Research article

Effect of dye bath pH in dyeing of cotton knitted fabric with reactive dye (Remazol Yellow RR) in exhaust method: impact on color strength, chromatic values and fastness properties

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HIGHLIGHTS

- Dye bath pH has played a significant role in cotton fabric dyeing with reactive dyes (Remazol Yellow RR).
- The physical and chemical test characteristics of the dyed fabric continuously change along with the dye bath pH.
- The color strength (K/S), chromaticity (C), L*, a*, b* and visual uniformity values show significant results.

ARTICLE INFO

Keywords:
Reactive dyes
Cotton fabric
Dyeing
Color strength
pH
Caustic soda
Color fastness

ABSTRACT

The significance of textile coloration is inevitable as coloration is a critical process and it can be affected by several parameters i.e. time, temperature, pH, and liquor ratio (M:L). This research reveals the behavior of different dye bath pH (2 to 12) on the basis of spectrophotometric characteristics and colorfastness using the most useable brand of reactive dye in Bangladesh i.e. Remazol Yellow RR. Earlier researches were performed on the basis of other coloration controlling factors i.e. temperature, time and liquor ratio (M:L) where pH acts as the dependent variable. However in this study, pH was taken as an independent variable where lower pH (2, 4 and 6), medium pH (8 and 10) and higher pH (12) were taken into consideration for verifying the dyeing performance. Meanwhile, the mentioned pH was achieved by adding acetic acid, soda ash and caustic soda respectively. In this study, the impact of pH is analyzed on the basis of color strength, chromaticity, hue angle, CIELAB color space, spectrophotometric image, reflectance percentage and color fastness tests i.e. color fastness to rubbing, color fastness to wash, color fastness to perspiration, color fastness to light. Gradually elevated pH boosts up the color strength and chromaticity, at the same time downgrades of hue angle results orangish tone in the dyed fabric. Even more, the lightness of dyed fabric decreases and redness rises with the increment of dye bath pH which results in orangish red darker tone of dyed fabric. Consequently, fabric dyed at acidic dye bath pH exhibits higher resistance in case of color change and color staining from the dyed fabric and multifiber fabric respectively and vice versa.

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https://doi.org/10.1016/j.heliyon.2022.e11246
Received 22 April 2022; Received in revised form 3 July 2022; Accepted 20 October 2022
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1. Introduction

Cotton is the most admirable fiber among all fibers due to its excellent hand feel, absorbency, availability and globally inexpensiveness. Cotton is a highly valued cash crop due to its supreme α-cellulose % and lesser lignin % in its chemical content. As an anionic dye, reactive is the best choice with cotton fiber coloration due to its bright and brilliant color in various shade ranges with excellent color fastness (Hosen et al., 2021). Exhaustion and fixation are the stages by which reactive dyes are easily applied to cotton (Bafoun, 2019). Exhaustion can be attained by the addition of Glauber’s salt (Na2SO4) or common salt (NaCl). Fixation can be done by adding sodium hydroxide (NaOH) or soda ash (Na2CO3) (Alam et al., 2008).

Reactive dyes are contended by a reactive group which creates a covalent bond with the hydroxyl and amino group of the cotton fiber. It was found that dyeing temperature and pH increase with the increases in the reactivity of the reactive group and vice-versa (Ahmed et al., 2020). The fixation % of the reactive dye is 60–90% as well as dyeing and washing off required enormous amount of water (Bahadir, 2007). Researchers performed various types of research based on different points of view. Reactive dye is highly compatible & it is explored more effectively through exhaustion, fixation and adsorption isotherm based on three primary colors which was explained by pigment theory. The blue color was found as better an exhaustive color although fixation is not praiseworthy. But the red color was detected as a highly exhaustive color and also carries good fixation%. In deep concentration, three primary i.e. blue, red and yellow are less compatible (Naser et al., 2015). Sustainability of dyeing processes with the reactive dyes, different effluent treatment methods, formulation and use of different biodegradable organic compounds in dye bath during dyeing, before the dyeing process chemical modification of the cotton fibers, modification of the dyeing methods and the machinery was summarized (Khatiri et al., 2015).

Mainly, the significance and the limitations of these ways improved and developed to increase the sustainability during reactive dyeing. The color strength (K/S) and color fastness properties are governed by alkali concentration (Paul et al., 2017). They revealed that with the elevation of alkali concentration color strength (K/S) increases but color strength (K/S) decreases at alkali concentration (10 gm/L). Developed dyeing technology has various advantages of high dye utilization and lowly-colored dyeing wastewater discharge, which is beneficial to protecting the environment (Shu et al., 2019). Active species of photo-generated holes are responsible for the degradation of the reactive dyes (Sane et al., 2018). The reactive dyeing of cotton is exerted by the ionic strength of the dye solution rather than the concentration of inorganic salts used as an electrolyte (Khatiri et al., 2018). The effect of concentration of nonionic surface-active agent on the hydrolysis of vinyl sulphone reactive dyes (Cai et al., 2015). Bread and age effects on the dye fixation on wool using a reactive dye and optimization of the dyeing process (Bouagga et al., 2019). During the decolorization of the reactive dyes on the intelligent nano-engineer of nanostructures pH responsive (Ahmed et al., 2020). Adsorption of the reactive (anionic) dye on the basic sample is favored and calculating the K/S values during the initial dyeing at low dye concentrations is carefully carried out (Becerin, 2005; Orfão et al., 2006).

In this research work, performance of pH in reactive dyeing was analyzed from technical point of view based on spectrophotometric data analysis and colorfastness test. This research work will help dyer to take decision by anticipating the final required shade of the most useable brand of reactive dye i. e. Remazol Yellow RR. The effects of pH on dyed fabric were measured by assessment of color strength (K/S), chromaticity, hue angle, CIELAB color space, spectrophotometric image, visual uniformity, reflectance %, color fastness to washing, color fastness to water, color fastness to perspiration both acid and alkali, color fastness to rubbing, color fastness to light respectively. Comparatively highest wash and water fastness rating was achieved at the lowest dye bath pH 2 and 4, the highest (4–5) perspiration fastness rating was achieved at the lowest dye bath pH 2 and 4, better-wet rubbing fastness rating has been found at alkaline pH, color fastness to light rating and color strength value increases with the elevation of dye bath from pH 2 to pH 12 and so on.

2. Materials and methods

2.1. Materials

100% cotton scoured bleached knitted fabric (constructional structure: single jersey, number of course per inch (CPH) is 51, number of wales per inch (WPI) is 32, stitch length is 2.76 mm, the weight of the fabric is 150 GSM and the fabric count is 28 Ne) was sourced from NZ Textile Ltd., Narayanganj, Bangladesh. In these research, Remazol Yellow RR was used as reactive dye, ALBATEX® DBS (Huntsman, UK) were used as leveling agents, Matquest 1090 liquid (Matex, Bangladesh) was used as a sequestering agent, Glauber salt (Na2SO4·10H2O) was used as an electrolyte, Soda ash (Na2CO3) and caustic soda (NaOH) is used to control the pH. Mat wash DSP (Matex, Bangladesh) was used as a soaping agent (detergent) and acetic acid was used to achieve acidic pH and neutralization purposes. All the chemicals were laboratory grade and used without any purification.

2.2. Methods

In this comparative study, single bath scoured and bleached fabric (10 gm) was dyed with Remazol Yellow-RR by conventional exhaust dyeing method. The addition of glauber salt was performed as per the directive of the technical data sheet of the supplier. In this research work, pH was varied and effect analysis was performed. Here pH 2, 4, 6, 8, 10 and 12 were achieved by the addition of 7 gm acetic acid, 5 gm acetic acid, 5 gm soda ash, 8 gm soda ash and 11 gm caustic soda at the dye bath respectively by keeping other recipe content as Remazol Yellow RR-1%, sequestering agent 2 gm/L, leveling agent 1 gm/L, glauber salt 50 gm/L, acetic acid 1 gm/L, detergent 2 gm/L with liquor Ratio 1:20.

2.2.1. Conventional exhaust dyeing procedure

The process curve of conventional exhaust dyeing and the fabric dyeing procedure is shown in Figures 1 and 2 respectively. Dyes, glauber salt, sample fabric, leveling agent, sequestering agent and water were loaded at room temperature in a sample dyeing machine. Then the temperature was raised to 45 °C and the sample dyeing machine run for 40 minutes. After that soda ash or caustic soda or acetic acid was added to the dye bath to achieve the required pH. Later the dye bath temperature was raised to 60 °C by 1.5 °C/min and dyeing was continued for 60 min. After completion of dyeing, cooling was done at 40 °C and dye liquor was drained out. The dyed fabric was treated by 5 minutes cold wash and consecutive hot wash at 50 °C temperature for 10 minutes followed by neutralization with 1 gm/L acetic acid solution at 45 °C for 10 minutes then soaping with 2 gm/L Mat wash DSP at 90 °C for 10 minutes. Finally, the samples were rinsed with normal water for 10 minutes and followed by drying.

2.2.2. Measuring color strength and chromatic values of dyed fabrics

The color strength (K/S value) and CIELAB colorimetric values (L*, a*, b*, C and h°) of the samples were measured using the color measuring spectrophotometer (Model No. Data Color 650® USA) with the illuminant D-65 and 10° observer. The K/S value was calculated by the CIE Kubelka-Munk equation.

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

Where, R is the reflectance at any specific wavelength.

2.2.3. Measurement of color fastness properties

The testing methods of the different pH dyed samples are shown in Figure 3. The colorfastness test properties of all dyed samples were carried out in accordance with the ISO standard. Colorfastness to washing, water and perspiration was determined in accordance with the test method of ISO 105-C06, ISO 105-E01 and ISO 105-E04 standards respectively. Where, the numerical values 5, 4, 3, 2 and 1 indicate excellent, good, fair, signification staining and deep staining respectively.
The colorfastness to rubbing was determined in accordance with the test method of ISO 105-X12 (dry and wet) standard and colorfastness to light test was followed by the ISO 105-B02:2013 standard. The color strength of the dyed samples was measured by the K/S value.

3. Results and discussion

3.1. Effect of dyebath pH on color strength, chromaticity and hue angle of the dyed fabric

The color strength indicates the relative measure of the amount of dye in the fiber. The chroma is the attribute of color used to indicate the degree of depth of the color from the gray of the same lightness. It can be termed as the colorfulness of an object. The higher chroma indicates more colorfulness on an object. Every color on an object is expressed by an angle ranging from 0° to 360° in CIELAB color space. The impact of dyebath pH on color strength, chromaticity (C) and hue angle of the samples was observed and plotted as shown in Figure 4(a), Figure 4(b) and Figure 4(c) respectively. The amount of dye in the fiber increased with the increase of dye bath pH. The dye bath pH changed the chromaticity of the dyed samples differently. The higher pH produced more colorful samples with higher chromaticity in most of the cases. Hue angle of the dyed fabric decreased with the increase in dyebath pH. As a result, the physical appearance of the dyed samples became orangish with the gradual increase in dye bath pH.

3.2. Effect of dyebath pH on L*, a*, b* values and spectrophotometric image of the dyed fabric

The colors of the dyed materials were specified using CIELAB color space. The value of L* in the CIELAB system, recommended by CIE in 1976 gives a measure of the lightness of the color. The L* value varies between 0 (perfect black) and 100 (perfect white). The impact of dyebath pH on L* value, a* value and b* value of the dyed fabric samples was observed and plotted as shown in Figure 5(a), Figure 5(b) and Figure 5(c) respectively. Figure 5(d) shows the spectrophotometric image of dyed fabric at different dye bath pH. The higher value of L* denotes the lighter hue and the lower value refers to the darker hue of a dyed material. The value a* denotes the redness or greenness quality of the dyed fabric. The positive value of a* denotes the redness and the negative value of a* denotes the greenness quality of the dyed fabric. The value b* denotes the yellowness or blueness quality of the dyed fabric. The positive value of b* denotes the yellowness and the negative value of b* denotes the blueness quality of the dyed fabric. From the present study, it was found that dye bath pH changed the lightness of the dyed fabric in a regular manner. The lightness of the dyed samples decreases with the increase of dye bath pH. The dyebath pH has little impact on the b* values of the dyed samples but it has a notable influence on the a* values of the dyed samples. The dyed samples got redder with the increases in dyebath pH which is also visible in the spectrophotometric images of the dyed fabric.

3.3. Impact of dye bath pH on visual uniformity of the dyed fabric

Another effect is the visual uniformity of the different dyed samples. Visual uniformity of reactive dyed samples at different pH is shown in Figure 6. Here, Figure 6 shows the effect of dye bath pH-2 to pH-12 on the visual uniformity during producing yellow shade (Remazol Yellow RR 1%) for cotton knit fabric. Figure 6 shows the dye bath pH 2 to 6 gave very good uniformity compared to the pH 8 to 12. The samples of pH 2 to 6 give good uniformity because it contains an acidic condition. The dye bath pH 8 to 12 treated fabric shows a dark shade in comparison to the...
acidic medium dyeing. Here, the pH 2, 4, 6 conditions fabric samples show uniform color with pure yellowish and above pH 8 to 12 it shows reddish color.

3.4. Effect of dye bath pH on reflectance % of the dyed fabrics

The reflectance percentage of the dyed fabric samples at different pH is shown in Figure 7. Figure 7 reveals maximum diffuse reflectance at 700 nm which is considered as \( \lambda_{\text{max}} \) for the below spectral profile. The ratio of reflected radiant flux to incident flux stands at the outermost location for pH 2 and the innermost location for pH 10. Textile coloration at acidic dye bath pH results in the formation of less covalent bonds among cellulose and reactive group of reactive dye and vice versa. The concentration of dye at the core of cellulose fiber is higher for fabric dyed at alkaline pH and vice versa as well. As a result, reflected radiant flux is highest at pH 2 and lowest at pH 12.

3.5. Impact of dye bath pH on color fastness properties of the dyed fabrics

The color fastness of a dyed fabric is the most important properties which determines the performance level of particular dyes or colorants for textile application. In order to be conversant with the performance
the colorfastness to washing, perspiration, rubbing and light were determined and discussed in the following subsections.

3.5.1. Impact of dye bath pH on color fastness to wash of the dyed fabrics

The gray scale ratings of the dyed fabric in terms of the color change and the color staining are shown in Table 1. Samples dyed at acidic pH exhibited more resistance against washing with a gray scale rating of 4–5 to 4. On the contrary, samples dyed at alkaline pH exhibited poor resistance against washing with a gray scale rating of 3–4. The reason behind this phenomenon might be the presence of a less amount of dyes in the samples dyed at acidic dye bath pH, so there is less chance of desorption of dyes from the samples after washing and vice versa. The samples dyed at various dye bath pH stained no color in cotton, polyester and polyacrylonitrile portions of the multifiber fabric. Nevertheless, the fabrics dyed at alkaline pH exhibited less resistance against staining on polyester-acrylic and wool portions of the multifiber fabric and vice versa.

3.5.2. Impact of dye bath pH on color fastness to water of the dyed fabrics

The Table 2 reveals the gray scale ratings of the dyed fabric in terms of color change and color staining. Dye bath pH has also a substantial effect on the color fastness to the water of the dyed fabrics. Samples dyed at acidic pH exhibited more resistance against the water with a gray scale rating of 4–5. In contrast, samples dyed at high alkaline pH exhibited poor resistance against washing with a gray scale rating of 3–4. The reason behind this phenomenon might be a less amount of dyes in the samples dyed at acidic dye bath pH. So, there is less chance of desorption of dyes from the samples against water and vice versa. The samples dyed at various dye bath pH show a little or no color staining with gray scale rating 4, 4–5 and 5 on cotton, cotton acrylic, polyester and poly-acrylonitrile portions of the multifiber fabric. In addition, the fabrics dyed at alkaline pH exhibited less resistance against staining on polyester acrylic and the wool portion of the multifiber fabric and vice versa.

3.5.3. Impact of dye bath pH on color fastness to acid perspiration

The values are shown in Table 3 reveal the gray scale ratings of the dyed fabric in terms of color change and color staining. As like with other fastness properties, dye bath pH has significant effects on acid perspiration of the dyed fabrics. Samples dyed at acidic pH exhibited more resistance against acid perspiration with a gray scale rating of 4 to 4–5. In contrast, samples dyed at high alkaline pH exhibited poor resistance against washing with a gray scale rating of 3–4. The reason behind this phenomenon might be a less amount of dyes in the samples dyed at acidic dye bath pH. So, there is less chance of desorption of dyes from the samples against acid perspiration and vice versa. The samples dyed at various dye bath pH shows a little or no color staining with gray scale rating 4, 4–5 and 5 on cotton, cotton acrylic, polyester and poly-

Figure 5. Impact of dye bath pH on: (a) L* value (b) a* value (c) b* value and (d) spectrophotometric image of dyed fabric.

Figure 6. Visual uniformity of reactive dyed samples at different pH.

Figure 7. Impact of dye bath pH on reflectance% of dyed fabric.
acrylonitrile portions of the multifiber fabric. In addition, the fabrics dyed at alkaline pH exhibited less resistance against staining on the polyester acrylic and wool portion of the multifiber fabric and vice versa.

### 3.5.4. Impact of dye bath pH on color fastness to alkali perspiration

The gray scale ratings of the dyed fabric in terms of color change and color staining is listed in Table 4. The samples dyed at acidic pH exhibited more resistance against alkali perspiration with a gray scale rating of 4 to 4.5. In contrast, samples dyed at high alkaline pH exhibited poor resistance against washing with a gray scale rating of 3–4. The reason behind this phenomenon might be a less amount of dyes in the samples dyed at acidic dye bath pH. So there is less chance of desorption of dyes from the samples during wet rubbing at acidic dye bath pH and the highest alkaline dye bath pH. So there is more chance of desorption of dyes from the samples during wet rubbing at acidic dye bath pH and the highest alkaline dye bath pH.

### 3.5.6. Impact of dye bath pH on color fastness to light

The impact of dye bath pH on color fastness to light is represented in Figure 9. It is observed that at alkaline pH, cotton fabric is least oxidized by oxygen radicals produced by high energy radiation from the sun and vice versa. In the plotted Figure 9, the color fastness to light of the dyed samples of different pH is noticeable due to color bleaching or fading. In reference to Figure 4(a) it can be said that more color strength denotes more presence of color bearing chromophore in fabric samples and vice versa. It is usual that samples of different dye bath pH will experience equal no of breakage of color bearing chromophore as they are exposed for the same time against light. Once samples of different dye bath pH are exposed to sunlight for a certain time then more chromophore bearing samples of higher dye bath pH experiences less bleach out or fading of color and vice-versa. Consequently, the best result (5 in rating scale) is achieved at highest alkaline condition (pH 12) and lowest result (2 in rating scale) is achieved.

### Table 1. Impact of dye bath pH on color fastness to wash of the dyed fabrics.

| pH Values | Grade (Color Change) | Grade (Color Staining on Multi-fiber) |
|-----------|----------------------|--------------------------------------|
|           | Cotton Acrylic | Cotton | Poly-Lester Acrylic | Polyester | Poly-Acrylonitrile | Wool |
| 2         | 4-5       | 5       | 4-5               | 5         | 5                   | 4-5  |
| 4         | 4-5       | 5       | 4-5               | 5         | 5                   | 4-5  |
| 6         | 4         | 5       | 4                 | 5         | 5                   | 4    |
| 8         | 3-4       | 4       | 5                 | 3-4       | 5                   | 3-4  |
| 10        | 3-4       | 4       | 5                 | 3-4       | 5                   | 3-4  |
| 12        | 3-4       | 4       | 5                 | 3-4       | 5                   | 3-4  |

### Table 2. Impact of dye bath pH on color fastness to water of the dyed fabrics.

| pH Values | Grade (Color Change) | Grade (Color Staining on Multi-fiber) |
|-----------|----------------------|--------------------------------------|
|           | Cotton Acrylic | Cotton | Poly-Lester Acrylic | Polyester | Poly-Acrylonitrile | Wool |
| 2         | 4-5       | 5       | 4-5               | 5         | 5                   | 4-5  |
| 4         | 4-5       | 5       | 4-5               | 5         | 5                   | 4-5  |
| 6         | 4         | 5       | 4                 | 5         | 5                   | 4    |
| 8         | 4         | 5       | 4                 | 5         | 5                   | 4    |
| 10        | 3-4       | 4       | 5                 | 3-4       | 5                   | 3-4  |
| 12        | 3-4       | 4       | 5                 | 3-4       | 5                   | 3-4  |

### Table 3. Impact of dye bath pH on color fastness to acid perspiration of the dyed fabrics.

| pH Values | Grade (Color Change) | Grade (Color Staining on Multi-fiber) |
|-----------|----------------------|--------------------------------------|
|           | Cotton Acrylic | Cotton | Poly-Lester Acrylic | Polyester | Poly-Acrylonitrile | Wool |
| 2         | 4-5       | 5       | 4-5               | 5         | 5                   | 4-5  |
| 4         | 4-5       | 5       | 4-5               | 5         | 5                   | 4-5  |
| 6         | 4         | 5       | 4                 | 5         | 5                   | 4    |
| 8         | 4         | 5       | 4                 | 5         | 5                   | 4    |
| 10        | 3-4       | 4       | 5                 | 3-4       | 5                   | 3-4  |
| 12        | 3-4       | 4       | 5                 | 3-4       | 5                   | 3-4  |
at acidic condition (pH 2 and pH 4). Moderate result (3 and 4 in rating scale) is obtained at pH 6, 8 and 10.

4. Conclusions

Dye bath pH has a remarkable impact on achieving higher color strength in fabric. Determination of specific pH to achieve higher color strength as well as satisfactory light fastness rating at a particular pH is the novelty of this research work. After turning the fabric in different pH environments, it was concluded that good color strength and remarkable lightfastness were achieved at pH 12 using reactive yellow dye. Hence, specific pH determination has solved the major problem, i.e. poor or moderate lightfastness of reactive dyed fabric enhanced dyers to attain maximum color strength and lightfastness to secure the right first time (RFT) in dyeing.

Table 4. Impact of dye bath pH on color fastness to alkali perspiration of the dyed fabrics.

| pH Values | Grade (Color Change) | Grade (Color Staining on Multi-fiber) |
|-----------|----------------------|---------------------------------------|
|           |                      | Cotton Acrylic | Cotton | Polyester Acrylic | Polyester | Poly-Acrylonitrile | Wool |
| 2         | 4-5                  | 4-5           | 5      | 4-5              | 5         | 5                  | 4-5  |
| 4         | 4-5                  | 4-5           | 5      | 4-5              | 5         | 5                  | 4-5  |
| 6         | 4                    | 4             | 5      | 4                | 5         | 5                  | 4    |
| 8         | 4                    | 4             | 5      | 3-4              | 5         | 5                  | 4    |
| 10        | 3-4                  | 4             | 5      | 3-4              | 4-5       | 3-4                | 3-4  |
| 12        | 3-4                  | 3-4           | 4-5    | 3-4              | 4-5       | 3-4                | 3-4  |

**Figure 8.** The color fastness to dry and wet rubbing at different pH.

**Figure 9.** The color fastness to light at different pH.

Declarations

Author contribution statement

A.T.M. Gulam Moula, Md. Dulal Hosen, Md. Abdullah Al Mamun: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Md. Abu Bakar Siddiquee, Zihan Kaisar: Contributed reagents, materials, analysis tools or data.

Md. Abdul Momin: Performed the experiments; Contributed reagents, materials, analysis tools or data.

Md. Azharul Islam: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Funding statement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Data availability statement

Data included in article/supp. material/referenced in article.

Declaration of interest’s statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.
Acknowledgements

The authors gratefully acknowledge the support and guidance provided by the Department of Textile Engineering, Mawlana Bhashani Science and Technology University, Tangail, Bangladesh and Uttara University, Dhaka, Bangladesh for carrying out the research work. The authors also like to thank the NZ Textile Limited for providing technical support. Finally, the authors express gratitude towards the reviewers for their extensive review in improving the quality of the manuscript.

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