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Improvement of wool dyeing quality and its antimicrobial activity using nano forms of silver

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Abstract In conjunction with the increasing public awareness of the infectious diseases, textile industry and scientists are developed hygienic fabrics by the addition of various antimicrobial compounds. The current investigation was carried out using nano-silver applied to wool fibers for studying its effects on wool quality and antimicrobial resistance. Antimicrobial activity is investigated against a broad range of microorganisms including bacteria, yeast and fungi. Moreover, dyeing of wool fibers treated with silver nitrate with extracted dye from the filamentous fungus, *Pencillium purpurogenum* by using innovation technique to save energy and time. In order to obtain color of aimed specific red hue, the influence of certain dyeing process conditions namely dyestuff concentration, pH, temperature, and duration of the dyeing process were studied. Antimicrobial effect is derived from nano-silver particles (diameter between 1 and 100 nm) which are adhered to the fibers. The fibers treated with silver particles was dyed by the investigated dye, and the antimicrobial activity was recorded. The wool materials showed antimicrobial effects by killing and/or suppressing growth of a broad spectrum of microbes such as *Bacillus subtilis*, *Escherichia coli*, *Candida albicans* and *Aspergillus niger*. The results indicated that nano-silver application was reflected positively on the zones of growth inhibition of wool fibers *E. coli* gave the highest diameter of the inhibition zone. On the hand, the applied pigment and the nano forms showed minimum effect on *C. alibicans and A. niger*.

Keywords: Wool, Dyeing, Antimicrobial, Natural pigment, Nano-silver nitrate

Introduction

The modification of textile fiber surface using nanomaterials is an interesting avenue for research. Silver is considered of particular interest because it is an efficient antibacterial agent. It has antimicrobial efficacy against bacteria, viruses, and microorganisms (Gao and Cranston, 2008; Rivero *et al*., 2015, Firdhouse and Lalitha, 2015). This excellent antibacterial activity makes it possible to use in different fields such as food preservation, safe cosmetics, medical devices, water treatment, and textiles fabrics. The large surface of nano materials and high surface energy ensures good immobilization of nanoparticles onto fabrics leading to increase in durability of the textile functions (Patra and Gouda, 2013).

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It also has other excited properties especially antibacterial activity to the treated fabrics (Patra and Gouda, 2013).

Recently, an awareness of general sanitation, contact disease transmission, and personal protection had led to develop antibacterial fibers to protect wearers against the spread of bacteria and diseases rather than to protect the quality and durability of the textiles (Chun and Gramble, 2007). The antimicrobial effect derived from silver ions which have advantaged over the conventional antibiotics, as it did not induce resistance in the microorganisms, and did not lost the antimicrobial strength over time. Because of the developing resistance of bacteria against bactericides and antibodies, the irritant and toxic nature of some antimicrobial agents, biomaterial scientists are focused their research on nano-sized metal particles such as silver, titanium dioxide, and copper. Silver, in its many oxidation states (Ago, Ag+, Ag2+, and Ag3+) has been recognized as an element with strong antimicrobial action against many bacterial strains and microorganisms (Lansdown, 2002). Thus, silver nano particles have one of the best anti-microbial characteristics covering a wide range of pathogenic micro-organisms. Silver nano particles are exhibited very strong bactericidal activity against both gram-positive and gram-negative bacteria including multi resistant strains (Morones *et al.,* 2005; Panacek *et al*., 2006). Additional results demonstrated that Ag-NPs can inhibit fungi at 10 microgram per milliliter (µg/ml).Regards to this concentration, silver nano particles will lead to high antimicrobial activity compared to bulk silver metal (Gozde and Zenrin, 2018).They reacted with the sulfur based proteins of the cell wall in microorganisms, and inhibited the metabolism and destroy the microbe(Ki *et al*., 2000).Clothing is known to be susceptible to different microbial attack since, it showed largesurface area and absorb moisture required for microbial growth**.** A variety of antimicrobial textile materials are reported, and employed different approaches like development of antibacterialnylon fiber by attaching a phosphate glass as an antibacterial agent (Alexandra *et al*., 2014). Pigments are already used in various field such as medicine, food, paper, textiles, animal feed etc. If these dye pigments are antimicrobial affected, it would be desired feature for quality of life**.** Antimicrobial activity and minimum Inhibition concentration (MIC) of dyestuff were investigated and antimicrobial effect of wool yarn treated with selected dyes were also determined against test microorganisms (Ali, *et al*., 2014).

The main purpose of this study was investigated the effect of time and concentration of natural dye obtained from the fungus, *Penciliium purpurogenum* on wool dyeing. It also extended to investigate the effect of nano-silver application on dyeing process. The antimicrobial activity of the dyed wool were tested to inhibit different microbial strains.

Materials and methods

Wool fibers were supplied by El Mahalla Company, Egypt. Silver nitrate and glucose were purchased from ELGmhoria company. 100 up 500 Watt. Microwave, and the microwave equipment used in this experiment was Samsung M 245 with an output of 1,550 watts operating at 2450 MHz. This research was carried out in National Research Center, Textile Research Division, Dyeing and Printing Department, Cairo, Egypt.

Microorganism

The culture of *Penicillium purpurogenum* was purchased from Assiut University Mycology Centre (AUMC. The spore suspension of 2×10^7 spores/ml was used to transfer to Czapeck yeast agar (CYA) growth media that contained the following ingredients (g/l):- glucose 30 g, yeast extract 2g, peptone 10 g, $NaNO_3$ 3 g, KCl 0.5 g, MgSO4 0.5 g and agar 25 g, which incubated for 7 days at 28 ℃ and stored at 4 ℃ (Abd-Elsalam and Ali, 2020).

Dyeing procedure

Dyeing of wool fibers was carried out using microwave heating. The dye under investigation was applied before and after treatment at different pH (3-,7) in dyeing bath, for periods of time (1-5 minutes) at different conc. (2-8) g/L. After dyeing, wool fibers were rinsed with water and then dried at room temperature. K/S values of dyed wool fibers were measured (Ali *et al*., 2019).

Measurements of color strength (K/S value)

An Ultra Scan PRO spectrophotometer was used to measure the reflectance of the samples and hence, the K/S was measured spectrophotometrically at wave lengths of λmax 385nm. The K/S of untreated and pretreated wool fibers with chitosan and nano-materials dyed with saffron red and yellow mixture were evaluated. K/S where K and S are the absorption and scattering coefficients, respectively.

CIELAB coordinates (L* a* b*) of undyed and dyed wool fibers were determined using an Ultra Scan PRO spectrophotometer (Hunter Lab) with a D65 illuminant and 108 standard observer (Ali *et al*., 2019).

Testing antimicrobial activity measurement

Nutrient agar medium (g/L) consists of peptone 5.0 g, beef extract1.5 g, yeast extract 1.5 g, NaCl 5.0 g, and agar 20 g at pH 7.5 which was prepared and autoclaved at 121 ℃ for 20 min. Sterilized petriplates were prepared with an equal thickness of nutrient agar. Test organisms involved gram negative bacteria (*Escherichia coli*) and gram positive bacteria (*Bacillus subtilis*) and fungi *Candida albicans*, *Aspergillus niger* were grown overnight at37 ℃, 120 rpm in 2 mL nutrient broth. This broth was used for seeding the agar plates. Wool samples were placed on top of the seeded medium under sterile conditions. After overnight incubation at 37 ℃, the zones of inhibition were measured. In the second set of experiments, untreated wool samples were tested (Dhanalakshmi *et al*., 2013).

Results

Effect of time on the color strength

The results indicated that the color strength of dyed wool fibers dyed by microwave affected by duration of time (1-5 min) as seen in Table 1. The dye under investigation was high affinity done for the fibers. The results showed that the time increased the dyeing efficiency increased and reached to the maximum at 5min (Figure 1, 2).

Figure 1. Samples dyed with the investigated at different time intervals (1-5) min. by microwave

Table1. Effect of time on the color strength and Color data (maximum wave length 370nm)

Time (min.)	K/S	L*	a*	b*	C	H
1	19.32	33.55	9.49	12.93	16.04	53.72
2	19.19	32.07	9.73	12.47	15.82	52.05
3	19.66	29.54	9.15	12.28	15.32	53.31
4	22.83	26.76	10.64	12.64	16.53	49.92
5	22.97	28.95	9.23	11.68	14.88	51.69

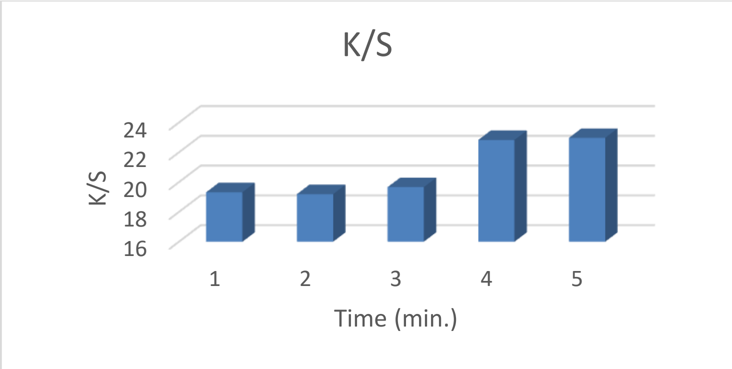

Figure 2. Effect of time on color strength

Effect of dye concentration

In the present experiment, the effect of different dye concentrations of 2,4,6,7 and 8 g/l was investigated. The results indicated that the maximum dyeing was obtained at concentration of 2g/l after which a reduced color strength which noticed in Table 2 and Figure 3. On the other hand, at low concentration showed that remarkable lowered in color strength.

Table 2. Effect of concentrations on the color strength and color data at maximum wave length 410

Conc. (g/L)	K/S	L*	a*	b*	C	H
2	22.29	33.55	14.05	20.78	25.09	55.94
4	15.73	33.04	10.35	14.80	18.06	55.04
6	22.35	27.74	9. 53	14.15	17.05	56.05
7	19.81	30.24	11.37	14.40	18.35	51.69
8	17.28	32.50	11.02	16.07	19.49	55.58

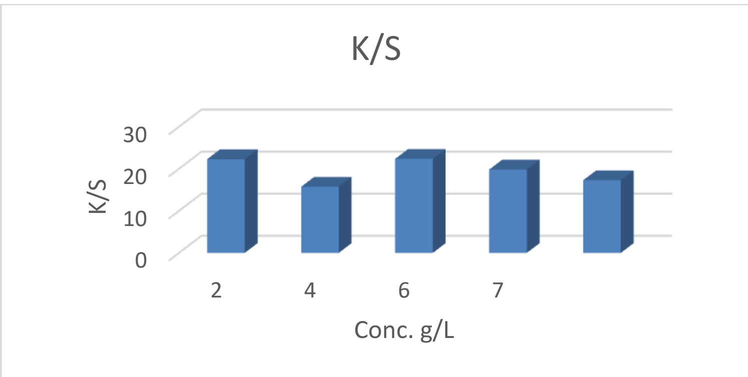

Figure 3. Effect of dye concentration on the color strength(K/S)

Effect of pH on the color strength for dyed wool fibers pretreated with nano silver nitrate

The color strength of wool fibers dyed with natural dye under investigation were affected by pH. The dye is high affinity to the fibers in acidic medium. In acidic medium, the cat ionized amino groups can absorb anionic dye molecules by the electrostatic attraction. Result showed that the color strength of wool fibers dyed with the dye under investigation gave the highest value of color strength (K/S) at pH 5 as presented in Table 3 and Figure 4.

Table 3. Effect of pH on the color data and color strength at maximum wave length 410

pH	K/S	L*	a*	b*	C	H
3	15.43	38.7	13.60	20.78	25.09	55.94
4	16.54	30.38	13.22	14.80	18.06	55.04
5	20.10	32.34	12.28	14.15	17.05	56.05
6	19.54	29.71	11.20	14.40	18.35	51.69
7	1.85	94.10	1.78	16.07	19.49	55.58

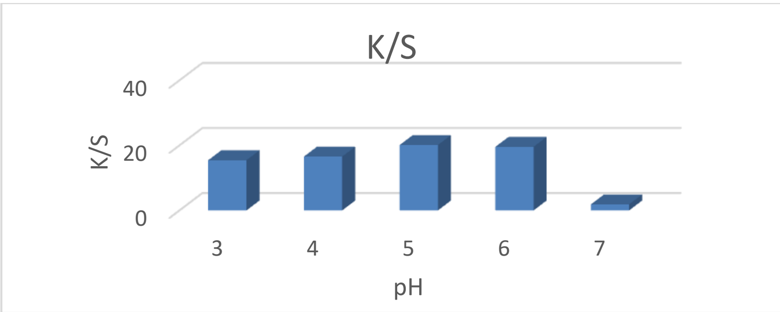

Figure 4. Effect of pH on color strength (K/S)

Application of the optimized conditions

The present study investigated the optimized conditions which obtained from the previous experiments on wool dyeing and compared with control wool. The tested optimum conditionswere5min, 2g/l at pH 5. The results showed the enhancement effect of nano-silver nitrate (Table 4 and Figures 5 and 6).

Table 4. Color data and K/S of samples at optimum condition for5 minutes with a concentration of 2g/L and pH 5 at maximum wave length 460

Sample	K/S	L*	a*	b*	C	H
Blank	0.4	14.12	77.65	0.4	77.65	14.12
untreated	2.24	11.47	51.49	17.95	51.49	21.30
Treated	11.16	23.45	37.41	12.41	37.41	26.53
Treated and dyed	15.31	16.61	29.35	10.65	29.35	19.73

Figure 5. Dyed samples at optimum condition (time 5 min., conc. 2g/L, and pH 5)

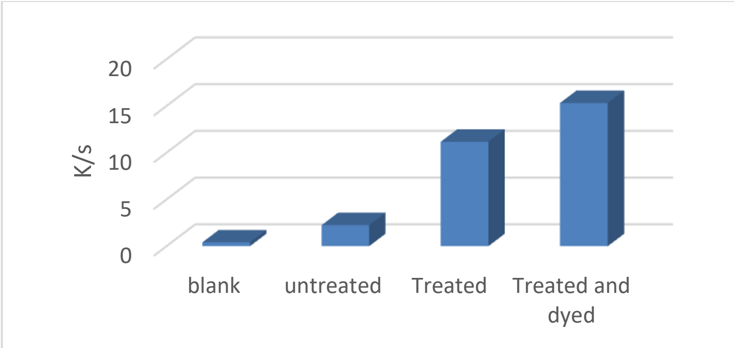

Figure 6. Relation between K/S of samples (blank, untreated, treated and treated dyed) at optimum condition

Antimicrobial activity for wool samples

In the present set of experiment, the wool samples were tested against *C. albicans* and *A. niger*. The results presented that at the start of incubation process had no apparent prevention of growth to the tested microbial strains after 24h of incubation. The treated wool sample with dye showed inhibition growth of *E. coli*. But there were no effects on gram positive bacteria as shown in Table 5. On the other hand, the dyed wool and the nano silver treated one showed a clear inhibition zone for the tested microorganism.

Table 5. Antimicrobial activity of wool samples dyed with the investigated dye

Microorganism		**samples**		
		inhibition	zone	**(mm)**
	control	treatedwithpigment	Nano silver	**+ pigemnt nanosilver**
Escherichia coli	-	5	15	**20**
Bacillus subtilis	-	4	12	**15**
Candida albicans	-	-	5	**7**
Aspergillus niger	-	-	4	**5**

Discussion

Different concentrations of extracted dye were doneby microwave method. It can be concluded that the increase in extracted dye concentration used, there was decreased in L* values and thus the color of the samples got darker. As the dye concentration increased, a* and b* values increased in the positive direction. The color of dyed wool fibers turned to reddish yellow color and became darker with increased dye concentration from 2 to 8 g/ L. Microwave dyeing considers the dielectric and the thermal properties of matter. The dielectric property refers to the intrinsic electrical properties that is affected the dyeing by dipolar rotation of the dye and influences the microwave field upon the dipoles. The aqueous solution of dye was two components which are polar in the high frequency microwave field. It influenced the vibration energy in the water molecules and the dye molecules. The color strength of dyed wool fibers was affected by concentrations. The dye under investigation was high affinity for the fibers. Result showed that the color strength of wool fibers dyed with the dye under investigation gave the highest value of color strength (K/S) at conc 2g/L. The color strength of wool fibers dyed with investigated natural dye was affected by dye bath pH. The dye had high affinity for the fibers in acidic medium. In acidic medium, the cationized amino groups can be adsorbed an anionic dye molecules by the electrostatic attraction. The color strength of dyed wool fibers is dyed by microwave that affected by duration of time. The dye under investigation was high affinity for the fibers, the color strength of wool fibers dyed with the dye under investigation gave the highest value of color strength (K/S) after 5 min which similar to research work of Abd-Elsalam and Ali (2020).

The results showed that the untreated wool had no antimicrobial activity against the selected gram positive and gram-negative bacteria and the fungi. The pigment treated wool revealed a remarkable antimicrobial properties in slight extent against *E. coli* and *Bacillus* sp. used. On the other hand, the application of nano silver with the tested pigment revealed the best ant microbial activity with broad spectrum activity gram negative and gram negative activity and *C. albicans.*

The nanoparticles of silver showed high antimicrobial and bactericidal activity. The size of nanomaterials was similar to that of most biological molecules and structure, therefore it can be used in many biological and industrial application. It is seen that silver nanoparticles are nontoxic to human at low concentration (Alexandra *et al*., 2014, Dhanalakshmi *et al*., 2013 and Pabba, 2015).

The first one postulated that AgNPs act at a membrane level as they are able to penetrate the outer membrane, accumulating in the inner membrane where the adhesion of the nanoparticles to the cell generate their destabilization and damage the increasing membrane permeability and inducing leakage of cellular content and subsequently its death. It is also showed that AgNPs can interact with sulfur containing proteins in the cell wall of bacteria an interaction that may cause structural damage leading to cell wall rupture. The second postulation, AgNPs would have an affinity to interact with sulfur or phosphorous groups present in intercellular content such as DNA and protein slathering their structure and functions. A third mechanism is proposed to occur in parallel with the two other which is released silver ions from the nanoparticles, which due to their size and charge. It can interact with cellular component altering metabolic pathways, membrane and even genetic material (Oini *et al*. 2020; Nicoara *et al.,* 2020; Mukhopadhyay *et al.,* 2019; Rajni *et al*., 2005). The nanoparticles of silver showed high antimicrobial and bactericidal activiti**es**. The size of nanomaterials is similar to the most biological molecules and structure, therefore it can be used in many biological and industrial application. It is seen that silver nano-particles are nontoxic to human at low concentration (Ali and Abd-Elsalam, 2020; Abd-Elsalamand Ali, 2020).

It concluded that antimicrobial activity was investigated against a broad range of microorganisms including bacteria and fungi. Moreover, dyeing of wool fibers treated with silver nitrate with extracted dye from the filamentous fungus *P. purpurogenum* by using innovation technique to save energy and time. The results indicated that nano-silver application was reflected positively on the zones of growth inhibition of wool fibers. *E.coli* gave the highest diameter of the inhibition zone. On the hand, the applied pigment and the nanoforms showed minimum effect on *C. alibicans and A. niger*. The influence of certain dyeing process conditions namely dyestuff concentration, pH, temperature, and duration of the dyeing process were elucidated.It can be concluded that the increase in extracted dye concentration, there was decreased in L* values, and thus the color of the samples got darker. As the dye concentration increased, a* and b* values increased in the positive direction. The color of dyed wool fibers turned to reddish yellow color and became darker with increased dye concentration from 2 to 8 g/ L.

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