Effect of Metallic Salts on Dye Ability of Cotton Fabrics

Sh. Shahidi*

Abstract
In this research work, the antibacterial activity of mordant treated cotton fabrics and also dye ability of them after mordant treatment was investigated. Copper chloride, nickel nitrate and silver nitrate were used as mordant. Very good antibacterial activity for mordant treated fabrics were achieved. The Scanning Electron Microscope (SEM) was used for morphological study. The fabric samples were incubated in mordant solution, then were dyed using Acid, Basic and direct dyes as a synthetic dyestuffs and with natural dyes such as madder and Indigo. The effect of mordant procedure on dye ability was investigated. In this research work, type of dye and metal mordant effect were studied. Various colors have been achieved by using different mordants. The results showed that, the effect of mordant treatment temperature on dye ability is not significant but the changes of mordant type causes changes in hue, great deal and purity of colors. Color intensities of the dyed fabrics were measured by using a UV VIS-NIR Reflective Spectrophotometer, over the range of 200-800 nm. The results showed that, by using different mordant before dying, the relative color strength (K/S values) of the dyed fabrics have been increased and it has shown that, more amount of dye can be absorbed. This effect is more significant in case of using basic dyestuffs. Also the shade achieved after mordant treatment is more brilliant as compare to untreated dyed fabrics. The results are discussed in full paper.

Keywords: Textile, cotton, Metallic salts, dye ability

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1. Introduction

Polymers having intrinsic bacteriostatic and/or bactericidal activities are of interest as wound-dressing materials. Polyamide (PA) synthetic textiles are among the most used fibers in the biomedical textile field[1,2]. Also natural textiles such as those made from cellulose and protein fibers are often considered to be more vulnerable to microbe attack than man-made fibers because of their hydrophilic porous structure and moisture transport characteristics. Thus, the use of antibacterial agents to prevent or retard the growth of bacteria is becoming a standard finishing for textile goods. There is, however, an increasing public concern over the possible effects of antibacterial finishing on environmental and biological systems since many antibacterial agents are toxic chemicals. They are also lack of efficiency and durability. Thus, an ideal textile antibacterial finishing should be safe and environmentally benign besides killing undesirable micro-organisms[3-5].

In recent years, the demand for antibacterial fabrics in domestic and abroad markets has grown significantly because of more awareness of the potential threat of spreading diseases. Bio-protective fabrics such as medical clothes, protective garments, and hygienic textiles are the main application of the antibacterial fibers. The general methods of preparing antibacterial fibers are adsorbing or grafting some antibacterial materials such as silver, metal complex and quaternary ammonium group on fibers' surface [6,7].

The antimicrobial properties of the silver ion Ag+ have been exploited for a long time in the biomedical field. The significant feature of the silver ion is its broad-spectrum antimicrobial property, which is particularly significant for the polymicrobial colonization associated with biomaterial related infections. The general finding is that bacteria show a low propensity to develop resistance to silver-based products, and therefore both metallic and ionic silver have been incorporated into several biomaterials such as polyurethane, hydroxyapatite (HA) and bioactive glasses. Silver containing products are also interesting materials for wound repair applications. When metallic silver reacts with moisture on the skin surface or with wound fluids, silver ions are released, damaging bacterial RNA and DNA, thus inhibiting replication[8-13]. Sustained silver release products have a bactericidal action and manage wound exudates and odor.

In the other point of view, Over the years a number of studies on the finishing of cotton fibers have been carried out to improve dye-uptakes and exhaustion properties. The focus of most research was to provide cationic sites on the cotton fibers for interacting with anionic dyes. These studies which were commonly involved chemical modification employing
cationization of cotton fibers with suitable cationic agents, providing attractive sites for anionic dyes [14].

While several classes of dye can be successfully applied to the cotton fibers, including direct, azoic, vat and reactive dyes, the application of cationic dyes has not gained widespread success. So in this research work, effect of mordant treatment on antibacterial activity of cotton fabrics were investigated, also the influence of them on dyeability of fabrics with different classes of dyes was examined.

2. Experimental part

Woven Cotton fabrics were kindly supplied by the Baft Azadi Co (Tehran, Iran). Before mordant treatment, fabrics were washed with 1 g. lit⁻¹ nonionic detergent and the washed samples were dried in a laboratory oven.

Silver nitrate, Copper chloride and nickel nitrate were purchased from Merck. The samples were incubated in a 0.01 M solution of metallic salts, protected from light, and mordanting of them was started at room temperature, the temperature was raised for 2°C/min to boil and heated for 60 min. After this period expired the samples were rinsed 3 times with deionized water, subsequently analyzed by the various surface characterization techniques, or submitted to bacterial activity test. Immediately before surface analysis the samples were dried in the laboratory oven.

The morphology of the fabrics was observed using a Scanning Electron Microscope (SEM) (TESCAN Brno, Czech Republic). All of the samples were gold coated before conducting the SEM examination. For Bacteria counting test, Luria Bertani media (LB) broth was used as growing medium for Staphylococcus aurous. Bacteria were dripped in 10 mL of LB broth to reach a cell concentration of $1 \times 10^8$ (CFU)/mL. Then it was diluted to a cell concentration of $1 \times 10^6$ (CFU)/mL. 1cm×1cm size fabric sample was cut and put into 1mL Bacteria suspension. All samples were incubated for 24 h at 37° C. From each incubated sample, 100 μL of solution was taken and distributed onto an agar plate. All plates were incubated for 24 h and colonies formed were counted. The percentage reduction was determined as follows:

\[
\text{Reduction (\%)} = \frac{(C-A)}{C}
\]
Where C and A are the colonies counted from the plate of the control and treated sample, respectively. The fabrics were dyed using Acid, Basic and direct dyes as a synthetic dyestuffs and with natural dyes such as madder and Indigo.

For synthetic dyeing processes, aqueous solutions, containing 3.0 wt.% of the dye were employed. The bath ratio was 1:30 (1 g of fiber in 30 ml of dye solution). The following dyeing conditions were adopted: For Direct, Anionic and Cationic dyeing processes: Initial temperature 40 °C, followed by a temperature increase of 3 °C.min⁻¹ up to 80 °C, holding for 30 min at 80 °C. 30 g/lit NaCl were included in the direct dyeing medium. 5 g/lit of acetic acid for pH adjustment, were added for anionic dyeing processes. After dyeing, the fabrics were rinsed with cold hot-cold water and then dried at room temperature. In case of natural dyeing, the samples were steeped in the dye bath with liquor to Good ratio (L: G) of 40:1 that prepared by 50% (o.w.f) on weight of fabrics of extracted dye at PH=4.5-5.5 (adjusted by acetic acid).

Dyeing of sample was started at room temperature and the temperature was raised for 2°C/min to boil and heated for 60 min. The sample were rinsed with tap water and dried at room temperature.

Color intensities of the dyed fabrics were measured by using a UV VIS-NIR Reflective Spectrophotometer, over the range of 200-800 nm. And the reflection factor (R) was obtained. The relative color strength (K/S value) was then established according to the following Kubelka-Munk equation (2), where K and S stand for the absorption and scattering coefficient, respectively:

\[ \frac{K}{S} = \frac{(1-R)^2}{2R} \]  

The effect of mordant procedure on dye ability was investigated. In this research work, type of dye and metal mordant effect were studied.
3. Results and Discussion

3.1. Antibacterial Efficiency

In this research work, the antibacterial activity of mordant treated cotton fabrics and also dye ability of them after mordant treatment was investigated. For testing the antibacterial efficiency of the samples as mentioned before, the bacterial counting test were used. The results of bacterial counting test are shown in Table 1. As it can be seen, the colonies of bacteria can spread over the plate of untreated samples. But in case of treated samples, no bacteria can distributed over the plate.

It means that, mordant treatment on cotton samples, causes decrease in amount of survival bacteria. Also the results show that, silver, copper and nickel salts causes killing of bacteria and percentage reduction of bacteria in case of Ag incubated samples reach to 100%. It means that, no bacteria can spread over the agar plate. Also the results of antibacterial efficiency for Cu and Ni loaded samples are very good.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Reduction (%) of Staphylococcus aureus</th>
<th>Counted bacteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 times diluted 0.5 Mc farland suspension</td>
<td>-</td>
<td>1500</td>
</tr>
<tr>
<td>Untreated cotton</td>
<td>6.66</td>
<td>1400</td>
</tr>
<tr>
<td>Ag loaded cotton</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>Cu loaded cotton</td>
<td>99.93</td>
<td>1</td>
</tr>
<tr>
<td>Ni loaded cotton</td>
<td>99.73</td>
<td>4</td>
</tr>
</tbody>
</table>

3.2. Morphological study

SEM is the best known and most widely used tool for surface morphological analyses. SEM micrographs of untreated and mordanted samples with metallic salts are shown in Figure 2. As shown, some new particles were created on the surface of treated cotton fabrics that
did not exist on the surface of untreated cotton fibers. These new particles are attributed to metallic salts.

![Figure 1. The SEM images of untreated and mordant treated cotton](image)

### 3.3 Dyeing properties

As it mentioned before, in this study, the effect of three kinds of metallic salt on dyeability of cotton fabrics were investigated. Three kinds of synthetic and two kinds of natural dyes were used. As it can be seen in Figure 3, the mordant treated cotton has more affinity to basic dye. The hue of Cu and Ni incubated cotton is more brilliant than untreated cotton. In case of Ag-loaded dyed cotton with basic dye, it is shown that, the color and purity have changed. The results of reflection factor of dyed samples with basic dye are shown in Figure 4.
The results show that, the reflection factor of untreated cotton is more than mordant treated samples. It means that, by mordant treatment on cotton fabrics, the absorption of basic dye has been increased. The reflection factor of Ag-loaded sample is less than the others. The results of K/S values are shown in Table 2. The amount of K/S value by pre mordant treatment have been increased and the maximum amount of K/S is related to dyed Ag-loaded cotton.
Table 2. Amount of relative color strength (K/S) for dyed cotton with Basic dye

<table>
<thead>
<tr>
<th>samples</th>
<th>Average of Reflectance factor</th>
<th>Average of K/S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated dyed with basic dye</td>
<td>0.39</td>
<td>0.47</td>
</tr>
<tr>
<td>Ni-loaded dyed with Basic dye</td>
<td>0.342</td>
<td>0.63</td>
</tr>
<tr>
<td>Cu-loaded dyed with Basic dye</td>
<td>0.321</td>
<td>0.69</td>
</tr>
<tr>
<td>Ag-loaded dyed with Basic dye</td>
<td>0.299</td>
<td>0.82</td>
</tr>
</tbody>
</table>

The photos of dyed cotton with acidic, Indigo and madder dyestuffs are shown in Figures 5-7. It can be seen that, pre-mordant treatment causes increase the dyeability of cotton with mentioned dyestuffs. In figure 5, it is shown that, Ni and Cu mordant treatment make no changes in dye absorption of cotton but Ag loaded cotton have more affinity to acid dye.

The Cu and Ag mordant treatment cause more absorption of indigo dye. But in case of dyeing of cotton with Madder, the results show that, the Ni treated cotton has less amount of absorption, but Cu treated sample can absorb more amount of dye, however, Ag treatment causes changes of hue of madder dye on the cotton.

Figure 4. The photos of dyed untreated and mordant treated cotton with Acid dye.
As it is shown in Figure 8 and 9, the hue and brightness of dyed samples are near each other. It means that, the effect of metallic salts on direct dye absorption of fabrics is not significant. The reflection factor for the dyed samples cover each other.
4. Conclusion

In this research work, cotton fabrics were treated with 3 kinds of metallic salt. Cupper chloride, Nickel Nitrate and Silver Nitrate were used as metallic salts for mordant treatment. The SEM images show that, after mordant treatment some new particles appear on the surface of cotton fibers. Also the antibacterial and dyeability of untreated and mordant
treated samples were investigated. The results show that, very good antibacterial activity has been achieved on the mordant treated samples. The antibacterial efficiency of silver nitrate treated samples is more than copper and Nickel treated fabrics. Moreover, the results related to dyeability of fabrics show that, the affinity of cotton to cationic dye after mordant treatment is increased significantly. More amounts of acid, madder and indigo dyes can be absorbed by Mordant treated cotton. But in case of direct dyes, the effect of metallic salts is not significant.

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6. References