

Effective Process Optimization Of Indigo Rope Dyeing: A Case Study

Shumaila Meraj¹, Amtul Qayoom^{1*} and Saeeda N. Al¹

¹Department of Chemistry, NED University of Engineering and Technology, Karachi-75270, Pakistan.

Corresponding author: email: amtulq@gmail.com

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Abstract

Despite of limited availability and high cost of utilities, Pakistan is now declared as the second largest denim exporter country. With the advent of denim garment, it has been crucially important to face the upcoming challenges of the process optimization, cost effectiveness and utilities consumption from pre-treatment of raw yarn to quality finished denim. The focus of this study was to understand industrial scale optimization of sodium dithionite and water consumption for indigo rope dyeing process. Consumption of sodium dithionite was reduced from 16.99 gm/meter to 10.62 gm/meter from the month of July-15 till February-16 which resulted in steady utilization of indigo. Reduced utilization of chemicals decreased water requirement for post washing dyed yarn. These factors lead to comparatively cleaner, cost effective indigo dyed yarn production with required shade consistency.

Keywords: denim; indigo; optimization; utilities consumption; raw yarn; dyeing

1. Introduction

The denim industry has been playing an important role in the textile sector with approximately 636,000 tons per annum production [1],[2]. Pakistan is the second largest exporter of denim fabric with 40 major production units producing around 50 million square meters of finished denim fabrics per month [3]. Traditionally denim fabric is produced by dyeing cotton yarn with naturally occurring indigo dye but nowadays due to enormous consumption of denim fabrics and articles, a larger percentage of synthetic indigo dyes are also used for dyeing[4],[5]. There are number of techniques used for indigo dyeing but rope dyeing is one of the most suitable indigo dyeing processes in terms of uniformity of dyeing yarns [6]. Indigo is water insoluble vat dye and cannot mobilize itself towards cotton fiber until it is converted into water soluble form known as "leuco" through the process of reduction. Sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$) aka sodium hydrosulphite or hydro is one of the most commonly used reducing agent in indigo dyeing process. Approximately 150 liters water per kg fabric is utilized in a typical indigo dyeing process. Consumption of large quantity of chemicals and fresh water ends up in increased product cost and larger volume of chemically loaded effluents; particularly excess sodium dithionite, unfixed indigo dye and their byproducts. Optimization of various dyeing parameters can reduce consumption of chemicals and water requirement for typical denim dyeing processes. For an optimum rope indigo dyeing process, it is recommended to regulate some of the dyeing parameters e.g. concentration of sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$), pH value of caustic soda (NaOH), dipping time and airing time etc.

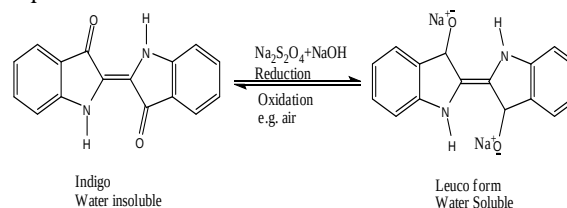
This case study presents the statistical comparison of eight months data based on optimization of amount of sodium hydrosulphite ($\text{Na}_2\text{S}_2\text{O}_4$), and water consumption control for rope dyeing process in a denim industry which will ultimately result in the reduction of unit cost per meter in dyeing yarn.

2. Reduction Mechanism Of Indigo

The indigo bath consists of indigo dye, caustic soda (NaOH) and sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$) as reducing agent. During reduction process $\text{Na}_2\text{S}_2\text{O}_4$ react with NaOH and break it into NaO and nascent hydrogen; both of which adhere to indigo dye molecule. Nascent hydrogen opens the indigo vat dye molecule by letting it join with water and the water carry the vat dye to the cotton fibre. This reduction process is termed as vatting[7].

During dyeing process the concentration of leuco is maintained in the dye bath by the addition of sodium dithionite. Once indigo is applied on fabric, it oxidizes back to its original insoluble form in order to make it stay within the fibre by skying process [8].

Reduction oxidation mechanism is shown in following equation.



3. Indigo Dyeing Process

Indigo dyeing process involves number of stages shown in Figure 1. Rope of 300 to 400 individuals threads are continuously dyed in the following steps:

- Pre-treatment
- Prewashing
- Indigo dip
- Post washing

On top of each dip squeeze rollers are arranged to oxidize dye with air.

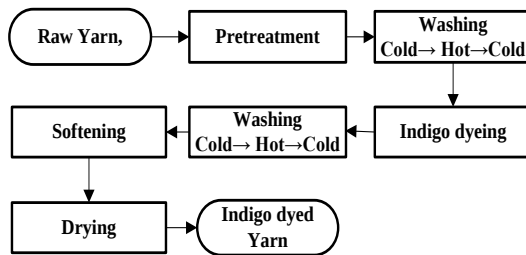


Figure 1 Steps involved in typing indigo rope dyeing process

3.1. Pre-treatment of raw yarn

In the pre-treatment process, ropes are fed into scouring baths where warp yarn is treated with wetting agent, detergent and caustic soda at 70-80°C. The purpose of this step is to remove trapped air from cotton yarn and guarantee uniform wetting and dyeing. Then, ropes are fed into one or more water rinsing baths.

3.2. Prewashing

Pretreated raw yarn is thoroughly washed with hot water to remove remains of caustic which might change the pH of the dye bath. After hot wash, yarn is cold washed to lower down the yarn temperature as indigo dyeing is carried out at room temperature [9].

3.3. Recipe for dyeing liquor and vat dye stock [8].

The dyeing tanks contain about 2000 liters of bath comprising:

- Caustic soda (27% by weight), 5 ml/l
- Sodium dithionite, 1.5g/l
- Stock vat, 62.5 ml/l

The bath (about 16 000 liters) is constantly re-circulated and automatically reinforced by mother solution on the basis of the weight of the yarn being dyed.

The dyeing stock is prepared as follows

- 85 g/l Indigo Pure
- 76 g/l Hydro

- 57 g/l Caustic Flakes or 74 ml/l caustic 50°Be
- 1 ml/l Dispersing Agent
- 1 gm/l wetting agent

Mostly the vat stock is prepared by adding indigo dye with 0.8-0.9 time's sodium dithionite and 0.7-0.8 time's caustic soda. The quantity of caustic soda and sodium dithionite must be monitored regularly according to required amount.

3.4. Indigo Dyeing

After prewashing the yarn ropes are fed into dyeing equipment which consists of series of dye baths where multiple lower and upper rolls are provided to twist and squeeze yarn ropes between the dye baths. Above the middle set of multiple dye baths, there are skying rolls. After wetting out of the first dye bath, they pass at about 20 to 30 m/min to the skying rolls and dipped for 20 -30 seconds in leuco indigo dye liquor and then skied for 2 minutes after leaving the dye box[9]. The skying floor should be high enough for complete oxidation of the dye. This step is repeated for each dye liquor box followed by skying for oxidation. In the successive dye box more leuco indigo is consumed followed by oxidation and the fibre surface is gradually dyed with indigo pigment. There are more than six dye boxes used for finer layers of indigo on yarn surface [10].

3.5. Post washing and softening

After dyeing the unfixed die and residual of caustic from the yarn surface are removed by washing the ropes with the water at about 60°C. Washed ropes are lubricated so that individual threads can be easily separated during re-beaming process. For lubrication the bath contain water, softener, acetic acid and this application occurs at room temperature.

3.6. Drying

In the last, the ropes are dried at about 120°C with the residual of 8% humidity. Dried yarn ropes are stored in a can in such a way that threads will not be twisted.

4. Process Control Parameters Of Rope Dyeing For Denim

4.1. Concentration of dye in dye bath

Mostly the concentration of indigo dye depends on the need of the depth of the shade and the number of dips used. It is usually in between 2 to 6 g/l [11]. If the dye concentration is high, it will show a poor rubbing fastness with more reddish duller shade of indigo dyeing. When yellow leuco indigo oxidizes, it transforms into greenish and then gradually turns into blue oxidized form. Small quantity of sodium dithionite must be added to dye bath for maintaing clear yellow leuco and to prevent premature oxidation of dye into greenish form.

4.2. Dipping time

Longer dipping time results in high penetration of dye in the yarn which reduces the ring dyeing effect. On the other hand for short dipping time, there must be a negative effect on dye penetration depth. The dipping time should be in the range of 20-30 seconds for each dye bath. On exceeding more than 60 seconds, darker shades are obtained due to high take up of dyestuff [11].

4.3. Concentration of Sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$)

Concentration of sodium dithionite plays very vital role in indigo dyeing process. Higher concentration of sodium dithionite results in lighter bluer shade and ultimately consumption of indigo will be much higher if darker shade is required. At relatively low concentration of sodium dithionite, darker shades can easily be produced. A further decrease in sodium dithionite concentration results in unreduced indigo which will bleed in the next bath and post washing.

4.4. Redox potential

The redox potential depends on the final desired shade. Typically it should be in the range of -760 to -860 millivolts. In order to keep the leuco indigo stable, the level of redox potential should be low. The potential of insoluble indigo in dye bath remains same until all the leuco indigo molecules are oxidized to raise the potential in the positive direction. Reduction capacity is monitored by millivolt meters to ensure optimum redox potential mentioned above.

4.5. Caustic soda or pH value

pH of indigo dye bath greatly affect dye penetration and its affinity to fabric. Being weakly acidic, indigo dye exists as mono-phenolate ions at pH 10.5-11.5 which is most substantive form for ring dyeing as compared to its other ionic forms [10]. Therefor alkaline pH (10.5 to 11.5) is maintained by adding caustic soda to dye bath in order to achieve a high strike rate of the dye to the yarn and for good wash down activities[12].

4.6. Airing time

Airing time for the fixation of dye should not be more than 60 to 75 seconds because longer time results in high tension on the yarn and subsequent process becomes difficult.

4.7. Optimization of sodium dithionite concentration

The indigo and sodium dithionite concentration must be efficiently managed through continuous feeding of both ingredients otherwise shade will be altered after vatting[13]. Although sodium dithionite enables very short fixing times in various dyeing methods and produces levelness in

continuous dyeing [7], it is unstable and can be easily oxidized by atmospheric oxygen. Higher temperature further reduces stability of its alkaline solutions and it gets oxidized even in the absence of oxygen. Therefore, excess quantity of sodium dithionite and caustic soda are needed over the stoichiometric requirements of the reduction process. Concentration of sodium dithionite should be enough to completely reduce insoluble indigo dye into its soluble leuco form. Sodium dithionite is also required to prevent possibility of unwanted oxidation of soluble leuco dye back into insoluble indigo. At lower concentration of sodium dithionite, oxidized dyestuff present in dye bath with adheres on the fibrous material and results in duller red shades. On the other hand, high concentration of free dithionite in the dye bath will cause insufficient oxidation in skying and will bleed the colour in the next successive bath resulting in low colour yield. In this case, more indigo dosing is required to get the desired shade which will disturb equilibrium of dye bath.

This case study refers to an unstable process control resulting in higher concentration of sodium dithionite in the dye bath which caused huge shade fluctuation. For an ensured optimized dyeing process, sodium dithionite concentration was regularly monitored and controlled which ultimately resulted in stabilized indigo consumption.

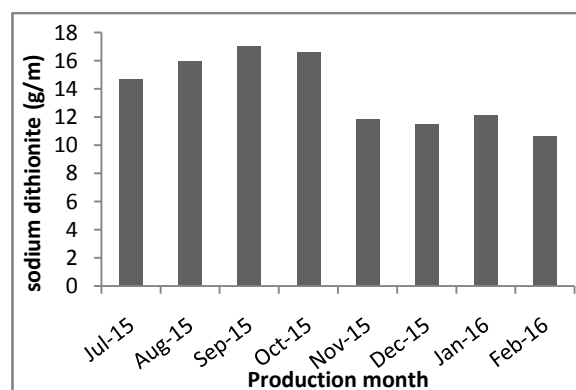


Figure 2 Month wise consumption of sodium dithionite (g/m)

Figure 2 shows the gradual reduced consumption of dithionite per meter of dyed yarn from the month of October-15 to February-16. But from July-15 to September-15 it was not effectively optimized which caused high consumption of dithionite. Sodium dithionite consumption was monitored and control was achieved from October-15 to February-16 keeping in view that there must not be any shade alteration. The statistics shows that the consumption of dithionite gm/m reduced from 16.99 gm/m to 10.62 gm/m from the month of July-15 till February-16. This optimization of sodium dithionite not only lead to the steady consumption of indigo and washing water but also reduced the cost of dyed yarn as mentioned in further discussion.

4.8. Steady Consumption of indigo

Figure 3 shows the steady utilization of indigo from November-15 to February-16. While before November-15 there was abrupt consumption of indigo because of an un-optimized dithionite concentration which resulted in higher consumption of indigo for dyeing yarn.

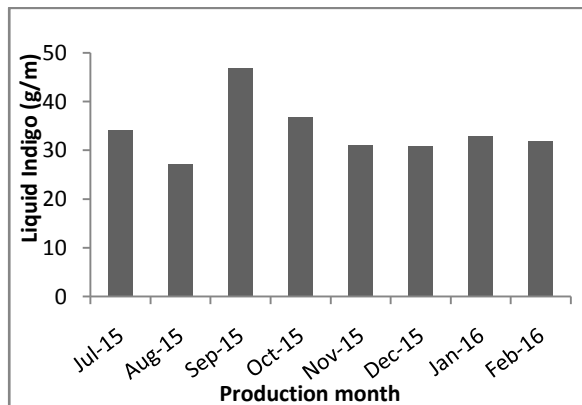


Figure 3. Production (m) versus liquid indigo consumption (grams per m)

4.9. Reduced water consumption

This study also includes optimized water consumption during pre and post washings of cotton yarn. The fix dosing of inlet fresh water for prewashing in order to remove caustic from fiber was controlled by installing digital flow meter while in post washing the water consumption became steady from October-15 to February-15 just by optimizing the concentration of $\text{Na}_2\text{S}_2\text{O}_4$. Month wise water consumption is shown in Figure 4.

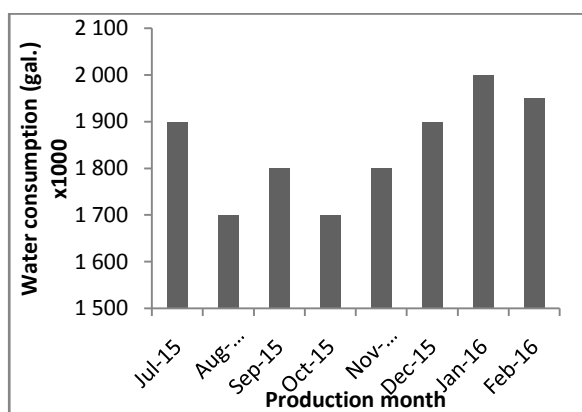


Figure 4. Month wise consumption of water (gallons) for rope indigo dyeing process

As the high concentration of sodium dithionite in the indigo baths ultimately resulted in insufficient oxidation during aeration this caused bleeding of unfixed dye in post washing chambers. The more unfixed dye stuff on the surface of the yarn, the more water required to wipe it off. In this manner an effective reduction in water

consumption has been seen by optimization of sodium dithionite concentration.

4.10. Effective cost reduction per meter

Due to stable circulation and concentration of sodium dithionite, the cost required for chemicals has been effectively reduced as shown in Figure 5. The cost reduction is about 2.88 Rs./m of dyed yarn which is specifically good for consistent and reproducible dyeing.

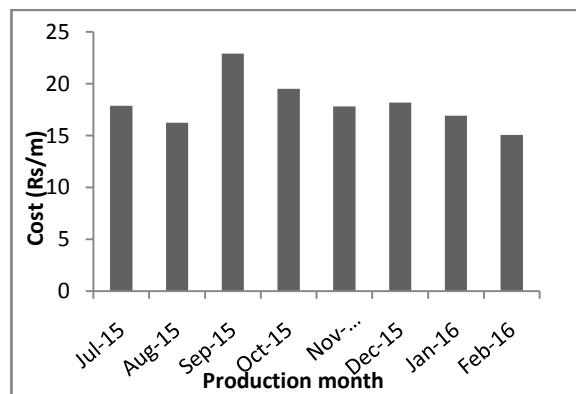


Figure 5. Month wise production cost (Rs/m)

5. Introduction About Industry

The textile unit under study was established in 1949. Operating on an area of 165,922 square meters and employing 7,650 people, it has the ability to cater to specific client needs with package deals such as product development at source, design support, shorter lead times, on-time deliveries and warehousing facilities. Their latest undertaking is the new state of the art denim mill which will take their production capacity to 36 million meters per annum.

6. Aims of Case study

There are number of factors affecting optimal indigo dyeing process. This case study was particularly focused on the optimization of indigo dyeing process by reducing consumption of overall sodium dithionite and rinsing water which ultimately reduced the dyeing cost per meter of dyed yarn. This was done by continuous monitoring and controlling of various factors for eight months without hurting the shade consistency with reduced consumption of sodium dithionite in gm/m of dyed yarn.

7. Process Optimization

Table 1 represents eight month's data for optimization of indigo rope dyeing process in the industrial unit under study.

Table 1. Month wise production data collected from industrial unit under study for indigo rope dyeing.

Month	Product (m)	Na ₂ S ₂ O ₄ (g/m)	Liquid Indigo (g/m)	Water (gal.)	Cost Rs/m
Jul-15	1,399,470	14.64	34.28	3,700	17.88
Aug-15	1,326,400	15.94	27.28	3,850	16.25
Sep-15	1,423,500	16.99	46.89	2,800	22.89
Oct-15	1,348,720	16.55	36.88	2,300	19.5
Nov-15	1,464,150	11.85	31.17	2,100	17.81
Dec-15	1,600,320	11.46	30.9	1,990	18.18
Jan-16	1,532,660	12.13	33	2,300	16.91
Feb-16	1,210,079	10.62	31.92	2,500	15.05

8. conclusion

This case study was focused on controlling the concentration of sodium dithionite in the dye bath for an effective optimized indigo dyeing process with the required shade consistency. Optimization of Na₂S₂O₄ concentration reduced the water consumption during pre and post wash and also stabilized the indigo consumption in grams per meter. Reduction in water and chemical consumption ultimately resulted in reduced cost per meter of dyed yarn. It was also expected that process optimization would maintain lesser discharge of effluent containing harmful byproducts of indigo dyeing process hence producing comparatively cleaner, cost effective and consisted dyed products.

References

- [1] M. Sala and M. C. Gutiérrez-Bouzán, "Electrochemical Techniques in Textile Processes and Wastewater Treatment," *Int. J. Photoenergy*, vol. 2012, pp. 1-12, 2012.
- [2] A. Roessler, D. Crettenand, O. Dossenbach, W. Marte, and P. Rys, "Direct electrochemical reduction of indigo," *Electrochim. Acta*, vol. 47, no. 12, pp. 1989-1995, 2002.
- [3] N. A. Memon, "Denim: Pakistan second largest exporter of denim fabrics," *Pakistan Text. J.*, vol. 64, no. 6, p. 36, 2015.
- [4] A. Gürses, M. Açıkıldız, K. Güneş, and M. S. Gürses, "Dyes and Pigments: Their Structure and Properties BT - Dyes and Pigments," A. Gürses, M. Açıkıldız, K. Güneş, and S. M. Gürses, Eds. Cham: Springer International Publishing, 2016, pp. 13-29.
- [5] A. Rutkin, "Tint without taint," *New Sci.*, vol. 229, no. 3055, p. 18 -, 2016.
- [6] S.M. Imtiazuddin and M. S. Tiki, "Indigo and sulphur rope dyeing in Denim," *Pakistan Text. J.*, vol. March, p. 22, 2010.
- [7] M. Božič and V. Kokol, "Ecological alternatives to the reduction and oxidation processes in dyeing with vat and sulphur dyes," *Dye. Pigment.*, vol. 76, no. 2, pp. 299-309, 2008.
- [8] N. Meksi, M. Ben Ticha, M. Kechida, and M. F. Mhenni, "Using of ecofriendly α -hydroxycarbonyls as reducing agents to replace sodium dithionite in indigo dyeing processes," *J. Clean. Prod.*, vol. 24, pp. 149-158, 2012.
- [9] A. Gürses, M. Açıkıldız, K. Güneş, and M. S. Gürses, "Dyeing and Dyeing Technology BT - Dyes and Pigments," A. Gürses, M. Açıkıldız, K. Güneş, and S. M. Gürses, Eds. Cham: Springer International Publishing, 2016, pp. 47-67.
- [10] M. G. Uddin, "Indigo Ring Dyeing of Cotton Warp Yarns for Denim Fabric," *Chem. Mater. Eng.*, vol. 2, no. 7, pp. 149 - 154, 2014.
- [11] J. H. Xin, C. L. Chong, and T. Tu, "Colour variation in the dyeing of denim yarn with indigo," *Color. Technol.*, vol. 116, no. 9, pp. 260-265, 2000.
- [12] R. S. Blackburn, T. Bechtold, and P. John, "The development of indigo reduction methods and pre-reduced indigo products," *Color. Technol.*, vol. 125, no. 4, pp. 193-207, 2009.
- [13] Dogan Dogan and H. Turkdemir, "Electrochemical Treatment of Actual Textile Indigo Dye Effluent," *Polish J. Environ. Stud.*, vol. 21, no. 5, pp. 1185-1190, 2012.