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# Effect of Ultra-sonication on Bio indigo Dyeing on Silk

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**ABSTRACT: Bio Indigo dyestuffs used various types of reducing agents to convert dye into leuco form in different parts of clusters in the northeastern region. The bacterial, ultrasonic, electrochemical etc. reduction process is a promising method for the reduction of Bio indigo dyestuffs. These different types of reducing agents affect the different degrees of effluent load in our environment. Sodium dithionite is well established reducing agent used for both natural and synthetic indigo which leads to environmental pollution.**

**The present study was comprised of the different vatting processes used in combination with ultrasonication to assess the redox potential of the dye bath and dye uptake by the silk yarn & fabric. The use of an ultra-sonicator enhanced the reduction of bio-indigo dyes and improved dye exhaustion on the silk material. The fastness property of the dyed silk fabric was also improved with the use of an ultra-sonicator. However, treatment of bio-indigo dye with ultrasonication along with vatting of acetol resulted in very less reduction of dye and subsequently reduced dye uptake.**

**The cat ionizer further enhanced dye uptake. Eco-friendly bio indigo dyeing process was investigated on silk yarn and fabric. Dyeing performance on Silk yarn and fabric was evaluated by measuring the colour yield parameters (K/S) of the dyed samples, Light, Washing, rubbing fastness, of bio indigo process.**

**Key words:** Bio Indigo dyes, Silk yarn and Fabric, Sodium dithionite, Acetol K/S value

## INTRODUCTION

Indigo is one of the oldest known vat dyes used in different materials by human kinds. This is the one employed in the world renowned the denim textile. The dye is insoluble in water and do not show affinity to cloths until it exists in leuco form. Caustic Soda and sodium dithionite are commonly used as reducing agent for indigo dye which was one of the causes of environmental pollution. Replacement of hydrogenated indigo solution against the stock vat reduces the chemical load from indigo dyehouses Schrott, & Saling (2000). Indigo are mainly used for yarn dyeing under the technique of continuous exhaust (Hossain, (2020); Parmar *et al.,* 1996). The pH level and dyeing temperature are the main important parameters for the indigo dye bath to produce the best affinity result of the reduced dye (Etters, 1994). Having high demands of indigodye in commercial applications, considerable work has been carried out to optimize or even substitute reducing agents both in dyestuff reduction and dyeing Xin *et al.,* (2000). However, literature information revealed the less application of indigo dyes in the silk yarn and fabric in particular, due meager knowledge among the traditional sericulture silk weaver and dyeing fellows. The conservation of dyes and optimization of the concentration of chemicals used in dye fixation has a huge advantage in dyeing improvement. Superior dye fixation could be achieved under oxygen-free dye bath where high concentrations of dye in a small amount of liquor are required. Optimal concentrations and reaction conditions in the vat could be obtained by controlling the concentration of dye, reducing agents and sodium hydroxide by adopting appropriate modern analytical methods. Therefore, in the present study, attempt has been made as been made to obtain high vatting with the application of ultrasonication at fixed timing. Ultrasonic-assisted dyeing is reported as a novel and eco-friendly method for improving the dyeing process in different decorative materials of plant origin species (Wang, 2018). Ultrasonic energy accelerates both physical and chemical reactions and homogenize chemicals uniformly due to the cavitation in a liquid medium (Atav and Yurdakul, 2016). Cavitation phenomenon is the formation of collapses and growth of microscopic bubbles (Kamel *et al.*, 2005). The cavitational bubbles undergo sudden implosion by generating "hot spots", in the localized high pressure and temperature thereby developing severe shear force and shock waves that are capable of breaking down the chemical bonding (Suslick, 1986). The special mechanical and thermal effects assisted in decreasing the size of dyestuff particles and making dye solutions more homogenized and soluble (Lee *et al.*, 2001). Generally, ultrasonication enhances the vatting rate by disintegrating the dispersed water-insoluble dye aggregates into smaller particles. Ultrasonication will make the increase of the surface of the dye as well as the shortening of the diffusion interface at the same time, as a result, the chances for collisions between the indigo molecules and the reducing agent molecules increases thus enhancing the reaction faster. Hence, under the current experimental sets, conditions are made where less application of reducing agents and improvement of dye affinity uptake under low alkaline conditions for silk yarns and fabrics dyeing were presented and discussed.

## MATERIALS AND METHODS

*A. Sources of Bio Indigo dye sample*

Bio indigo dye was procured from AMA Herbal Laboratories Pvt. Limited, Lucknow, India. Mulberry Silk fabric was used for testing indigo vat dye uptake at three different treatments each for yarn and fabrics. The raw silk fabric used was, G.S.M = 60 and mulberry silk count is of 21-22 denier.

*B. Degumming of Silk*

Raw silk fabric was degummed to remove unwanted sericin from the silk fabric before dyeing. The mulberry silk fabric was first degummed by following below described parameters: washing soap– 5 g/l, temperature – boiling, time – 2 hours, and M: L ratio – 1:50. After degumming, the silk fabric was rinsed in cold water then dried. The degummed fabric was used for further indigo dyeing treatments.

*C. Vatting Treatments*

Three different vatting treatments were tested for dye uptake affinity with silk yarn and fabric by application of ultra-sonication processes and the description details as outlined in Table 1.

**Table 1: Procedure of vatting treatments.**

| Treatments | Procedure |
|---|---|
| *Treatment-1 ($T_1$)*<br>*(Standard vatting method)* | Standard vatting method: where, for1 g indigo dyes,2 g of caustic soda (NaOH) & 2 g sodium hydrosulphite ($Na_2S_2O_4$)is added kept for 20 minutes at 50 °C for inducing reduction, Yellow colour changed in the solution indicated reduced form. |
| *Treatment-2 ($T_2$)*<br>*(Ultra-sonication + standard vatting method)* | In this treatment, indigo dye and water mixture were ultra-sonicated for 5 min at 60% amplitude (3 sec on and 3 sec off) pulsation rate followed by the conventional vatting method. |
| *Treatment-3 ($T_3$)*<br>*(Ultra-sonication + Acetol vatting method)* | Indigo dye (20 GPL) and water mixture was ultrasonicated for 5 min at 60% amplitude (3 sec on and 3 sec off) pulsation rate followed by Acetol vatting (where, Acetol 10 GPL (Gram per litre) & NaOH 12 GPL kept for 120 minutes at 70$^o$C. |

*Measurement of redox potential:*

The redox potential of dye bath for all the three treatments of both silk yarns and fabrics were measured after vatting with the help of oxidation-reduction potential meter and measured the reduction of dye.

*Bio-indigo dyieng procedure of silk yarn & fabrics:*

After vatting processes of indigo dye, all the three treatments followed same procedures of dyeing method. Color depth of 20 GPL (g/L) was taken and the dyeing of both silk yarns and fabrics was carried out at room temperature with 6 Dips & 6 nips for a period of 45 minutes. For fabric, M: L ratio of 1:50 and for yarn, 1:30 was used in dyeing. After removing fabric/yarn from the dye bath, oxidation (air oxidation) was carried out for 15 minutes with subsequent washing (2 GPL soap at 70 °C for 10 minutes) to removed unfixed dye from the fabric.

*Dye Uptake and fastness property testing:*

The dye uptake of the fabric was determined by dissolving the dyed fabric in 70% $H_2SO_4$and the absorbance reading at 436 nm in spectrophotometer (Hitachi U1900) was recorded. The K/S value L a, b was measured using. Measurement of color The K/S values of indigo dyed silk fabric and yarn were recorded from the color strength of dyed silk by using a color measuring instrument (Color spectrophotometer, model 2080). Where K/S values were calculated using the Kubelka–Munk equation from the reflectance values, where K is the absorption coefficient, S is the scattering coefficient, and R is the reflectance of the indigo dyed silk yarn and fabric (Shen, 2016).

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

The fastness property of fabric was assessed following the standard protocol of Achwall, 1985.

## RESULTS AND DISCUSSION

*A. Efficacy of different treatments on the redox potential of bio-indigo dye:*

The vatting of bio-indigo dye with caustic Soda and sodium hydrosulphite in dye bath showed redox potential of -860 mv. However, ultrasonication of dye bath for 5 min followed by normal vatting increased the redox potential by -1130 mv, subsequently the dye uptake by fabric also increased. Ultrasonication proceed by acetol vatting in treatment-3 resulted low redox potential of -440 mv with compared to other two treatments (Fig. 1). With the used of ultra-sonication for a few minutes, 100% vatting can be accomplished by generating completely oxygen free atmosphere in the dye bath (Marte, 1990). Ultra-sonication improves the vatting performance by disintegrating the detached water-insoluble dye aggregates into smaller particles. With the increase in the surface of dye molecules and simultaneous reduction of the diffusion interface, the possibility for collisions within the reducing agent and the indigo molecules increases, as a result the rate of reaction become faster (Poulakis *et al.*, 1996).

*B. Effect of dye Uptake of ultrasonic-assisted of indigo dye agent on silk materials:*

All the three different treatments were maintained for both mulberry silk yarns and fabrics. The highest absorbance of dye uptake was observed in $T_2$ treatment for both the tested silk materials followed by $T_1$ and $T_3$ (Fig. 2) which indicated the absorption fixing of more dye molecules into the silk particles. Ultra-sonication enhances the absorbance in indigo dye was also showed in extraction process too where the sonication enhances the indigo formation reaction and also the formation of other colorants too (Hidayati, 2018). Among the dyed silk materials deep blue colour uptakes are produced both in the T1 and T2 treatments but brighter and shinning intensity are developed under the ultra-sonicated solutions in both the yarn and fabric dyeing (Plate 1). Wang et al., 2018 revealed that dye uptake increased with increased ultrasonic power. Comparative study of dye uptake with and without application ultrasonic power was showed in dyeing of wood samples where a higher ultrasonic power obtained higher in dye uptake. It was also further described that uniform dispersions in the dye bath was produced in ultrasonic vibration cavitation processes. Ultra-sonication made expelled by dissolving or entrapping air molecules from fiber capillaries and interstices at the crossover of fabric into dye solution by cavitation, thereby facilitating dye-fiber contact. Further, the rate of diffusion of the dye inside the fiber and the chemical reaction involved in fiber and dye molecules are also enhanced by distorting of the insulated covered layer of fiber (Kamel *et al.*, 2003 & 2007).

*C. K/S values of ultrasonic-assisted effect in indigo dye-fixing on silk materials*

The K/S value of different treatments of ultrasonic-assisted in indigo dye showed higher value in silk yarn than the silk fabrics in the treatment, T2. More affinity of dye uptake in yarn revealed by K/S value could be due to more chance of exposing surface areas in three dimensional sides than the fabric (Fig. 3 & Table 2). The K/S value observed higher in standard vatting then in ultra-sonication with standard vatting. Wang *et al*., (2018) mentioned that ultrasonic-assisted effect during the dyeing process, the repulsive force between chromophores of dye and fiber surfaces are reduces by the sodium chloride electrolyte thereby promoting the combination of dye molecules and wood fiber. Furthermore, with increase in addition of dyeing assistant, the concentration of sodium ion increased, hence the activity of dyes improved. Among the three treatments, application of ultrasonication in acetol vatting indicated lesser K/S value than the standard vetting treatment, this may be due to the poor reducing property of acetol in compare to caustic soda and sodium hydrosulphite. The alkalinity of caustic soda and sodium hydrosulphite might develop stronger fixing agent that enhances the dye uptake (Hu *et al*., 2016).

*D. Fastness Property of indigo dyed silk materials*

The fastness property of indigo dyed silk fabrics showed that $T_1$ has wash fastness rating of ¾ with similar rating to $T_2$ (Table 3). Among the three treatments, $T_3$ obtained highest rating of 5, it could be due to very less dye release by the silk fabric compare to other treatments.

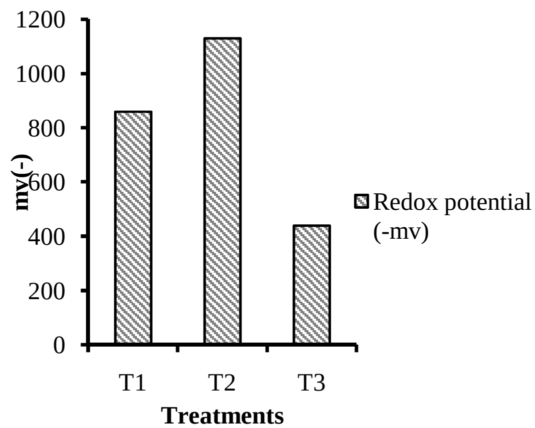


**Fig. 1.** Redox potential of different vatting treatments.

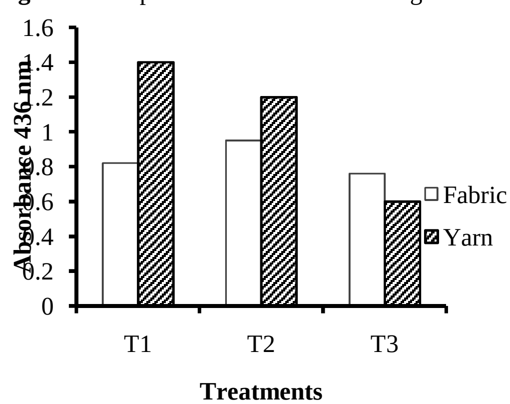


**Fig. 2.** Effect of different treatments on dye uptake absorbance.

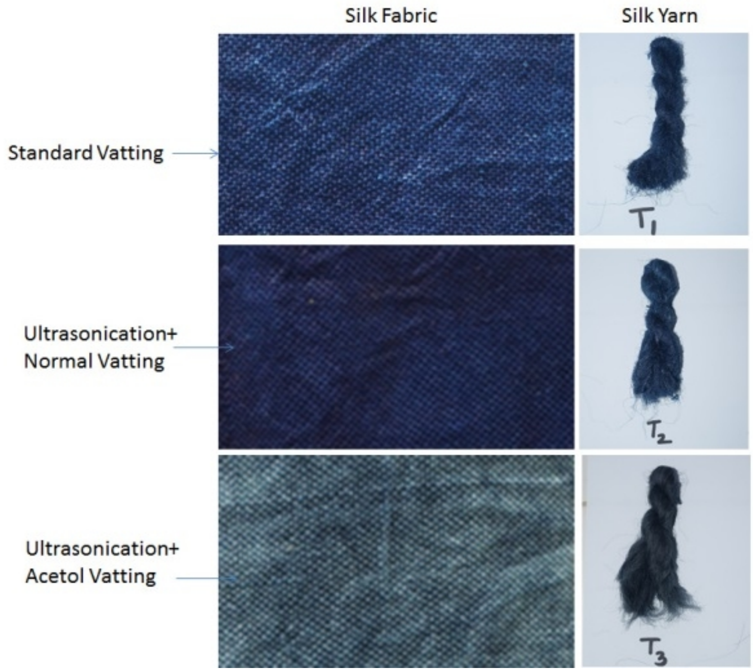


**Plate 1.** Effect of different treatments on shades on silk fabric and yarn.

**Table 2: Spectrophotometeric reading of dye silk fabric and yarn.**

| | K/S | L | a | b |
|---|---|---|---|---|
| **Fabric** | | | | |
| Standard Vatting | 12.50 | 17.49 | 8.20 | -15.69 |
| Ultrasonication + Standard Vatting | 10.75 | 19.57 | 6.91 | -15.44 |
| Ultrasonication + Acetol Vatting | 0.64 | 64.69 | 0.49 | -4.26 |
| **Yarn** | | | | |
| Standard Vatting | 7.76 | 25.58 | 1.76 | -17.08 |
| Ultrasonication + normal Vatting | 12.16 | 19.33 | 3.63 | -16.12 |
| Ultrasonication + Acetol Vatting | 5.52 | 30.95 | -0.04 | -5.26 |

Further, it also noted that ultra-sonication together with standard vatting application improved the rubbing fastness property of the silk fabrics. Light fastness property also observed improvement in both the ultra-sonicated applied treatments, $T_2$ and $T_3$.

It was also apparent that irrespective of dye used, the vat dye exhibits good wash fastness in terms of shade change and very little staining occurred to the adjacent fiber. The wash down will be influenced by the total amount of indigo, which had been fixed on the yarn or fabric. Depending on the depth of penetration of indigo, during the garment processing, the part of the dyestuff removed will vary (Bechtold *et al*., 2008).

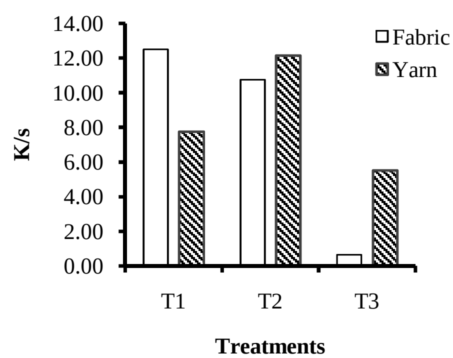


**Fig. 3.** Colour strength (K/S) of the indigo dyed on silk materials**.**

**Table 3: Color fastness property of indigo dyed silk fabric to washing, rubbing and light fastness.**

| Treatments | Wash fastness | Rubbing fastness | | Light Fastness |
|---|---|---|---|---|
| | | Dry | Wet | |
| **Fabric** | | | | |
| Treatment-1 | ¾ | 3 | 3 | 4/5 |
| Treatment-2 | ¾ | ¾ | ¾ | 5 |
| Treatment-3 | 5 | 4 | 4 | 5 |
| **Yarn** | | | | |
| Treatment-1 | ¾ | 3 | 3 | 4 |
| Treatment-2 | ¾ | ¾ | ¾ | 5 |
| Treatment-3 | 3/4 | 3/4 | 3/4 | 4/5 |

## CONCLUSION

The use of ultra-sonicator enhanced the reduction of bio-indigo dyestuffs and better uptake on silk yarn. The fastness property of the silk fabric was also improved with the use of ultra-sonicator. However, vatting with acetol resulted in less reduction and poor dye uptake in silk fabric. Application of ultra-sonication incorporating with standard vatting in indigo dye improve overall performance and will require less amount of dye and reducing agents to obtain similar shades as compared to standard vatting process, which in turns could have economic benefit and lesser environmental pollution. Ultrasonic dyeing technique has also been widely adopted to decrease energy consumption and increase productivity in textile dyeing industry. Ultrasound-assisted dyeing treatment approved not only in increasing the dye uptake, but also in reducing the amount of dyestuff used. Therefore, ultrasonic energy application in dyeing improved environment impact by reducing consumption of supportive chemicals, and lowering the overall cost of processing. It will enhance the use of natural dye with less pollution.

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