDS spectra is related to a gold layer which covers the outer layer of the sample to prepare for taking SEM images. ***Figure 9*** presents the reflectance spectra of samples before and after the washing process, as

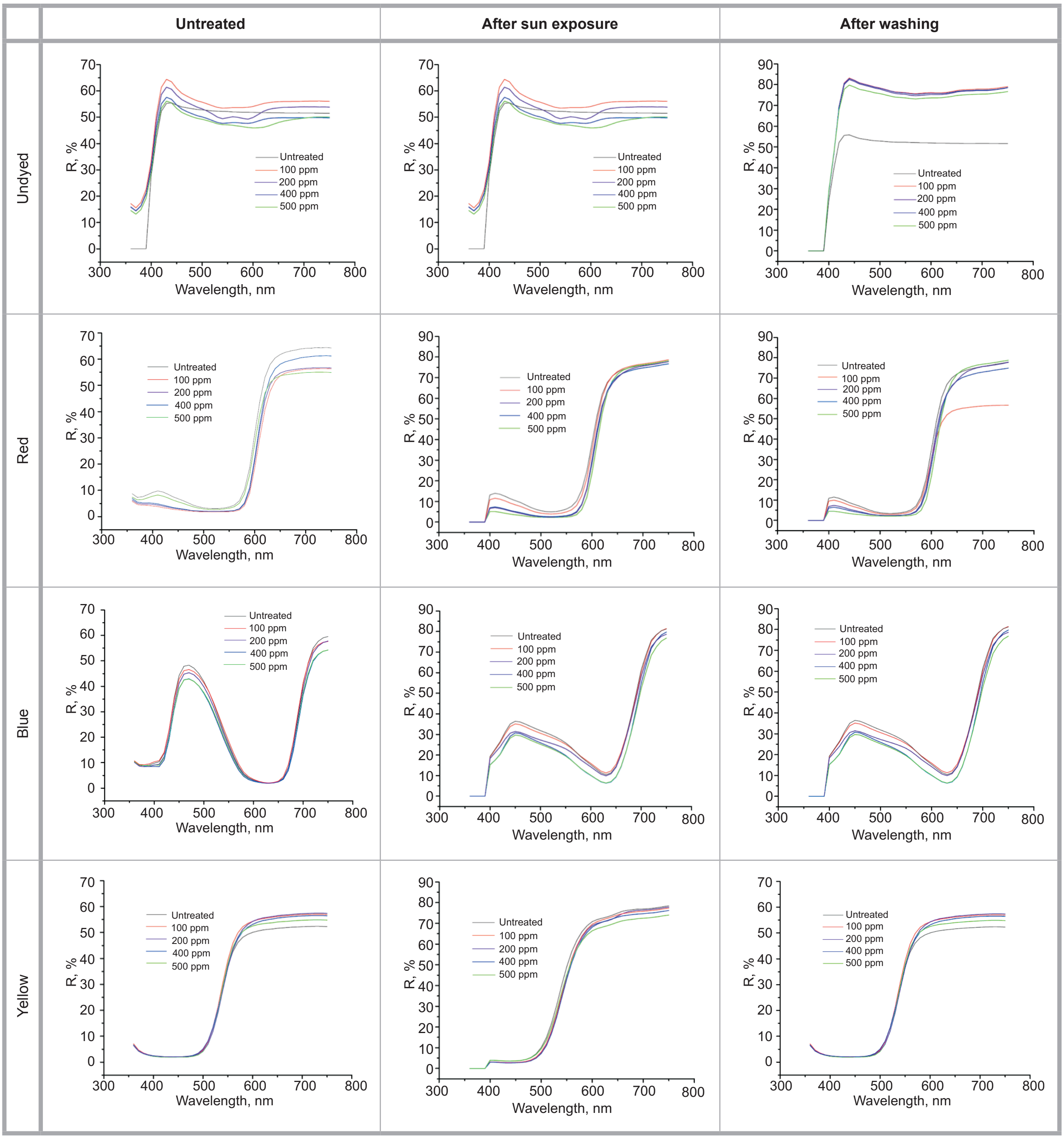


Figure 9. *Reflectance spectrum of samples before and after washing and sun exposure.*

well as after sun exposure. After treating fabrics with red color in the presence of silver nanoparticles, the reflectance of fabrics decreased in 400 - 450 cm^{-1}, which can be due to the surface Plasmon of nanoparticles. This phenomenon was observed for the color blue, but it was from 400 - 600 cm^{-1}. In yellow coloured fabrics, the UV absorbance was not observed because the colour yellow absorbs UV radiation and the reflectance at 400 - 500 cm^{-1} is low. In addition, good fastness against washing and the sun was considered, showing that Ag and Au were two major elements on the nylon samples treated. The results were reported based on both the weigh (wt%) and atomic percentages (at%) of the elements discovered.

Washing and crocking fastness

Figure 9 represents the reflectance spectrum of samples before and after the washing process, as well as after sun exposure. The Surface Plasmon bands appearing in the visible region are characteristic of noble metal nanoparticles. Silver nanoparticles have a surface Plasmon resonance absorption in the UV-Visible region. After treating fabrics with the colour red in the presence of silver nanoparticles, the reflectance of the fabrics decreased in 400 - 450 cm^{-1}, which can be due to the surface Plasmon of nanoparticles. This phenomenon was observed for the color blue, but it is from 400 - 600 cm^{-1}. In yellow coloured fabrics, the UV absorbance was not observed because the colour yellow absorbs UV radiation and

Table 2. Colour fastness evaluated by the Gray scale after 5 laundering cycles and rubbing fastness in dry conditions.

Samples	Color fastness to washing	Crocking fastness wet	Crocking fastness wet after 5 times
Undyed	5	5	5
White100	4	5	5
White 500	4 - 5	5	5
Yellow 100	4	5	5
Yellow 500	4 - 5	5	5
Red 100	4	5	5
Red 500	4 - 5	5	5
Blue 100	4 - 5	5	5
Blue 500	4 - 5	5	5

the reflectance in 400 - 500 cm^{-1} is low. Thus it is proposed that the nanosilver on polyamide fabric acts as an absorber of UV wavelengths. In addition, good fastness against washing and the sun was considered.

The dye and nanoparticles should have penetrated into the Nylon fabric, but stay mostly on the internal side of the surface since dyed fabrics possess very good crocking fastness. However lower washing fastness was achieved (***Table 2***), which can be due to the polymeric structure of polyamide fabrics because they have high crystallinity and low swelling ability in water in comparison to other polymeric fabrics, such as wool and cellulose.

Conclusion

In this research, nano silver particles imparted an efficient antibacterial property to nylon fabric with excellent washing durability. The coordinates of the CIE Lab colour system showed that more silver nano particles loaded on the nylon fabric surface leads to colour change. Also XRD patterns, SEM micrographs and the EDS spectrum confirmed the presence of nano silver particles on the nylon fabrics. It is important that dyeing and finishing with nano silver particles be done simultaneously for reasonable results.

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