

# Optimizing Soaping Process Parameters for Reactive Dyed Cotton Knitted Fabrics – Towards Sustainable Dyeing

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## Abstract

Wet processing of textiles has recently focused attention on sustainable dyeing. Reactive dyeing is one of the eco-friendly dyeing processes, aside from nano dyeing, which is yet to commercialize. The hydrolyzed unfixed dye and loosely fixed dye molecules, however, tend to yield erroneous color strength readings following dyeing. The goal of this research is to improve the frequently applied soaping after-treatment procedures employed after reactive dyeing. With infrared exhaust dyeing equipment, a single jersey cotton knitted cloth weighing 160 grams per square meter was colored with VIVIZOL blue in 4 hues (1%, 3%, 5%, and 7%). For the post-treatment of colored materials, the soaping chemicals Exoline Magic M (EXO-M) and RG-Hxc were employed. The color strength ratings were used to determine the samples' effectiveness at removing dye. To develop samples, Taguchi L9 orthogonal array experimental planning was employed. The elements that affect soaping efficiency were determined to be liquor ratio, concentration of soap used, temperature, and time. The responses were evaluated using the signal-to-noise ratio (S/N). MINITAB software was used for taguchi design of experiments. According to the study's findings and observations, the RG-Hxc soaping agent removed hydrolyzed dye with a removal efficiency of 24.8%, while the EXO-M washing agent had a removal efficiency of 20.6%. Using 2 g/L RG-Hxc, the soaping operation was performed at a temperature of 90 °C with a bath liquor ratio of 1:10. Compared to EXO-M soaping agent, RG-Hxc soaping agent for 15 min showed greater hydrolyzed dye removal efficiency.

## Keywords

Reactive dye, Hydrolyzed dye, Soaping agent, Taguchi design, Optimization

## Introduction

The environmental impact of the coloring process is substantial, especially when it comes to the use of energy, water, chemicals, and air pollutants. Over 20% of the contaminants found in water globally are mostly created by the textile dyeing process. Excessive quantities of chemicals, especially salt, are the source of water pollution during dyeing processes. To develop an environmentally, socially, and economically feasible process, conventional dyeing must be reconsidered to establish a sustainable dyeing system. The continuous dyeing method known as nano-dye, which keeps dye molecules in discrete nanoscale for absorption into cellulose, uses significantly less water and energy and doesn't require salt as compared to the standard dyeing process.

Reactive dyes with a vinyl sulfone group as the reactive group (also known as the reactive dye's "reactive hook") are called vinyl sulfone dyes. vinyl sulfone

dyes are frequently used in automated batch-wise dyeing machinery, cold pad batch processes, and exhaust dyeing. During the soaping procedure, reactive dyes that have collected on the fabric and are yet unfixed are eliminated [1-3].

The performance of eco-alkali (a mixture of multiple alkalis) and soda ash is assessed in one of the studies on the reactive dyeing of cotton fabrics [4]. Since the examination of several dyeing properties, including fixation efficiency (%), pH dyebath after dyeing, and color yield (K/S value), acceptable conclusions are drawn regarding the efficacy of eco-alkali. On the impact of soaping settings on color removal rate, neither data nor research are provided. In a different study, research has been done on how reactive dyes behave when used on kenaf fiber [5-7]. Studying the elimination of reactive dyes from binary solutions makes use of the Langmuir model. No reference is made to the behavior of reactive dyes in soaping solutions. Many research reports examine the influence of soaping and process parameters on reactive dyed fabrics [8]; however, they do not address the efficiency aspects of soaping. Some work is also carried out on dye transfer inhibition, but not on the soaping process [9]. In one of the studies, the effects of fixation agents and the wet fixation process have been reported, but not the optimization aspects of the soaping process [10, 11]. So, the need is to develop and optimize a strong soaping process with better soaping properties that can remove the dyestuff, prevent redistribution of unfixed dye, and remove hydrolyzed dye particles.

Many dyeing process parameters influence the aggregation of reactive dye on the surface, depending on dyeing process parameters like temperature, pH level, dyeing time, etc. During dyeing operations, hydrolyzed dye is produced mainly because of temperature and alkali on breaking the bond between the dye and substrate. This research aims to address and determine the ability of soaping agents to remove unfixed and hydrolyzed dye under different soaping conditions. This research is important because the hydrolyzed dyes can hold onto the fabric through adsorption and redeposit themselves on the fabric surface. These unfixed and hydrolyzed dyes result in lower fastness values to rubbing and washing, coupled with lower values of color strength (K/S). Previous studies have revealed that factors affecting the effectiveness of the soaping process include the soaping temperature, the concentration of soaping agent used, and the liquor ratio employed. The effects of four soaping process variables, including material-to-water ratio, soaping agent concentration, soaping temperature, and soaping time, are investigated and optimized in this study.

The orthogonal arrays are used to carry out a series of laboratory tests as part of Taguchi's approach, which also includes determining the appropriate control elements to obtain the greatest outcome from the process. The outcomes of these trials are used to analyze the data and forecast the quality of the component developed during the process or for the product. Orthogonal arrays are used in the Taguchi experiment design to establish factors and affect strategies. In a small number of experiments, Taguchi procedures collect the data necessary to identify the factors that have the most effects on process quality. It consequently conserves resources and time. The commonly used parameter to evaluate the response is the S/N

ratio. Three categories of Taguchi orthogonal array proficiency properties are used: minimal is best, higher is best, and lower is best [12-15].

To ascertain how the soaping process improves the dye removal efficiency of knitted cotton fabrics that have been reactively dyed without completely removing all the hydrolyzed dye. Cotton knitted fabrics were dyed with different depths of color (1%, 3%, 5%, and 7%). The fabrics were scoured and bleached before dyeing. Two soaping agents were used in this study. RG-Hxc and EXO-M were used as soaping agents. The dye clearing or removal efficiency was evaluated for both dyed and unsoaped fabrics using the color spectrophotometer by evaluating its color strength values. There have also been studies examining how hydrolyzed dye removal is affected by washing temperatures, times, liquor ratios, and soaping agent concentrations [16, 17]. Taguchi orthogonal array analysis is used to determine the ideal washing method for eliminating hydrolyzed dyes.

### Overview of nano-dye process

Through a significant reduction in the huge pollution, water use, energy use, and climate change produced globally by cotton textile dye facilities, the nano-dye process is leading exhaust dyeing into the new millennium. Since the first two of its continuous, mass production systems have been started, the nano-dye process is raising the bar for exhaust dyers in the cotton and cotton blend industry. Cotton exhaust dye jets may now use up to 99 percent less dye (removing solid waste), 75 percent less water, and 90 percent less energy thanks to nano-revolutionary dye's dyeing technique. This results in more uniform fabric quality in all colors and improved lot-to-lot shade repeatability. Textile dyeing is now approaching clean business thanks to the decrease in pollutants in the effluent, which also makes zero discharge water treatment facilities more cost-effective to operate and establishes their future location.

The exhaust dye jet cycle is modified in the nano dye process to increase production and reduce overall dyeing costs. The product is a cloth with a color that is more tone-correct, softer to the touch, smells normal, and is more colorfast. With the development of the nano-dye system, sustainable exhaust dyeing is now possible after years of study. Although it is currently not on the market, this nano-dye technique will greatly enhance the current reactive dye exhaust dye system.

## Experimentation

### Materials

Ethiopian Textile Industry Development Institutes (ETIDI) provided a 100% cotton single jersey knit fabric that had been scrubbed and bleached and weighed about 160 g/m<sup>2</sup>. The chemical laboratory of the ETIDI provided the dye material and coloring chemicals. In these investigations, sodium chloride, soda ash, ANITISIL CONZL sequestering agents, vinyl sulfone reactive group (VIVIZOL Blue) dye, and 100% acetic acid were utilized. Two distinct soaping agents, such as EXO -M and RG-Hxc, were employed for

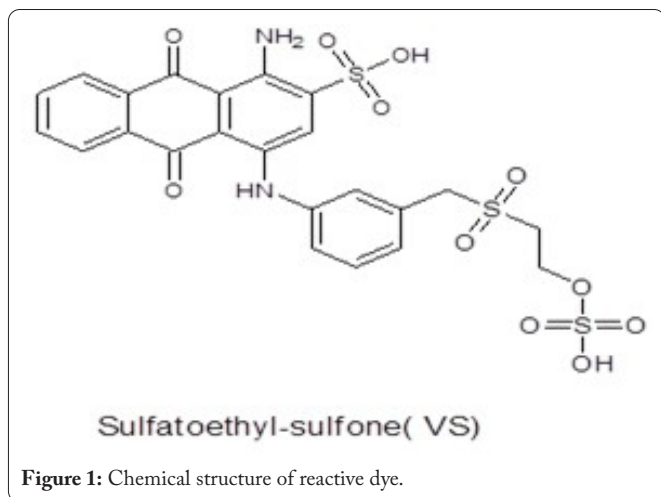


Figure 1: Chemical structure of reactive dye.

this experimental investigation. Figure 1 depicts the chemical composition of the reactive dye employed in this experiment, the vinyl sulphone reactive group (sulfatoethyl sulfone) dye (VIVIZOL blue).

### Soaping mechanism

Some technical features of the soaping process are described in this part because that is the main topic of the study article. In this study, the effectiveness of the soaping process is assessed using the metric of soaping efficiency. How well the dye soaps work will depend on the quantity of unfixated and hydrolyzed dye present in the colored fabric as well as how easily the hydrolyzed dye, the soaping agents, may be removed. The soaping method should remove the harmful effect hard-forming ions have on unfixated dye solubility and make it easier to remove unfixated dye molecules. The removal of excess or unreacted pigment from cellulose fiber during the soaping process enhances wash fastness. Soaking would therefore improve the stability and brightness of colored materials. There are three stages to the soaping of reactive dye or the elimination of unfixated and hydrolyzed dye [18, 19]. After dyeing, the fabric is twice cold rinsed to remove electrolyte (sodium chloride), once hot rinsed to remove some hydrolyzed color from the fibers, and once hotter rinsed to prepare it for boiling or high-temperature soaping. Figure 2 shows a schematic representation of the soaping procedure.

The earlier stages have an impact on the success of the soaping stage. After making the necessary preparations for soaping, this method produces outstanding results. With or without soaping agents, a low-foaming surfactant improves the effectiveness of cleaning. The effectiveness of the soaping process also depends on the water's hardness and the dye you intend to use; it also requires a chelating agent (which interacts with metal ions). The temperature during the soaping process might fluctuate between 60 °C and 90 °C for 9 - 15 min. Deep hues, notably blacks and reds, call for this stage.

After cooling and dropping the soap bath, rinsing follows. Rinsing once or twice in warm water between 50 °C and 60 °C gives better results. The number of rinses depends on the color of the rinse bath, the liquor ratio, and the equipment, all of which play a key role in the outcome. As a result, factors

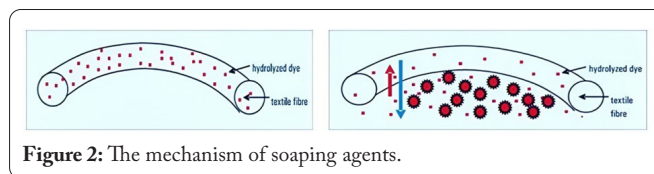


Figure 2: The mechanism of soaping agents.

like substantial dye, dye diffusion rate, water hardness, washing liquor ratio, and washing liquor pH are all related to how effectively these soaping and washing processes work. types of soaping agents and soaping agent concentrations. The main variables that affect dye aggregation are dye type, dye concentration in the dye bath, material to liquor ratio, temperature, and dyeing auxiliaries utilized.

### Methodology

This research method uses a cotton knit fabric (single jersey knit structure) dyed in four distinct colors after being treated with a chemical soap. The success levels of the removal of hydrolyzed dye are assessed by comparing the dyed and soaped fabrics color strength (K/S) values using a spectral approach. The removal of hydrolyzed dyes and the effects of the washing temperature, time, liquor ratio, and soaping agent concentration were studied. Finally, Taguchi orthogonal array analysis was used to determine the ideal washing process parameters that affect the removal of hydrolyzed dyes.

### Color strength of dyed and soaped fabrics

The colorimetry values of a dyed and soaped fabric are measured to determine their color strength (K/S). The color strength number can be used to analyze the inversely proportional relationship between shade percentage and color depth. Numerous dyeing factors, such as the dyeability and fixing percentage of dyed materials, can be estimated using the color strength values. For reactively colored materials, color strength values are essential. The color strength values of soaped and unsoaped fabrics are calculated from spectrophotometer data at the ( $\lambda$ ) wavelengths set at maximum absorption. The Kubelka-Munk equation (Equation 1) was used to calculate the reflectance value or color intensity of colored materials.

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

Where R is the reflectance of fabric after dyeing, S stands for scattering coefficient and K stands for absorption coefficient.

### Dye removal efficiency (% R)

The dye clearance or removal efficiency was calculated using a data color spectrophotometer [20, 21]. Based on the colour strength value indicated in equation 2, colour strength values were utilized to calculate the effectiveness of soaping agents.

$$\left( \frac{\left( \frac{k}{s_0} \right) - \left( \frac{k}{s_1} \right)}{\left( \frac{k}{s_0} \right)} \right) \times 100 \quad (2)$$

Where  $k/s_1$  and  $k/s_0$  represent color strength values of soaped and unsoaped fabrics, respectively.

### Reactive dyeing

The fabrics that had been scoured and bleached were then dyed using EKSOY dyeing method and reactive dye (Figure 3 and figure 4). The fabrics were dyed with four different depths (1%, 3%, 5%, and 7%). The chemicals added during reactive dyeing were BUFFERON R11 soda ash (15 g/L - 20 g/L), sodium chloride (30 g/L - 80 g/L) and ANITISILCONZL sequestering agent (1 g/L). The fabrics are dyed for 80 min at 60 °C.

A liquor ratio of 1:20 was used. A 5% shade was utilized to dye samples of cotton knit fabric for the Taguchi experimental design plan for optimizing the soaping process. Figure 5 displays the samples (samples 8, 9, 6, and 5) without soaping. These images are a collection of randomly dyed sample photos taken in accordance with the Taguchi experimental design plan. After being soaped, the samples were assessed, and their colorfastness was determined. After numerous trials and tests, the findings of the color fastness test were reported and graded following a soaping.

### Dyeing procedure

The fabric samples were first soaked in a wetting agent before being immersed in a dye solution that contained dyeing agents and auxiliaries in accordance with the recipe. After that, the cloth sample dye pots were labelled. Initially, the dye-bath temperature was maintained at 30 °C and dyed for 40 min. After adding 1/4 of the electrolyte first, followed by the remaining sodium chloride after a 10-min interval, the temperature rises to 60 °C at a pace of 10 °C per minute. When the temperature hits 60 °C after 10 min, levelling agents and BUFFERON R11 soda ash are added, allowing the dyeing to proceed for an additional 40 min. The mixture is then run for 30 to 60 min to allow color fixing to take place once the remaining soda ash has been added. The cloth sample is chilled to 40 °C after being dyed by keeping the cooling rate at 50 °C per minute (Figure 3).

### Washing, soaping, and rinsing

Standard soap, manufactured by the James Heals Company, is used for soaping. RG-Hxc and EXO-M were the two types of soaping agents used in this research. The structure of the soaps is like that of standard soaps. The common structure of soap contains a hydrophilic group, usually a carboxylate group, and a hydrophobic group, normally an aliphatic chain. The structure of the soap is shown below in figure 6.

After dyeing, the test fabrics underwent a 5-min neutralization process in a bath containing 1% acetic acid at 40 °C and a 5-min cold wash at that temperature. Following neutralization, the sample fabrics were subjected to a variety of washing conditions, including varied material liquor ratios (1:10 - 1:30), soap concentrations (1 g/L - 3 g/L), soaping temperatures (800 °C - 950 °C), and soaping periods (5 min - 15 min.). The fabric samples were then cleaned and allowed to air dry. After draining the washing liquor, the colored sample

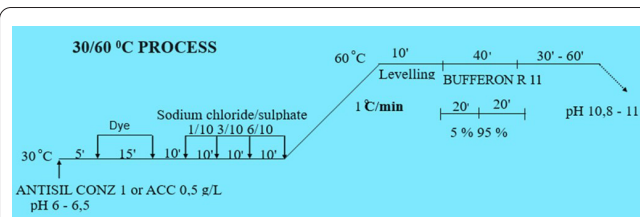


Figure 3: EKSOY process for dyeing.



Figure 4: Dyed samples.



Figure 5: Dyed fabric samples 8, 9, 6, and 5 (as per the Taguchi experimental design plan).

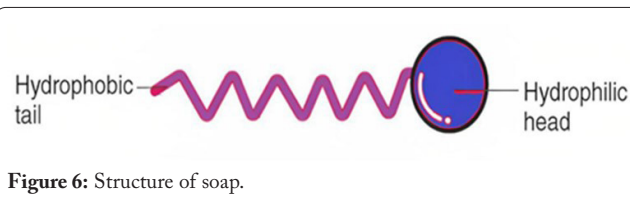


Figure 6: Structure of soap.

fabrics are once again washed in hot and then cooled water. The launder-O-meter is used to perform the soaping process, and samples of soaping at various concentrations are displayed in figure 7.

Figure 8 illustrates how RG-Hxc outperforms EXO-M in terms of hydrolyzed dye removal performance, with a higher percentage of dye removal. Based on the findings of the color strength value test, the calculated values of dye removal efficiency using equation 2 are shown in table 1.

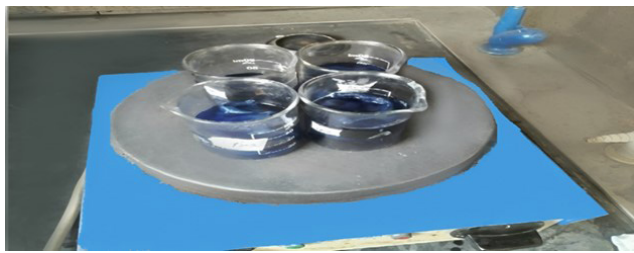


Figure 7: Dyed fabric soaping bath in different concentrations.

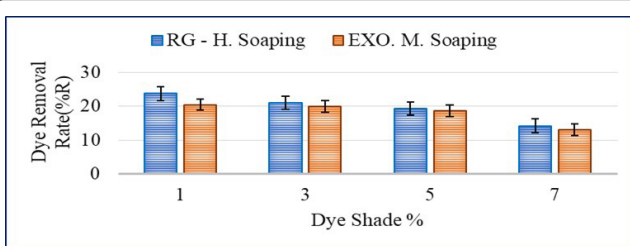


Figure 8: Dye removal percentage of dyed samples.

Table 1: Dye removal percentage.

Soaping agent	Shade %	K/S (Before soaping)	K/S (After soaping)	(%R)
RG-Hxc	1	6.5504	4.9196	24.8
	3	10.4330	8.2400	21.01
	5	20.6580	16.6700	19.3
	7	26.0744	22.3697	14.2
EXO-M	1	5.0512	4.0132	20.6
	3	10.4698	8.39400	19.83
	5	20.1764	16.4000	18.7
	7	25.163	21.8864	13.02

The EXO-M soaping agent showed a dye removal efficiency of 20.6% lower than the RG-Hxc soaping agents 24.8%. RG-Hxc soaping outperforms when compared to EXO-M soaping agent.

## Results and Discussion

### Taguchi's design of experiments

Creating an experimental matrix using Taguchi experimental design techniques results in a strong design. The

Table 2: Soaping process variables and experimental levels.

Code	Soaping process variables	Level 1	Level 2	Level 3
A	Soap concentration (g/L)	1	2	3
B	Bath liquor ratio	10	20	30
C	Temperature (°C)	80	90	95
D	Time (min)	5	10	15

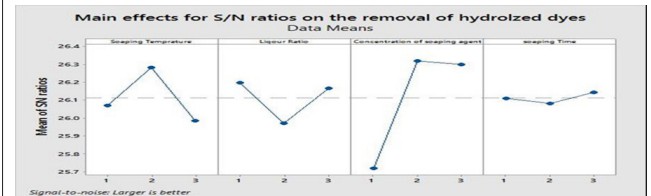


Figure 9: Soaping process's primary effect and interaction effects.

washing process parameter factors are first given the proper orthogonal array design, and their levels are displayed in table 2. The reactive dyeing range is normally 60 °C to 80 °C. Normally, soaping temperatures are kept slightly above dyeing temperatures or equal to dyeing temperatures so that unfixed dye can be removed. The soaping temperature range followed justifies the paper's objective. Reduced water and energy use, as well as pollution, will result from washing process optimization [22-25].

The response is analyzed using MINITAB software. The main and interaction effects of soaping process variables and dye removal percentage are shown in figure 9. For better understanding the effects of process parameters, standard deviation, static design or slope, and S/N ratios are studied. Although the optimal factor values are those that optimize the soaping process parameters based on their process parameter design settings (Figure 9), As a result, Taguchi-design of experiments can be used to simplify the removal of unfixed hydrolyzed dyes from reactively colored cotton while also optimizing the soaping procedure. Table 3 displays the experimental setup for the different soaping process variables.

Following the experimental fabric samples' dyeing, the washing procedure was carried out using the L9 Taguchi (orthogonal array) design's experimental arrangement. The color strength value of coloured samples on fabrics was then as-

Table 3: Taguchi experimental design plan and dye removal efficiency (%).

Exp. No.	Temperature (°C)	Bath liquor ratio	Soap concentration (g/L)	Time (min)	Dye removal efficiency (%)	S/N ratio
1	80	1:10	1	5	19.40	25.7560
2	80	1:20	2	10	20.20	26.1070
3	80	1:30	3	15	20.76	26.3445
4	90	1:10	2	15	21.40	26.6083
5	90	1:20	3	5	20.72	26.3278
6	90	1:30	1	10	19.76	25.9157
7	95	1:10	3	10	20.48	26.2266
8	95	1:20	1	15	18.80	25.4832
9	95	1:30	2	5	20.52	26.2435

essed using a data colour spectrophotometer before and after soaping. Based on the findings of the dye removal efficiency and color strength values for 5% dyed and soaped samples, suitable conclusions are drawn after running the experimental data using Minitab software. The larger the sample, the better the study, according to the Taguchi technique, which was used to investigate the influence of various elements Taguchi's L9 orthogonal array was employed in the experiment. to ascertain the effect of washing process settings on removing unfixed dye. The response tables for the results of the S/N ratio and dye removal efficiency % is indicated in table 3. The delta statistic (Table 4) is created by subtracting the highest average value from each element's lowest average value. Based on the delta statistical value, a rank was awarded. According to table 4 and observing the delta statistics, soap concentration, which had a delta statistic value of 0.6, was the factor that most significantly reduced the amount of unfixed hydrolyzed dye. The temperature of the soap was the second essential component of the washing process, and the delta statistical values were 0.30. The washing technique was the third component, the soaping liquor ratio was another S/N ratio, and the delta statistic value was 0.22. The delta statistic for soaping time was 0.06.

## Conclusion

In this study, the hydrolyzed dye was effectively removed by the RG-Hxc washing agent with a quantitative value of 23.8% as compared to the EXO-M soaping agent, which was 20.6%. Hence, the RG-Hxc soaping agent removes color more efficiently while using less water and energy. On the other hand, EXO-M. soaping agent utilizes more water while consuming less energy to reach a better level of dye removal efficiency. Overall, during the 15-min soaping process at a temperature of 90 °C and a 1:10 bath liquor ratio, RG-Hxc soaping agent outperformed EXO-M soaping agent in terms of hydrolyzed dye removal effectiveness.

Based on the results of the Taguchi orthogonal array analysis delta statistics data, the varied amounts of soaping parameters are ranked as A2, B1, C2, and D3 based on their best values and the removal of hydrolyzed dye in the soaping process. As a result, the temperature, the length of time spent washing, the quantity of alcohol employed, and the quantity of washing agent all have a positive and significant influence on dye removal efficiency. Removal of unfixed dye was negatively impacted by higher soap concentrations, whereas the soap concentration of 2 g/L had the maximum favorable impact. From this point on, the effectiveness of removing the hydrolyzed reactive dye is primarily influenced by the concentration of washing chemicals. From this analysis, maintaining the optimum washing process conditions is very important for the removal of unfixed hydrolyzed dye and to maintain a higher color strength value in soaped and washed fabric. Therefore, for 5% shade-dyed fabrics at 90 °C, the liquor ratio is 1:10. Soaping concentration of 2 g/L and washing time of 15 min were selected as the optimum process parameters for further soaping treatment.

Through these optimum washing process parameters, one can achieve the maximum hydrolyzed dye removal efficiency value. As a result, water and time consumption, as well as con-

**Table 4:** S/N ratio response table.

Level	Temperature (°C)	Bath liquor ratio	Soap concentration (g/L)	Time (min)
Level 1	26.07	26.20	25.72	26.11
Level 2	26.28	25.97	26.32	26.08
Level 3	25.98	26.17	26.30	26.15
Delta	0.30	0.22	0.60	0.06
Rank	2	3	1	4
	A2	B1	C2	D3

centration and utilization of washing agents, can be greatly reduced. The consumption of water and washing of auxiliary items is not only low but also reduces wastewater release to the drainage system, thus resulting in less environmental pollution. New dyeing methods like nano-dyeing and process that eliminate soaping process can be developed to present sustainable solutions for the textile processing sector.

## Acknowledgments

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## Conflict of Interest

None.

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