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Mercerisation Followed by Wet on Wet Reactive Dyeing of Cotton Fabric

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ABSTRACT
The commonly employed textile wet processing methods are singeing, de-sizing, scouring, bleaching, mercerising, dying, printing, and finishing. However, these methods are more time-consuming and require a lot of energy, manpower, and chemicals. Therefore, this research aims to develop a semi-continuous processing route that minimizes these problems. In this study, two dying routes were employed. The first is a control group, which was done by conventional mercerisation and dyeing procedures, whereas the second is a semi-continuous experimental group, which has been employed by mercerisation followed by wet-on-wet (WOW) reactive dying. That is, after mercerisation, the fabric, which contains the optimum alkali concentration, goes to dyeing without washing. Washing and titration were performed to determine the optimum alkaline concentration in the fabric at a specific washing cycle. Fabrics that were washed at different cycles after mercerisation were dyed, and their washing fastness, degree of dye exhaustion, fixation, and colour strength were evaluated, and they had a slightly greater colour strength than conventionally dyed fabrics. This is because with a wet-on-wet dyeing procedure, the fabric is relaxed after mercerisation, which allows it to absorb more dye, and the leftover alkali in the fabric is used to fix the dye to the cellulose. The study shows that the optimal washing cycle for passing the optimum alkali concentration is one. Furthermore, the optimal amount of alkali remaining in the fabrics is 3–3.5%. As compared to the conventional dyeing process, this process is economical.

KEYWORDS
mercerisation, wow reactive dyeing, fixation, cotton

INTRODUCTION

In the wet processing of textiles, the commonly employed steps are pretreatment, dying, printing, and finishing [1-3]. Sometimes mercerisation is done additionally for cotton fabrics, either as a pretreatment for dyeing as a finishing process or after dying for lustre [4,5]. It is the treatment of cotton fabric with either liquid ammonia or a strong caustic alkaline. This can be applied by two methods, which are cold and hot mercerisation under tension or relaxation. However, when compared in terms of affinity, cotton that mercerises in hot conditions has a higher affinity for dyes than cotton that mercerises in cold conditions, but cotton that mercerises under high tension has a lower affinity for dyes than cotton that mercerises under relaxation conditions [5-7]. This result might be connected to the crystalline areas being destroyed when the mercerised cotton swells and changes structurally

[8]. This treatment not only improves colour uptake but also increases the amorphous area, tensile strength, moisture recovery, number of free hydroxyl groups, surface impurity of fabrics, and enhanced colour fastness [5,8-10].

When the fabric needs to be mercerised before dyeing, the commonly utilized methods entail impregnating it with sodium hydroxide solution, squeezing it to a wet pickup of around 100%, and then washing it with running water. After being gently washed and neutralized in dilute hydrochloric acid, the mercerised cotton samples are allowed to dry outside [5,11-13]. Then it goes to the dyeing process. In this stage, the fabric can be dyed with reactive dyes through a standard dyeing procedure. Salt and alkali can be added to the dye bath under proper circumstances to enhance exhaustion and fixation of the dye molecules with the substrate, respectively. Finally, the dyed samples can be washed using a non-ionic detergent solution [14,15]. Dyes make a covalent link with cotton via nucleophilic substitution or addition and are known as substitutive and additive dyes, respectively. Because of the strong dye-fibre connection, fastness attributes are remarkably good, except for wash fastness, which is poor to moderate due to dye hydrolysis [15].

However, the usual continuous process of mercerisation to dyeing of cotton fabric involves mercerising, washing, drying, dyeing, washing, and drying (curing). This method uses a lot of energy, takes a long time, and requires more chemicals. Therefore, finding a solution to this serious problem is a concern for scholars and manufacturers. Consequentially, this study mainly focused on reducing washing, drying, and auxiliary chemicals from the conventional dyeing process by developing semi-continuous processes of mercerisation followed by wet-on-wet reactive dyeing of cotton fabrics. A comparison was made between the innovative dyeing procedure and conventionally dyed fabric using different parameters such as degree of fixing and degree of exhaustion.

EXPERIMENTAL

Materials and methods

Fabric sample specifications

For this study, 100% cotton fabric was produced at the Bahir Dar Textile Share factory in Bahir Dar, Ethiopia, under the specification that is illustrated in Table 1. The weft yarn is rotor-spun, and the warp yarn is ring-spun.

Table 1. Fabric sample specification

	Value
Fabric structure	Plain
Width (cm)	170
Warp yarn density (end/cm)	24
Weft yarn density (pick/cm)	21
Warp yarn count (Tex)	28.11
Weft yarn count (Tex)	28.11
Total number of warp ends	3330

Chemicals and auxiliaries

The required chemicals that were utilized for the mercerisation and dyeing processes were collected and illustrated in Table 2, and their specific application areas were also indicated there.

Table 2. Fabric sample specification

Chemicals	Used for;
Caustic Soda (NaOH)	mercerisation and dye fixation
Acetic acid (CH_3COOH) / Hydrochloric acid (HCl)	Neutralizing of alkaline chemical
Sulfuric acid (H_2SO_4)	Titration
Phenolphthalein ($C_{20}H_{14}O_4$)	Indicators
Heterobifunctional reactive dye (Somazin/Red3B-A)	Dyeing
Sodium sulphate (Na_2SO_4)	Dye exhaustion
Levelling agent	Uniform shedding
Soaping Agent	Washing after dyeing
Wetting Agent	Lower the surface tension of a liquid
Sequestering Agent	Deactivates metal ions

This study followed the general methodological framework shown in Figure 1. Following mercerisation, two experiments were conducted. The first root, known as the control group, goes through the conventional mercerisation-dyeing procedure. After tension mercerisation, the fabric in this experimental group was washed till it became neutralized against alkali. It then undergoes the standard dying procedure after being dried out.

The second method is based on a currently developing experimental procedure known as mercerisation followed by wet-on-wet reactive dyeing. This experimental group receives the same alkali solution treatment as the first group, but following mercerisation, constant washing and titration are carried out until the fabric retains the necessary amount of alkali. Afterwards, conventional dyeing techniques were used to dye it. Each experimental group processing step is detailed and discussed in each section below.

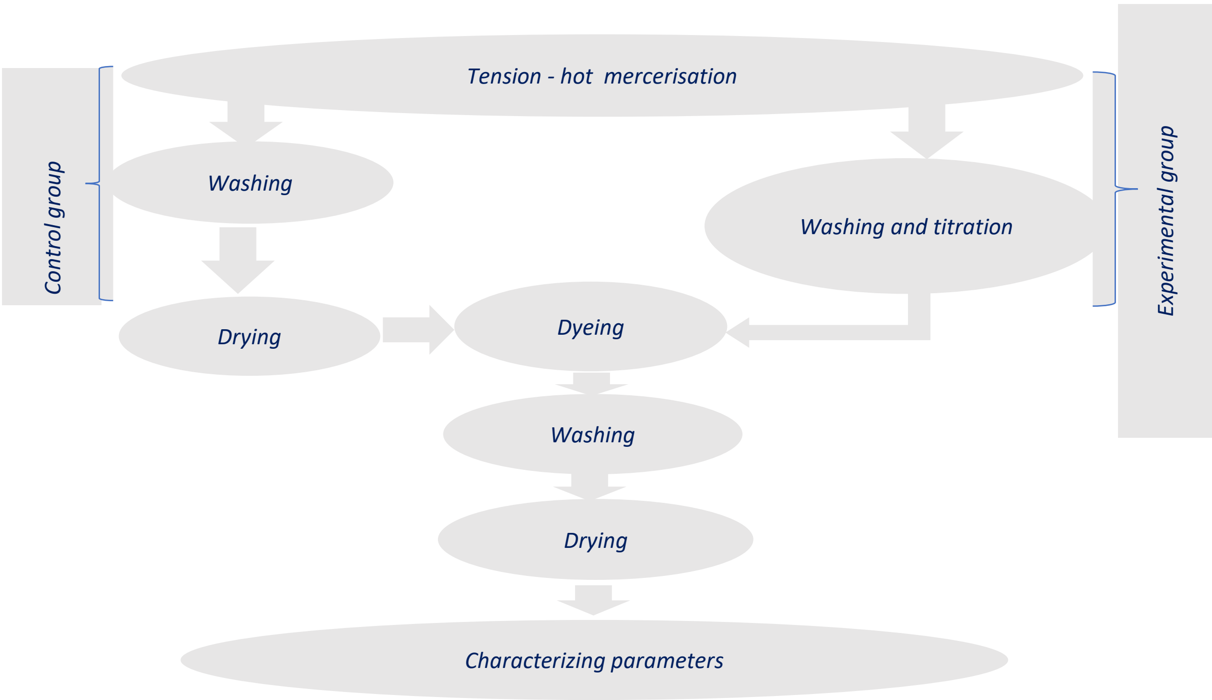


Figure 1. Experimental design

Mercerisation

Cotton fabric samples have been cut to 30 x 30 cm dimensions. On these fabrics, 20 x 20 cm and 10 x 10 cm were marked for dyeing and titration, respectively. According to several scholars [5,8,25] procedures, the fabrics were immersed in a caustic soda solution containing 23% w/w and a liquor ratio of 1:50. The fabrics were then left to stand for 90 seconds at a temperature of 65±2 °C. For common dyeing (control group), the fabric was then twice washed in hot water at 60–70 °C for 5 minutes and then it was washed in water containing 2 g L−1 HCl at room temperature for 5 minutes. Ultimately, it was washed with tap water until it became neutral, and then drying was completed [4]. Similar mercerisation treatments have been carried out for the experimental group. Then, washing and titration were repeated until a sufficient amount of NaOH remained in the fabrics, which was then utilized for fixing in the reactive dyeing process. Here, washing has been done for five minutes under hot water at 60–70 °C. The number of washing cycles used to obtain the optimum caustic soda solution in the fabric was recorded. The titration procedure in detail is covered in the following section.

Acid-base titration

Acid-base titrations are typically used to determine the concentration of a known acidic or basic material using acid-base reactions. The analyte (titrant) is a solution or treated cloth with an undetermined molarity. The reagent (titrant) is a solution of known molarity that will react with the analyte. To make 0.2 M, add 10 g of sulfuric acid (98%) to 500 mL of water.

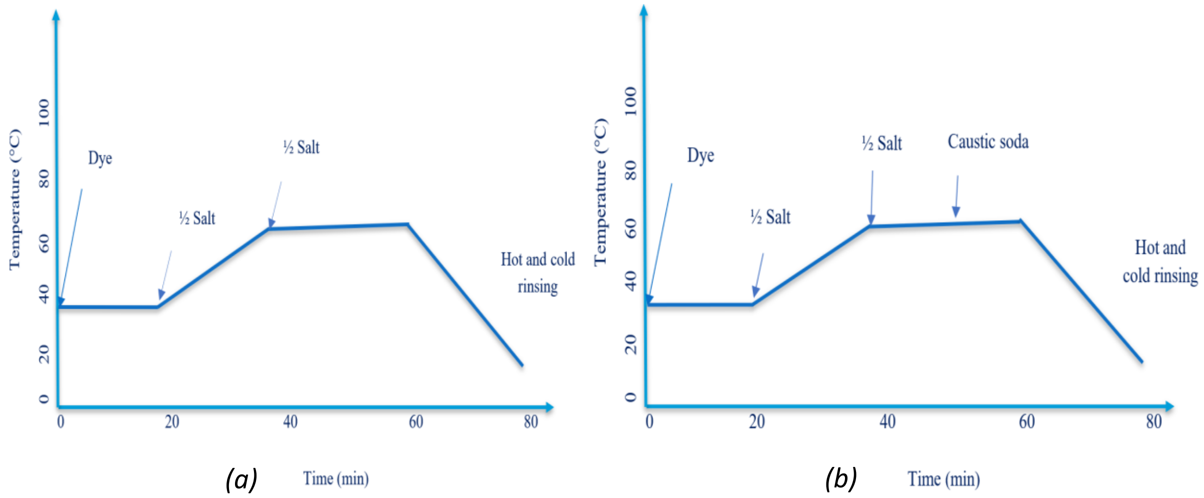
Titration and determination of the molarity of caustic soda in the fabric

All equipment had been washed and dried. Then 60 mL of water was added to the Erlenmeyer flask. In this water, two drops of indicator (phenolphthalein) were added. A funnel was placed on top of the open burette, and a sulfuric acid solution was added. A 10 × 10 cm piece of mercerised fabric is immersed. Drops of sulfuric acid were gradually added to the flask and shaken constantly until the pink hue faded. It was then noted how much sulfuric acid solution (H_2SO_4) was needed. Table 3 displays the results of the determination of the molarity of sodium hydroxide (NaOH) using the chemical equations indicated in Equation 1.

$$2NaOH(aq) + H_2SO_4(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l) \quad (1)$$

Wet-on-wet reactive dyeing

The experimental group cotton fabric samples that were mercerised were dyed by the pad-dyed method with heterobifunctional dye (Somazin/Red 3B-A) (2%) solutions at MLR 1:30 and 70 °C for 60 minutes. The whole dyeing procedure of the experimental group is illustrated in Figure 2, i.e., the sample was immersed at 40 °C and waited for 20 minutes, then the temperature was increased to 70 °C in 20 minutes. With this temperature, the fabric has been waiting in the solution for 20 minutes. Afterwards, the fabric was taken out and hot-cold washing was performed. For the dyeing of the control group, the dyeing condition and all parameters are similar in the experimental group except for the addition of alkali, and its dyeing curve is shown in Figure 2 b.



(a) *(b)*

Figure 2. (a) mercerisation followed by WOW reactive dying cycle of cotton fabric; (b) Reactive dying cycle of cotton fabric (Control group)

Evaluations

Washing fastness test

The washing fastness tests were conducted using the ISO 105-CO10-2018 technique [16]. A cotton and polyester sample measuring 10 x 4 cm was prepared and stitched. The paste was made by mixing 5 g/l of soap and 150 ml of water for each sample. The cups were properly closed after being loaded with 10 steel balls, a soap solution, and samples. Each cup is carefully kept in its holder. The machines were then configured and ran for 30 minutes at 60 °C. Ultimately, the fabric samples were removed, allowed to dry, and then assessed.

Determination of color strength (K/S)

The dyed samples were examined using a Datacolor-650 reflectance spectrophotometer once the dyeing procedure was completed. For sample analysis, 120 GSM fabric was used as a standard in all cases (0.5%, 1%, and 2%), and the colour coordinate values and relative colour strength were compared to such fabric for each dye concentration. All experiments were carried out with D65 as the standard illumination for measuring colour coordinates. The results were analysed using the K/S values of dye samples, which were initially determined using the Kubelka-Munk equation, as shown in equation [18-20]. The symbols 'K' and 'S' represent the coloured sample's absorption and scattering coefficients. The equation is used to calculate the relative colour strength (%) from reflectance (R). It represents the percentage difference in values between samples and a standard at the same wavelength. Based on the methods described above, the unwashed, soaping, and washing fastnesses were measured and recorded using a Datacolor spectrophotometer [21].

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (2)$$

RESULTS AND DISCUSSION

Various characteristics were investigated to validate (assess) the Currently invented semi-continuous process, which involves mercerisation and reactive dyeing of cotton fabric. In comparison to the control group, the amount of washing that was necessary, as well as the degree of exhaustion and fixation, were assessed. And so the outcome and the debate were given.

Determination of washing cycle

After subsequent washing and titration processes, the following results were obtained, which are illustrated in Table 3. It shows the required amount of sulfuric acid for titrating the fabric after each washing cycle. Ten replicates were performed for each experimental run, and the average values were presented.

Table 3. Title Results of the number of washes with alkali concentration present in the fabric

Washing cycle	Volume of H2SO4 required (ml)	Mass of titrated fabrics	Mass of NaOH	NaOH %(owf)
0	122	1.50	1.952	130
1	3	1.53	0.048	3.1
2	2	1.46	0.032	2.1
3	0.1	1.50	0.068	0.5
4	0	1.48	0	0
5	0	1.52	0	0

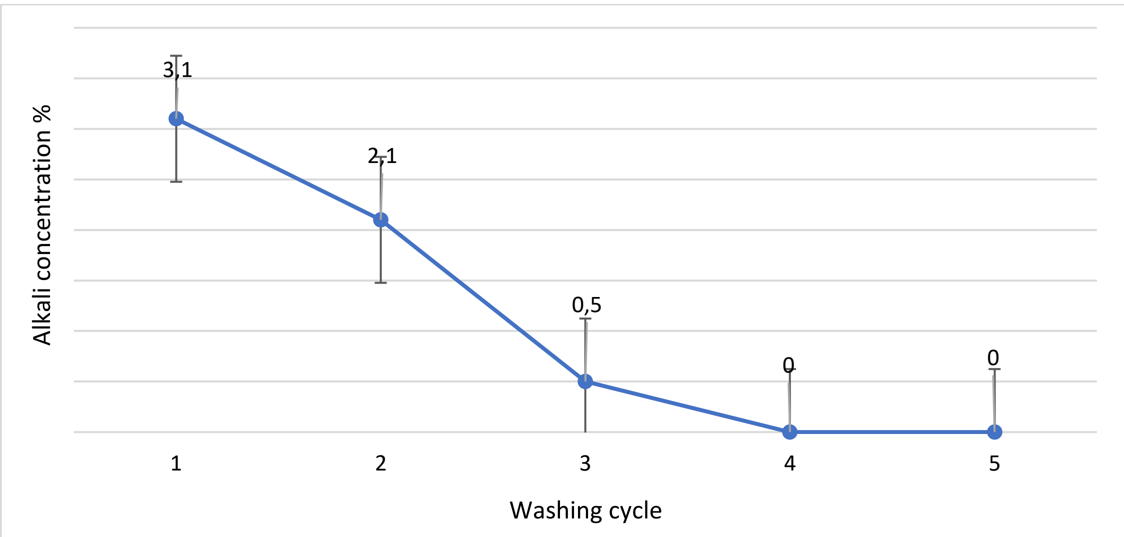


Figure 3. Effects of washing on alkali concentration

The above data shows that as the washing cycle increased, the amount of alkali left in the fabric reduced, requiring only a small amount of acid to titrate the given sample. This could be because when the fabric is immersed in a concentrated caustic soda solution, it immediately absorbs and swells. Since the hydroxyl group of cellulose was broken and linked to sodium ions, it yielded soda cellulose. This attraction is easily broken and eliminated with a subsequent hot and cold wash. Almost all alkalines were removed after four washing cycles. The graph in Figure 3 also depicts a nearly negative connection between the washing cycle and the alkali concentration remaining in the fabric.

Effects of alkali concentration on the degree of dye exhaustion

Every successive wash of materials was dyed and dried without washing to observe the effects of alkali concentration on dye exhaustion. Figure 4 displays the average K/S values for each fabric after ten replications were completed.

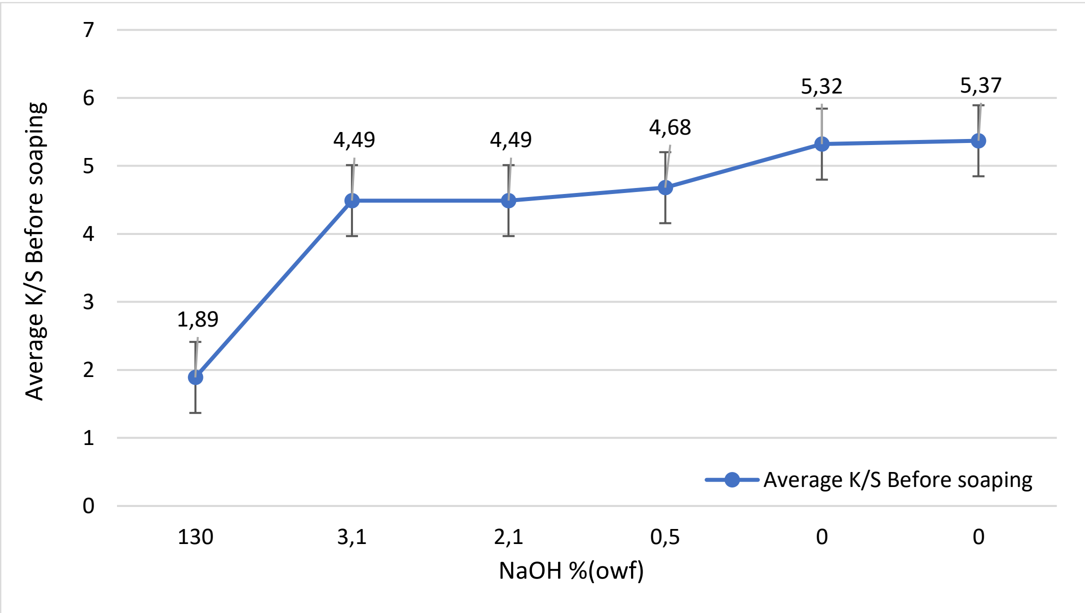


Figure 4. Effects of alkali concentration on rate exhaustion

This research demonstrates that the amount of alkali is one element that affects colour strength of colour or depth. When the amount of caustic soda used in the dying process exceeds the optimum value, nearly all of the hydroxyl groups in cellulose become saturated with soda. This means that they cause the soda cellulose to form an anionic charge that repels the dyes' anionic charge, leading to a low rate of dye exhaustion. However, if the material is frequently washed in both hot and cold water, the soda-cellulose interaction will break down, resulting in the removal of alkali and increasing colour exhaustion. Consequently, the aforementioned findings show that alkali content and dye exhaustion are inversely correlated. Some other researchers noticed similar effects [22,23].

Effects of alkali concentration on degree of dye fixation

Table 4 displays the average value for the colour strength of the dyed fabric before and after soaping, as well as after washing fastness tests. Ten replications were performed for every run. Subsequently, the degree of dye fixing was assessed by calculating the ratio between the colour strength value (K/Sc) of the fabric following the washing fastness test to the colour strength value (K/Sb) before soaping. Table 5 displays the average value of the degree of dye fixing for the fabric that was dyed using a common dyeing technique.

Table 4. The average value of colour strength and degree of dye fixation of mercerisation followed by wet-on-wet reactive dyed fabrics

Washing cycle	K/S before soaping (b)	K/S after soaping (a)	K/S After washing fastness test (c)	Using the grayscale method	Degree of dye fixation (F (%) =K/Sc/K/Sb*100)
0	4.89	4.69	3.45	4	70.55%
1	7.49	6.77	6.36	4-5	84.91%
2	7.49	5.31	5.83	3-4	77.83%
3	7.68	5.30	4.71	3	62.88%
4	8.32	5.18	4.76	1-2	57.21%
5	9.37	5.12	4.69	1-2	50.05%
Average (±Std)	7.54±1.48	5.40±0.71	4.97±1.02	4	67.24±0.13%

Table 5. The average value for colour strength and degree of dye fixation of the control group

No	K/Sb before soaping	K/Sb after soaping	K/Sc after washing fastness test	Using the grayscale method	Degree of fixation (F (%) =K/Sc/K/Sb*100)
1	7.61	6.16	5.94	4-5	78.05
2	7.18	5.99	5.84	4-5	81.33
3	7.13	6.17	5.79	4	81.20
Average (±Std)	*7.31*±0.26	*6.11*± 0.10	*5.86±0.08*	4	*80.19*±1.86

Where F (%) is the degree of dye fixation in percentage, K/Sb is the colour strength of fabric samples before soaping, and K/Sc is the colour strength of the fabric samples.

As different scholars confirmed [23-24], the concentration of alkali has a direct relationship with dye fixation. Since to fix a dye with fiber; it needs basic media [25]. Thus, the experiment's results, which are displayed in Tables 4 and 5, demonstrate that the unwashed (before soaping) fabric of both conventional and experimentally dyed fabric had the best colour strength when compared to fabrics that were soaped and had washing fastness tests conducted. This is because washing can remove the most loosely attached dyes from fibres, reducing the colour strength of the fabric. But if the dye solution has an appropriate amount of alkaline, those detachable dyes will be strongly bonded, form a covalent link, and have fibres that can endure any washing effects (have higher washing fastness). As can be observed from the fact that the degree of dye fixation decreased from 84.91% to 50.05% when the alkali concentration fell from 3.1 to 0%, this clearly illustrates that there is a significant positive link between alkali concentration and degree of dye fixation. Due to the dissociation of some hydroxyl groups in the cellulose caused by this alkali, the dye starts to react with the nucleophilic cellulose ions in the fibre. To reestablish the dyeing equilibrium, more dye is absorbed as a result of the fixing process. The process of dye absorption from the solution and its reaction with the fibre continues until the dye is no longer taken up. Thus, at the first washing cycle, the best dye fixing (84.91%) was recorded. Additionally, this value is somewhat better than the control groups, which were dyed using a conventional dyeing method. Similar results were seen in other scholars' investigations on the environmentally friendly salt-free exhaustion dyeing of cotton fabric with reactive dyes [22]. This research verified the appropriateness and efficiency of this novel dyeing method.

Furthermore, this wet fabric is in a relaxed state which allows it to absorb additional dyes. Therefore, the mercerised fabric should be washed once to get the optimum alkali concentration (3–3.5%) in the fabric. The photographic images of fabrics that have been dyed using WOW reactive dyeing and conventional dyeing procedures, respectively, are also depicted in Figures 5A and B. Subsamples were obtained from each sample after soaping, and their washing fastness test results were assessed. As shown in this figure, the samples were returned to their original places to examine the differences. For instance, sample washed 4 times (experimental sample) was taken from sample 1-4. These images show that the strength of the bond between the dye and the fibre has a significant impact on the amount of dye loss that occurs during fabric washing. Consequently, an optimal alkali concentration was needed to make a strong link. Since the dyes were fixed to the fabric when the alkali content was at its optimal, Therefore, the effectiveness of each fabric's washing depends on this level of fixation. Additionally, the experimental sample, labelled 1-1, has a superior colour look (deep shed) than the control group, coded 1-C1, 1-C2, and 1-C3.

Generally, when the WoW reactive dyeing fabrics were at their optimum alkali concentration—that is, after one cycle of washing—the degree of fixing, washing fastness, and exhaustion were just slightly better than dying after drying (conventionally dyed fabrics). The research's results for these parameters are also consistent with those of earlier studies [22-25]. The newly developed method (mercerisation of cotton fabric followed by reactive dyeing) produces results that are appropriate and effective. Comparing this dyeing approach to the traditional one, there are several advantages. Energy is saved for drying and washing, labour is saved for processing, and chemicals like acetic acid and alkali are saved for neutralization and fixation.

a)

b)

Figure 5. Washing fastness test results for a) mercerisation followed by WOW reactive dyed and b) conventionally dyed fabrics

Where figure A, 1-0, 1-1 up to 1-4, and 1-0w, 1-1w up to 1-4w are the experimental fabrics before and after washing the fastness test, respectively. Figures 1-A1, 1-C2, and 1-C3 indicate the sample numbers of the control group.

CONCLUSION

Two dyeing procedures are used in this experiment. The first is conventional dyeing (control group), and the second is mercerisation followed by wet-on-wet reactive dying (experimental group).

The optimal washing cycle was determined to find the optimum alkali contents of the fabric, which is used for fixation during the dyeing process. From the study, the optimum washing cycle is one, and at this washing cycle, the optimum alkali obtained in the fabric is 3.1%. At this optimal alkali concentration, the degree of dye exhaustion and dye fixation of the fabric have been assessed and found to be 4.49% and 84.91%, respectively. The alkali concentration has a direct relationship with degree fixing and an inverse relationship with dye exhaustion. Compared to fabric that has been dyed conventionally, fabric that has undergone mercerisation and then wet-on-wet reactive dyeing has a somewhat superior degree of dye fixation and colour strength. Thus, this process's path is cost-effective. The alkali that was added to reactive dyes was saved, as was the energy needed for washing and drying before dying. This research will be expanded by identifying the amount of saved costs by manpower, time, auxiliary chemicals, and energy.

Author Contributions

Conceptualization – Admas A and Assefa A; methodology – Admas A; Assefa A; formal analysis – Tsegaye M And Tessema A; investigation – Admas A; writing-original draft preparation – Admas A; writing-review and editing – Assefa A; All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare that there is no competing interest.

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