

Developments of standard procedure to identify dope dyed and exhaust dyed meta aramid yarn

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Abstract

Indian Air force uses meta-aramid overall for their pilots due to its excellent properties such as high strength, flame and chemical resistance, etc. Dyeing of those overall materials is recommended through a dope dyeing process to achieve good fastness properties and uniform color. Dope dyeing is the process of adding color during spinning, while in the exhaust dyeing process, dye is gradually transferred from dye bath to textile materials. However, the fabric of the same color with different preparation routes looks similar but behaves differently during actual application due to different fastness properties. No standard method distinguishes exhaust or dope-dyed meta-aramid fiber/fabric. So, in this work, meta-aramid samples were dyed using both the dyeing process and different properties were investigated to find a confirmative method to distinguish the dyeing process. Based on the investigation, the heat treatment method and light fastness is found to be most suitable for this. The overall of Air Force pilot was also tested by this method for confirmation.

Key words:

Exhaust dyeing, Dope dyeing, Meta-aramid, Dyes and Pigments

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1. Introduction:

Different types of colored textile materials are available in the market. These textile materials are based on natural or synthetic materials. Synthetic materials are gaining wide acceptance due to their low cost and better properties. The most widely used synthetic fiber for flame retardant textiles is meta-aramid (MA) because of its excellent properties, such as high strength, abrasion resistance, and high-temperature resistance [1]. They are colored through either dope dyeing during the spinning stage or exhaust dyeing after producing filaments/fibers for specific applications [2].

Dope dyeing of filaments/fibers has gained considerable interest in the defence and industrial community in recent years [3]. It is primarily due to the intrinsic dyeing of fiber during spinning. It has altogether better physical and chemical properties than exhaust-dyed material [4]. Dope-dyed meta-aramid yarn is preferred in specific application areas like Army uniforms in Defence due to its higher fastness properties [5],[6]. In this context, it has also been realized that doped dyed polymer puts less burden on the environment. Despite this, the textile processors might compromise on this front because of the marginal increase in profit. Therefore, exhaust dyeing is still prevalent in the

textile wet processing industry not only due to its lower cost and easy processability but also because of having many more choices of different colors, unlike dope dyeing, where only fiber/filaments manufacturer have their options with the limited number of color pigments [7].

The meta-aramid fabric of the same color but produced by dope [8] and exhaust-dyeing routes [9] looks similar. However, the performance of the dope-dyed and exhaust-dyed fabric in terms of color fastness to various chemical agents and environmental conditions is different [10], [6]. Consequently, this difference in fabric performance may create shade-off or a tonal variation, which creates hurdles to achieving the desired uniformity [11]. However, no standard method is available to distinguish exhaust or dope dyeing methods of meta-aramid fibers/fabrics. Efforts are being made in this direction, but no confirmative result has been reported yet [12], [13]. Therefore, it is necessary to develop a standard method to understand whether the sample is dope dyed or dyed using the exhaust dyeing route.

In this research work, we have prepared colored samples through dope dyeing and exhaust dyeing processes. Systematic investigations of dope-dyed and exhausted yarns have been conducted to find a confirmative method to differentiate the dyeing process.

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2. Materials and Methods

2.1 Materials

Meta-aramid dope dyed and raw white yarn was supplied by Arvind India Ltd., India. Coracryl Red Modified Basic dye was purchased from Colourtex Industries Pvt. Ltd., India. Sodium carbonate and acetic acid were procured from Merck, India. Dimethyl formamide (DMF) and sodium dithionite were procured from Loba Chemie Pvt. Ltd., India. All the received chemicals were used as they received.

2.2 Methods

2.2.1 Exhaust Dyeing of raw white yarn

Aramid yarns were dyed with cationic dye. The pretreatment process was carried out before the dyeing so that the complex structure could open up and facilitate dyeing [14], [15]. In this process, aramid yarn was pre-treated with DMF with MLR 1:15 at a temperature of 80°C for 15 minutes and then washed with hot water. It was dyed with Coracryl Red Modified Basic dye with 5% shade, MLR 1:35, and pH 4-5. Dyeing was done in a high-temperature, high-pressure beaker dyeing machine at 120°C for 1 h followed by treatment with 1 gpl sodium carbonate and 1 gpl sodium dithionite at 100°C for 15 minutes and subsequent regular wash [9].

2.2.2. Spectral Colour Analysis

Spectral colour analysis was done before and after testing to observe any change in colour of the sample. Macbeth Colour Eye 7000 A Spectrophotometer were used to analyze the color value of the exhaust-dyed and dope-dyed yarn. Testing was done in UVD65 light under the specular included condition.

2.2.3. Dye Stripping

Dye stripping tests were conducted on exhaust-dyed and dope-dyed aramid yarn specimens to assess the retention and removal of the colorant. The stripping procedure was performed in a solvent DMF at temperature 80°C for a period of 30 minutes. Following the treatment, the samples were removed, rinsed thoroughly with distilled water to remove residual solvent and detached dye, and then dried at room temperature until a constant weight was achieved. Visual observation and colour value measurement were done to observe change in colour.

2.2.4. Colour Fastness to Sublimation

Meta-aramid fabric samples from both exhaust-dyed and dope-dyed materials were cut and placed in contact with a standard multifiber adjacent fabric DW. The dyed specimen and multifiber was sewn or secured together to maintain intimate contact. The prepared samples were subjected to a sublimation fastness test by placing them between heated plates for 30 seconds at temperature 180 and 210°C in Preso Hot machine. After heating, the samples were allowed to cool. Samples were then analyzed for staining in adjacent

multifiber fabric, and the specimen's color changed with time.

2.2.5. Colour Fastness to Washing

Specimens of the dyed meta-aramid yarn were prepared for testing color fastness to washing. Each sample was cut and mounted onto a multifiber adjacent fabric. The color fastness to washing was determined using the standard ISO 105 C10:2006 (Test Method B, C, E), which simulates a severe wash condition typically seen in industrial or protective garment cleaning. Test conditions for different method is listed in Table 1. The test was conducted in a laboratory washing machine Roaches Washtec-P. Following the wash, the composite samples were rinsed thoroughly under cold running water, hydro-extracted, and dried at room temperature. Samples were then investigated for staining in adjacent fabric and change in sample color.

Table 1 : Parameters for Washing Fastness Test

Method	B	C	E
Temperature (°C)	50	60	95
Time (min)	45	30	240
Steel balls (No.)	0	0	10

2.2.6. Colour Fastness to Light

The color fastness of the dyed meta-aramid samples was assessed by exposure to a Xenon arc light source in Q-Sun Xe-3-HDS Xenon Test Chamber . Specimens of the dyed yarn were mounted onto cards. The test was conducted by progressively exposing and covering sections of the test specimen for a period of 4 hours. These were then analyzed for fading of the sample.

2.2.7. Heat Treatment

Samples dyed with different methods were heated to the temperature of 300°C for 10 minutes in muffle furnace and then compared with the color value of the original dyed sample for any change in color or tone.

3. Results and Discussions

3.1. Dye stripping from dyed samples

Dye stripping is one of the simplest and most reliable tests to distinguish exhaust-dyed yarn from dope-dyed yarn because the way color is incorporated into the fiber is fundamentally different. The dye stripping from both dope-dyed and exhaust-dyed samples of meta-aramid was carried out in DMF. The treated samples are shown in Figure 1 and the reduction in color strength value for both samples after stripping was analyzed as shown in Table 2. The dye bleeding from both the sample takes place, but it is observed minimal in dope-dyed samples but significant in exhaust-dyed sample. The significant color change demonstrate that the dye in exhaust-dyed aramid is more accessible to the solvent. Meta-aramid fibers are known to have a highly thermostable crystalline structure with strong intermolecular forces.

While polar solvents like dimethylformamide (DMF) can swell the structure by forming hydrogen bonds with the aramid chains [16]. This can facilitate dye removal from the surface and amorphous regions. However, In dope dyeing, the colorant is added to the spinning solution (dope) prior to fiber formation, thus dye is mechanically encapsulated and locked within the crystalline molecular matrix of the aramid fiber as it solidifies during the spinning process. This internal encapsulation shields the dye from chemical attack, making it highly inaccessible to the stripping solvent (DMF) under the test conditions. Therefore, the stripping test acts as a chemical diagnostic tool to differentiate between the surface-fixed nature of exhaust dyeing and the matrix-embedded nature of dope dyeing, but the amount of dye stripping may mislead to confirm if stripping is from dope dyed and exhaust dyed samples. Thus it can not be confirmative test methods, however it can be a supporting test method with other test methods.



Figure 1 (a) Exhaust dyed sample, (b) Dope dyed sample before (left) and after (right) dye stripping test

Table 2 : Color Value (K/S) of samples before and after stripping

Sample/ Colour Strength	Before Stripping	After Stripping	Change in Colour Strength%
Dope Dyed Yarn	6.52	5.93	9.1
Exhaust Dyed Yarn	10.41	6.41	38.4

3.2. Analysis of dye sublimation

A sublimation fastness test evaluates the stability of the color in a dyed testiles when exposed to high temperature and dry heat. It checks whether the dye vaporizes, migrates, or transfers to an adjacent fabric. Color fastness to sublimation was performed on dope dyed and exhaust dyed samples at temperature 180° and 210° C for 30 seconds. The tested samples is shown in Figure 2. It is clear fom the figure that no staining in adjacent fabric is observed. This is because aramid fiber is resistant to heat, due to which it does not get affected with testing temperature.

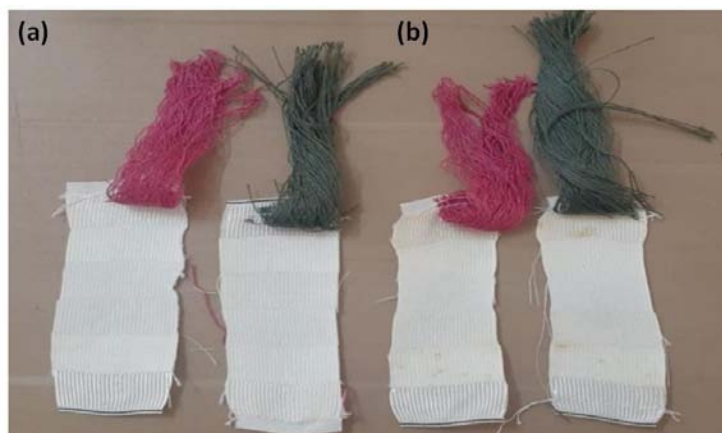


Figure 2 : Exhaust dyed (left) and dope dyed (right) sample at (a) 180°, (b) 210°C of sublimation fastness test

3.3. Analysis of color fastness to washing

Color fastness to washing was carried out to differentiate dope-dyed and exhaust-dyed samples because dope-dyeing incorporates color within the fiber core, resulting in superior retention during washing. In contrast, exhaust-dyed materials may have surface adsorbed or partially fixed dye that can wash off, leading to shade change and staining. The color fastness to washing of meta-aramid samples were investigated by using IS/ISO 105 C10:2006method. The samples tested with method B and C is shown in Figure 3. It is cleary seen from the figure that no staining in adjacent fabric was observed. Again this is due to the fact that mild conditions of washing does not have any impact on aramid yarn.



Figure 3 Exhaust dyed (left) and dope dyed (right) sample tested with (a) Method B, (b) Method C

3.4. Analysis of color fastness to light

Colour fastness to light was performed to differentiate between dope-dyed and exhaust-dyed samples because in exhaust dyed samples, dye present on the surface are more exposed to external environmental conditions as compared to the dye present in dope dyed samples, leading to distinct

differences in light durability. The samples were exposed to UV light for 4 hours. However the change in colour starts observing within one hour of exposure as shown in Figure 4(a). The change in the color value of aramid samples concerning exposure time is shown in Figure 4(b). In the case of exhaust-dyed samples, a reduction in color value was observed from 8.4 to 3.7 after 4 hours of exposure while in the case of dope-dyed yarn, no major change in color value was observed. This is because in case of exhaust dyed samples, surface dye interact with the photon of xenon arc light causes photochemical reaction that lead to the degradation of the dye molecule [17].

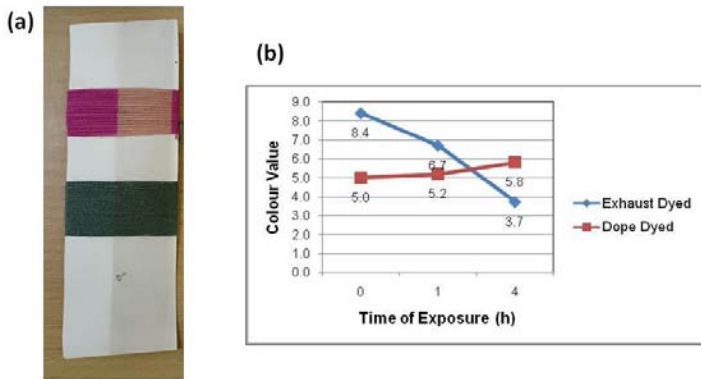


Figure 4 (a) : Exposed sample (b) Colour value (K/S) of exhaust dyed and dope dyed aramid yarn for 4 hours exposure

3.5. Effect of high temperature for a short time

Direct exposure of dye to high temperature leads to the degradation of dye present on the exhaust dyed sample. While colorant or pigments used in dope dyed sample are stable at high high temperature of polymer melting and spinning during its production process. So direct exposure of exhaust dyed and dope dyed samples were done at high temperature of 300°C in muffle furnace for 2 minutes was done to see the effect of high temperature on the colour change was observed. The treated sample of exhaust dyed and dope dyed is shown in Figure 5(a) and (b) respectively and colour value is shown in Table 3. There is clear colour change observed for exhaust dyed sample with decrease in colour value while no such change is observed for dope dyed samples.

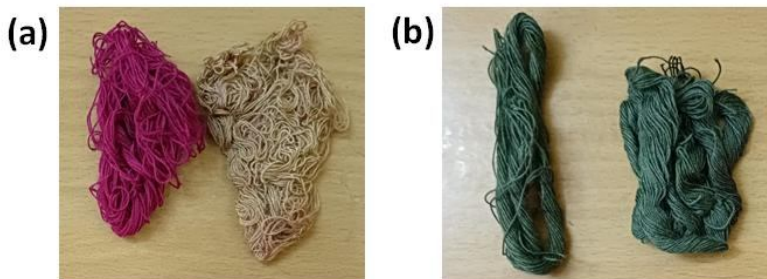


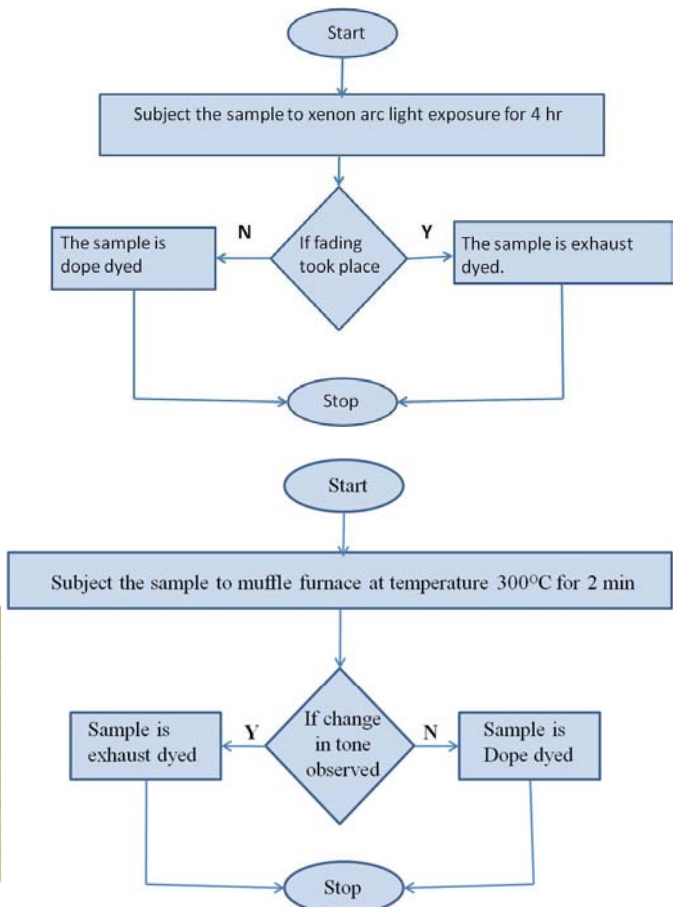
Table 3 : Colour Value of dyed meta-aramid yarn before and after heat exposure

Particulars	Before heat exposure	After heat exposure
Meta-aramid Cationic Exhaust Dyed Yarn	20.73	3.90 (Tone change)
Meta-aramid Dope Dyed Yarn	6.58	6.59

4. Conclusions

The investigations and obtained results conclude that the color change to sublimation and vigorous washing cycle does not provide any conclusive result for identification of dope dyed and exhaust dyed samples. In the other way, color fastness to sublimation and exposure to high temperature for a short period were the turning point of this work to confirm the dyeing process of meta-aramid sample quickly. The complete change in tone of the dyed sample was observed at high temperatures, whereas no change was observed in the dope-dyed sample. Based on this result, a flow chart is shown below for a confirmative quick method to differentiate the dyeing process from the dyed meta-aramid sample within a short time.

Flowchart 1 : Light fastness test



Flowchart 2 : Heating sample in a muffle furnace at temperature 300°C for 2 minutes

Conflict of interest

The authors declare no conflict of interests

Author contributions

KK performed the experiments, including sample preparation and testing. PKP conceptualized the study and supervised the research. All authors contributed to the preparation and review of the manuscript.

Data availability statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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