

One-bath dyeing and antibacterial finishing of cotton fabric using reactive dye and silver chloride: a sustainable approach

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ABSTRACT

One-bath dyeing and antibacterial finishing of cotton fabric using reactive dye as colorant and silver chloride (AgCl) as an antibacterial agent was conducted to streamline the process and enhance its economic efficiency. The effectiveness of the antibacterial agent in reactive dyeing of cotton fabric, was evaluated through exhaust method with 0.5% AgCl and continuous (pad dry cure) method with 5 g/l of AgCl, utilizing various dye concentrations (1%, 3%, 5% for exhaust; 1g/l, 3g/l, 5g/l for pad dry cure) according to a standard reactive dye recipe. Characterization of the one-bath dyed, and antibacterial finished cotton fabric was performed using K/S values to determine the optimal dye shade concentration and antibacterial activity was evaluated using agar diffusion method. It was observed that the exhaust method revealed an optimum dye concentration at (AgCl 0.5% and dye 5%), while pad dry cure method showed optimal dye concentration at (AgCl 5g/l and dye 5g/l). Antibacterial tests were conducted on the optimal specimens from both methods (AgCl 0.5%, 0.2%, dye 0.5% for exhaust; dye 5g/l, AgCl 5g/l, 2g/l for pad dry cure), showcasing resistance against bacterial growth. While inhibition zones were observed on treated specimens of the exhaust method, whereas the treated specimens of the pad dry cure method exhibited complete resistance to bacterial growth around the specimen.

1. Introduction

The textile industry is continually making progress towards innovative production techniques to enhance product quality, eco-friendly processes have been conducted to make cost-effective manufacturing. Therefore, in the application process, the conventional trend is moving towards reducing the number of steps to develop one bath application processes [1], such as consolidating the pre-treatment process and presenting usefulness along with dyeing [2]. The dyeing is the next step after the pretreatment of fabric and it tends to be done at any stage of manufacturing of textiles, like during the manufacturing of fibres, yarn, fabric,

finished textiles, as well as on garments. Cotton textiles are dyed using different synthetic dyes (reactive, vat, direct, sulphur and azoic) or natural dyes usually extracted from plants/vegetable/fruits [3]. Finishing is the last process of wet treatment in the textile industry. It is conducted to enhance the quality of fabric according to the end use which is given by the customers based on their needs. Finishing can be carried out by means of mechanical action or chemical treatments, which improves the fabric whiteness, crease resistance, and aesthetic appearance of the fabric. The finishing process of textile materials is mainly divided into two types: functional and

aesthetic. Aesthetic finishes are used to enhance the appearance of fabric, while functional finishes enhance the performance of fabric and make the fabric able to perform better under different conditions. The demand for antimicrobial textiles is increasing day by day; especially in pandemic times [4]. Textile materials act as a suitable medium for the growth of microbes which are found in everywhere under certain environmental conditions and under these optimal conditions i.e. nutrients (hydrocarbons, proteins), pH, and temperature due to which they can grow enormously [5]. The growth of these microbes on textile materials can cause various effects on human health. They give rise to bad smells, cause skin and various infections, and moreover affect the mechanical strength of fabric and aesthetic of fabric, etc [6]. The attentiveness towards harmless, durable, and economical antimicrobial finishing of textiles is increasing rapidly due to innovative techniques of production in the field of clinical, health protection, disinfection, and defensive textile materials [7]. The reduction of microbial development on the substrate is critical, as it directly influences human health [8]. The demand for antimicrobial finishing of cotton fibres has increased rapidly over the last decade. The cotton fibres are widely used in textile because of their excellent wearable properties. As cotton is a natural fibre, it is susceptible to microbial attack [9]. It is a suitable medium for the microbes' growth when it is in contact with the human body [10]. An antibacterial finishing can give stability to the textile materials against microbes. It holds an extraordinary incentive for modern materials that are presented for outside usages [11].

The need for antibacterial textiles for humans cannot be over-emphasized; however, their implementation as a finishing process is lengthy and a costly finishing process [12], so it is essential that this process can be implemented with other necessary processes of textile materials such as dyeing. The implementation of functional finishes along with the dyeing process not only saves energy but also has a significant impact on the environmental and economic process. Various types of antibacterial finishing agents have been used for the finishing of textiles, such as metal salts, chitosan, triclosan, N-halamines, per oxyacid, biguanides, quaternary ammonium have been used [13]–[16]; but many of them are not acceptable in terms of long-term durability, ecology and stability point of view [9]. However, silver, because of its physical and chemical properties, has been used for many years in different forms to alleviate injuries [17]–[20]. Silver chloride (AgCl) is well known because of its low water solubility and is considered

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an excellent antibacterial agent [21]. The antibacterial finishes can be applied either with other processes or separately, as well as with other finishing agents in a single process. By using one-bath dyeing and finishing or multi surface functionalization standard application procedure may require modification.

This research aims to develop a one-bath dyeing and finishing process with antibacterial properties, focusing on several key objectives. These include the development of an optimized recipe for single bath dyeing and antibacterial finishing using reactive dye and silver chloride (AgCl), investigating various parameters such as antibacterial activity, colour yield, tensile strength, and bending stiffness of the cotton fabric, and assessing the wash durability of the antibacterial finish on cotton fabric. Through these efforts, the research seeks to advance the development of efficient and durable one-bath dyeing and antibacterial finishing processes for textile applications.

2. Materials and Methods

2.1 Experimental Materials

Al-Karam Textile Mill Ltd Karachi supplied the bleached plain woven 100% cotton fabric (115 GSM) was used as test specimen. Commercial grade Drimaren® Yellow CL-2R reactive dye was purchased from Archroma Pakistan Limited. RUCO®-BAC AGP, a commercial grade silver chloride (AgCl) was used as an antibacterial agent produced by RUDOLF, Germany.

2.2 Experimental Methods

The cotton fabrics were treated with two different methods, i.e., exhaust and pad-dry-cure. The bleached cotton fabric was dyed with reactive dye using lab scale exhaust dyeing machine with different concentrations, as shown in Table 1. Common salt (NaCl) and soda ash were used as electrolyte and alkali respectively along with leveling agent and AgCl in the exhaust dyeing. The dyed fabric was cleaned by hot wash followed by cold wash to remove the unreactive dye from the surface of the fabric. The second method involved passing the fabrics through the padder machine at 70% pickup followed by dyeing and curing at 120 °C for 90 seconds and 180 °C for 60 seconds, respectively. The details of the padder liquor are given in Table 2.

Table 1

Dyeing recipe (exhaust)

Sample No.	Dye conc. [%]	AgCl conc. [%]	Temp. [°C]
01D1AgCl0.5	1	0.5	40-60
01D3AgCl0.5	3	0.5	
01D5AgCl0.5	5	0.5	
01D5AgCl0.2	5	0.2	
Untreated	5	-	

Table 2

Dyeing recipe (pad dry cure)

Sample No.	Dye [g/L]	AgCl [g/L]	Drying	Curing
01 D1AgCl5	1	5	120 °C for 90 sec.	180 °C for 60 sec.
02 D3AgCl5	3	5		
03 D5AgCl5	5	5		
04 D5AgCl2	5	2		
Untreated	5	-		

2.3 Characterizations

2.3.1 Colour strength evaluation

To check the colour strength of the fabrics, the K/S values were measured on the X-Rite Spectrophotometer CE7000A. Before taking the readings, the spectrophotometer was calibrated first by using the black tile and then calibrated by using the white tile. After calibration, the dyed sample, was measured using spectrophotometer.

2.3.2 Antibacterial activity

To assess the antibacterial activity of dyed-finished cotton. It was evaluated by standard test method AATCC 147, also called agar diffusion method used for qualitative assessment. For antibacterial test, the Mueller Hinton agar was used. The 3.8 grams of Mueller Hinton agar was dissolved in 100 ml of distilled water heated on the heating plate with a magnetic stirrer and boiled for 2-5 minutes to uniformly dissolve agar and then sterilizing in autoclave the solution for 15 minutes at 120 °C and naturally cooled at room temperature and poured into petri dishes.

It is a parallel streak and qualitative agar diffusion test method. The specimens were placed on the plates of nutrient agar. The incubation time is 24-48h with a temperature of 37 °C then investigation of agar plates was conducted to examine the growth of bacteria on the treated specimens which can grow at the surface of the specimens and inhibition zone formation is measured according to the given formula.

$$Q = (P - R) / 2 \quad (1)$$

Where:

Q= inhibition zone width

P= Total diameter clear zone in mm and of test specimen

R = diameter of test specimen in mm

Eq. 1 shows the actual width of the inhibition zone in mm, which is known as qualitative analysis. It specifies the quality of the antibacterial finish from which we get to know how much mm distance the bacteria resist from the specimens.

2.3.3 Tensile strength

The tensile strength of specimen is the property of a fabric to understand the tensile behaviour of textiles to break under load. The Titan-3 tensile tester was used following the ASTM D-5034 standard, the jaws adjustments (size of specimen) 75mm with rate of extension 300 mm/min were set. The specimens were clamped in between the upper and lower jaw. Samples should be clamped in such a way that there must be uniform tension across the width of clamp.

2.3.4 Fabric stiffness

Fabric rigidity and stiffness are considered as one of the main characteristics of fabric to determine the quality of fabric. It is important to manage the drapability of fabric. The stiffness in the fabric was measured by using the cantilever principle. The Shirley stiffness tester was used according to BS 3356 and (ASTM D1388) standard. To measure the bending length the specimens are measured in warp and weft direction. Each strip of specimens is placed on the plate of the tester and then a weight or load is applied on the strip with the help of the scale which is used to measure the bending length move the strip slowly towards the forward direction and allow it to fall by its own weight until the strip bend and coincides with the mirror lines corresponding to 41.5 degrees.

3. Results and Discussion

3.1 Colorimetric Analysis and Washing Fastness

The K/S values before and after the wash of both exhaust and pad dry cure methods are shown in Fig. 1-2. The optimized specimens were found at in case exhaust (dye 5%) and in case pad dry cure (dye 5g/l). The K/S values of treated specimens increased with AgCl concentration (0.5% exhaust and 5g/l pad dry cure) before and after a wash than the AgCl concentration (exhaust 0.2% and pad dry cure 2g/l). In comparison to the pad dry cure method, the one bath treated specimens of exhaust method before and after wash have greater k/s values than the pad dry cure method.

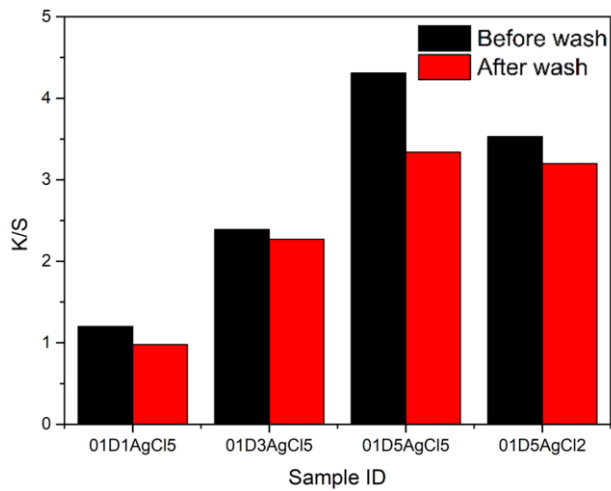


Fig. 1. K/S Values of Treated Samples Before and After Wash of Pad Dry Method

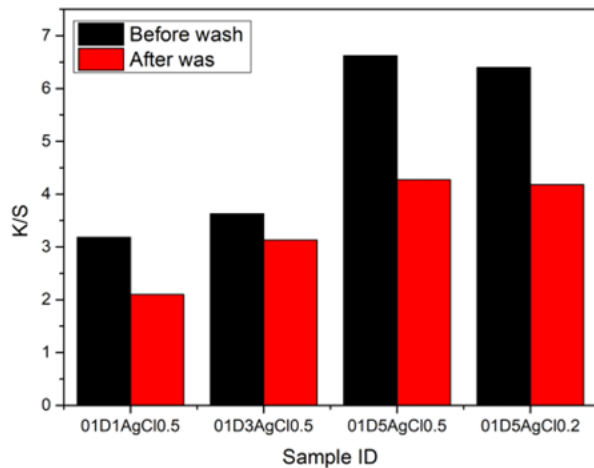


Fig. 2. K/S Values of Treated Samples Before and After Wash of Exhaust Method

3.2 Antibacterial Efficacy

The antibacterial test was performed according to the AATCC-147 method. The antibacterial activity of the treated fabric before and after washing is shown in Fig. 3 and Fig. 4 and Table 3. It has been observed that the treated specimens by pad dry cure and exhaust both have shown resistance against bacteria. The AgCl-treated surfaces by pad dry cure method completely resist the bacterial growth on it, as can be seen in Fig. 3. On the other hand, the untreated specimen showed considerable bacterial growth. The exhaust method specimens comparatively gave promising results both before and after washing. The inhibition zone width is greater around the specimens than the after wash, as can be seen in Fig. 4. It has been found that the treated specimens of the exhaust method have greater efficiency against bacteria than the pad-dry-cure specimens. This is because the exhaust method specimens were immersed in the dyeing bath for a longer period, unlike the pad-dry-cure treated specimens, which passed quickly through the padder and only received a surface coating that was removed after washing.

Table 3

Antibacterial assessment of treated fabrics

Pad dry method			
Sample ID	Growth of bacteria on specimen	Zone of inhibition [mm]	
		Before wash	After wash
Untreated	Yes	0	0
01D1AgCl5	No	1-2	No growth
01D3AgCl5	No	1-2	No growth
01D5AgCl5	No	1-2	No growth
01D5AgCl2	No	1-2	No growth
Exhaust method			
Untreated	Yes	0	0
01D1AgCl0.5	No	1-2	1
01D3AgCl0.5	No	1-2	1
01D5AgCl0.5	No	1-2	1
01D5AgCl0.2	No	1-2	1

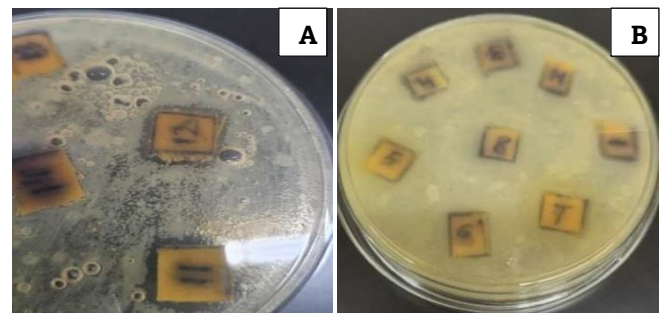


Fig. 3. Bacterial Resistance of Pad Dry Cure Before Wash (A) and After Washing (B)

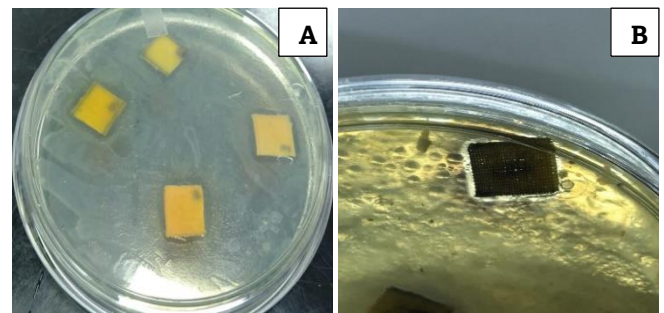


Fig. 4. Bacterial Resistance of Exhaust Method Before Wash (A) and After Washing (B)

3.3 Tensile Strength and Fabric Stiffness

The physical properties (tensile strength, extension, and bending length) are given in Table 4. It is important to check the effect of one bath dyeing and antibacterial finishing on the tensile strength of the fabric. Investigation reveals the tensile strength of treated and untreated specimens processed by exhaust method with 0.5% and 0.2% of AgCl has greater strength than the untreated specimens. It was observed that the specimen treated with 0.2% AgCl did not affect the fabric tensile strength like the untreated fabric. However, the 0.5% AgCl concentration treated has a greater effect on fabric tensile strength, it increases the strength of dyed fabric than untreated dyed fabric, possible reasons could be higher reactivity at higher concentration results in bond formation i.e. covalent bonds. Similarly, to exhaust the

tensile strength of treated specimens processed by pad dry cure method with 2g/l and 5g/l has tensile strength greater than the untreated specimens. It was found that treated specimens of pad dry cure method AgCl 2g/l and 5g/l had a greater effect than the untreated specimens.

Table 4

Physical properties of the treated cotton fabric

Pad dry method						
Sample ID	Breaking force [N]		Extension [%]		Bending length [cm]	
	Before wash	After wash	Before wash	After wash	Before wash	After wash
	Untreated	173.2	111	20.9	17.92	2.5
01D1AgCl5	238.8	238.6	8.18	9.1	2.84	2.81
01D3AgCl5	240	239.8	8.27	9.3	2.88	2.86
01D5AgCl5	245.5	184.7	8.43	9.7	2.9	2.7
01D5AgCl2	232.9	171.3	7.85	8.2	2.6	2.4
Exhaust method						
Untreated	160.8	141.6	12.29	15.31	2.02	2
01D1AgCl0.5	213.3	213	8.5	8.9	2.68	2.67
01D3AgCl0.5	215.4	215.2	8.81	9	2.69	2.69
01D5AgCl0.5	219.5	219.06	8.94	9.3	2.7	2.7
01D5AgCl0.2	164.8	149.3	20.84	20.14	2.15	2.13

It was observed that the treated specimens of exhaust method with 0.5% AgCl have increased the fabric stiffness than the treated specimens with 0.2% AgCl, while the treated specimens with AgCl 0.2% did not affect the tensile strength of treated specimens than the untreated before and after wash. The results of the pad dry cure are given in the Table 4.

It was analysed that the treated specimens of the pad-dry-cure method with AgCl have increased the fabric stiffness before and after washing. The 5g/l AgCl increased the specimen stiffness more than the untreated specimens, while 2g/l did not affect the stiffness of specimens compared to the untreated specimens. In comparison to both the exhaust and pad-dry-cure methods, the treated specimens of the pad-dry-cure method, both before and after washing, exhibited greater stiffness than those treated by the exhaust method.

4. Conclusion

The one-bath dyeing, and antibacterial finishing were successfully conducted using both the exhaust and pad dry cure methods. Optimization of dye shade and antibacterial activity for the dyed specimens was achieved with a dye concentration of 5g/l and AgCl concentration of 5g/l for continuous pad dry cure, and a dye concentration of 0.5% and AgCl concentration of 0.5% for exhaust. Initially, various concentrations of reactive dye and AgCl were applied to cotton fabric using both methods. Results indicated that a dye

concentration of 0.5% in exhaust and 5g/l in pad dry cure method exhibited a higher colour depth compared to other concentrations before and after washing. Furthermore, the addition of AgCl at different concentrations along with the reactive dye in both dyeing methods (i.e., exhaust and pad dry cure) increased the K/S value of the dyed fabric. It was concluded that incorporating the antibacterial agent AgCl in the one-bath dyeing process using both exhaust and pad dry cure methods demonstrated antibacterial activity against bacteria. Specifically, the application of AgCl via the exhaust method showed a greater inhibition zone against bacteria (i.e., *E. coli*) compared to the pad dry cure method, as the AgCl was surface coated in the latter, making it more prone to removal from the fabric surface after subsequent washings. It is anticipated that the one-bath antibacterial dyed fabric will have better laundering durability when dyed via the exhaust method compared to pad dry cure. Moreover, an assessment of fabric tensile strength and stiffness was conducted after applying reactive dye along with AgCl at different concentrations using both pad dry cure and exhaust methods. Results revealed that the inclusion of AgCl at different concentrations in the one-bath process significantly influenced fabric tensile strength and stiffness in both exhaust and pad dry cure methods.

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