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Studies on Sustainable Silk Wet Processing Practices in South Indian Silk Clusters

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Abstract: Silk has been celebrated for its superior textile qualities, including strength, elasticity, excellent drapability, and natural lustre. Despite these advantages the wet fastness of dyed silk remains problematic, particularly in the context of conventional / traditional dyeing processes prevalent in silk clusters of southern India. These traditional units commonly employ open pan vessels with direct heating and operate without adherence to standard processing methods and quality standards regarding dyes, chemicals, auxiliaries, and water. Such practices, often managed by experienced but informally trained personnel, lead to inadequate color fastness, diminished tensile strength, and uneven dyeing results. In response, the improved Tub dyeing method has emerged as a promising contemporary alternative. This process utilizes stainless steel tubs lined with steam pipes and steam boilers for controlled heating, alongside standardized dyes from reputable suppliers, thereby enabling improved process consistency and reproducibility. In this study, silk yarns were dyed using both conventional-traditional open pan method and the Tub dyeing technique with acid, direct, and metal complex dyes. The dyed samples were evaluated for dye uptake, color fastness, and tensile properties. Comparative analysis revealed that silk fabrics processed via the Tub dyeing method exhibited significantly higher exhaustion, better color fastness, and better retention of tensile strength relative to those treated with the traditional method. These findings highlight the importance of Tub dyeing technique as a viable means to upgrade traditional silk processing units and processing standards, enhance dyed silk yarn performance, and address key quality challenges, thereby contributing to the competitiveness of the Indian silk industry in both domestic and export markets

Keywords: *Silk; Conventional Dyeing; Tub Dyeing; Colour Fastness; Effluent Characteristics.*

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I. INTRODUCTION

Silk, a natural protein fibre from insect source is also known as "Queen of fibres" due to its strength, unparalleled lustre and wonderful feel. India is the second largest silk producer and highest silk consumer in the world. Table 1 below represents the production information of Mulberry, Tasar, Muga and Eri silks in India [1].

Table 1 Raw Silk Production Statistics of India (Unit: MT)

Year	Mulberry	Tasar	Eri	Muga	Total
2020-21	23,896	2,689	6,946	239	33,770
2021-22	25,818	1,466	7,364	255	34,903
2022-23	27,654	1,318	7,349	261	36,582
2023-24	29,892	1,586	7,183	252	38,913
2024-25	31,119	1,884	7,886	232	41,121

Silk fibre undergoes three distinct operations during its conversion from the cocoon stage to ready to use fabric/apparel stage. Broadly it can be classified as reeling, weaving and wet processing [2]. The final operation of wet processing involves combinational processes of chemical pre-treatments, dyeing and finishing [3, 4]. The wet processing of silk is possible at three stages viz, in the fibre form which is mainly used for spinning sector purpose, in yarn form prior to weaving and post weaving that is in the fabric form. In India, around 70-80% silk is processed in the yarn stage, where in the processes of degumming, bleaching, dyeing and finishing treatment is carried-out with water being the medium for most of the activities [5, 6]. The present study focuses on sustainable standing bath dyeing practices carried-

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out in the silk yarn processing units. Traditional dyeing unit and contemporary Tub dyeing units have both been studied in this attempt. Three classes of dyes (acid, metal-complex & direct) have been used for comparative analyses. Fastness assessment of the dyed silk hanks and effluent characteristics have been analysed to understand the sustainability aspects with respect to other textile sectors.

II. MATERIALS AND METHODS

➢ *Materials*

Two-ply twisted Mulberry silk (filature yarn, 20/22 denier) was used for all experimental dyeing processes conducted in both conventional and contemporary (tub dyeing) units. The dyes selected for the study include acid, direct, and metal-complex dyes, commonly utilized in the silk processing industry. The specific dyes used were:

- Acid Dye: Acid Red RS (C.I. No. 23635)
- Direct Dye: Chrysophenine G (C.I. No. 24895)
- Metal-Complex Dye: Bordeaux-B (C.I. Violet 90)

➢ *Methods*

The methodology adopted for the study is outlined in the flowchart below (Figure 1).

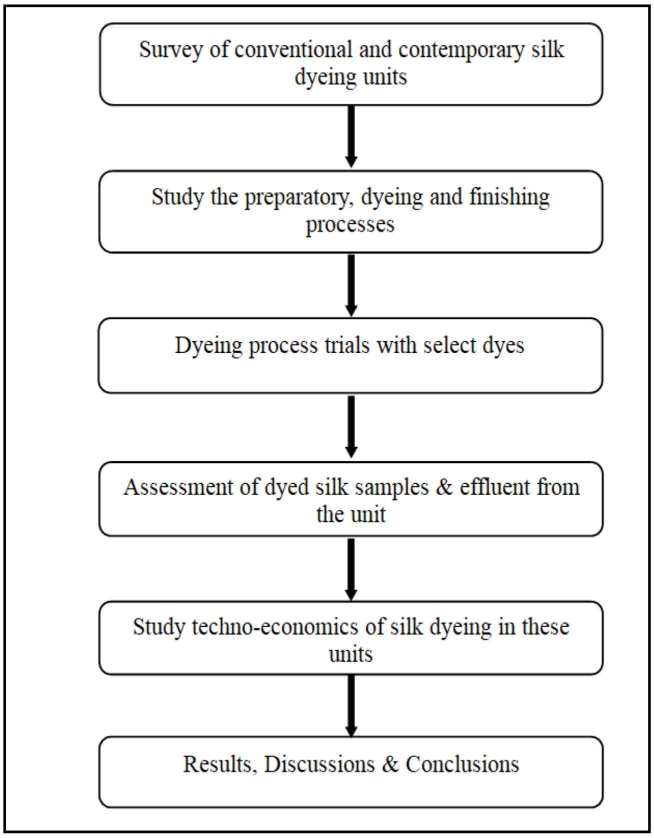

Fig 1 Flowchart of the Study

- *Traditional/Conventional Dyeing Units*

In conventional dyeing units a very traditional way of dyeing by employing open pan/vessels with direct heating are still in practise in many of the silk clusters of southern India. The conventional dyers are still not completely aware of standard processing methodology, dye class and other chemicals used in the silk dyeing. Generally, there is no control over the process parameters like temperature, pH, time and concentration of dyes and chemicals. Sanding bath concept is prevalent in silk yarn dyeing units (both conventional and contemporary tub dyeing units) wherein degumming and dyeing performed in single bath repeatedly for different batches. The procedure of this standing bath method is as follows: First, the degumming bath would be prepared by taking material to liquor ratio (MLR) 1:40, soap flakes 5-7gpl along with soda ash 0.5-1gpl. Here pH of the bath would be around 10.0-10.5 and the silk hanks are treated in this bath for 10-15 min at 90-100°C and end point is ascertained based on touch feel of silk hanks purely by experience. After the degumming process, silk hanks are taken out and washed with cold water and excess water in silk hanks is removed by using hydro-extractor. Now, along with acetic acid (to bring bath pH near neutral) the prepared dye solution would be added to the degummed bath according to the percentage of the shade required. Here MLR is maintained at 1:30 by adding additional amount of water to the bath. Dyeing is carried-out for degummed silk hanks in this standing bath for the first color for 5-10 min at 90°C. Then the dyed silk hanks are removed from the bath, washed with cold water. After washing the material, scrooping treatment is given with 2-4gpl glacial acetic acid at room temperature for 3-5min followed by drying under shade. Additional water is added to the standing dyeing bath and second dye bath is prepared by adding dye solution for second shade by mixing dye combinations, purely based on the experience of dyer. Starting from light shades, medium and darker shades are obtained by dyeing silk hank lots in the same standing bath through dye bath prepared by adding dye solution for next shade by mixing dye combinations. Such standing bath is used to dye at-least eight silk hank lots (in traditional units) whereas at-least four to five silk hank lots (in tub dyeing units) before partially draining and preparing new standing bath. Methodology of using standing bath is same in both conventional-traditional dyeing units and contemporary tub dyeing units, only differences being changes in processing parameters and use of dye chemicals.

- *Tub Dyeing Units*

Tub dyeing units are improved version of Traditional dyeing units. These dyeing houses are equipped with stainless steel Tubs fitted with steam pipes at bottom (indirect heating of bath), steam boiler, weighing balances, water softening plants and hydro-extractors to achieve better dyeing and working conditions. The steam boiler is used to heat the dye bath containing dye solution which helps to penetrate the dye into the fibre. Dyers have better experience and theoretical knowledge on the class of dyes and the chemicals used in the process and also better control over the process parameters.

➢ *Dyeing Procedure used for Silk Dyeing:*

- *Dyeing of Silk with Acid Dyes*

The required quantity of dyestuff solution is taken in the dye bath and 0.5% acetic acid is added (MLR 1:30) at 40°C. The silk hanks are suspended on smooth aluminium rods is worked in the dyebath for 10 minutes and the temperature is

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gradually raised to 85°C. During the next 30 minutes a further 3.5% acetic acid is added in three to four portions. Dyeing is continued for a further period of 30 minutes at 85°C. The yarn is then removed, rinsed thoroughly in cold water, scrooping finish imparted, squeezed well and dried under shade.

- *Dyeing of Silk with Direct Dyes*

The dye powder paste is first prepared with cold water and small amount of soda ash, in second stage boiling water is added to the paste with constant stirring to dissolve dye powder completely. This dye solution is added to dyeing bath. The silk hank is introduced into the dye bath containing dissolved direct dye solution and required amount of Glauber salt is added. The temperature of dye bath is then gradually raised to 85-90°C and dyeing is continued for 30-45 minutes. After dyeing process, the yarn is removed, rinsed thoroughly in cold water, scrooping finish imparted, squeezed well and dried under shade.

- *Dyeing of Silk with Metal-Complex Dyes*

The required amount of dyestuff solution is taken in the dye bath and 4-6% ammonium sulphate and 10% Glauber salt is added to the solution. Silk hanks are entered in the dye bath at 40°C. Temperature is gradually raised and dyeing is continued for 45-60 minutes at 85 to 90°C. The material is then taken out, rinsed thoroughly in cold water, scrooping finish imparted, squeezed well and dried under shade.

➢ *Tests Conducted for Silk Yarn*

In this study all the tests were carried out at 25°C±2°C temperature and 65%±2% relative humidity. Minimum of 10 readings were taken for each test, the average reading was considered.

- *Physical Tests*

The physical and mechanical performances of dyed silk were assessed using standard method as listed below in Table 2.

Table 2 Physical and Mechanical Tests Performed on Silk

Sl. No.	Test Parameter	Test Method	Instrument Used
1	Breaking load(grams), Tenacity (grams /denier) Elongation (%) test	IS 1670-1991	Instron Tensile Testing Machine (Model 5500R)
2	K/S (colour measurement)	CIE Reflectance method	Computer Colour matching

- *Chemical Tests*

The following chemical tests were performed on dyed silk yarn for comparative study between conventional and contemporary (Tub Dyeing) silk processing, as listed below in Table 3.

Table 3 Chemical Tests Performed on Dyed Silk

SL. No	Test Parameter	Standard Test Method	Instrument Used
1	Wash Fastness	IS 687:1979	Laundrometer
2	Rubbing Fastness	IS 766: 1988	Crock meter
3	Perspiration Fastness	IS 971:1983	Perspirometer
4	Light Fastness	IS 2454:1985	Xenon test
5	Biological Oxygen Demand	IS 3025	BOD incubator
6	Chemical Oxygen Demand	IS 3025	COD glass flask

III. RESULTS AND DISCUSSIONS

➢ *Tenacity and Elongation*

Silk exhibits high strength, with raw silk yarn showing a tenacity of 3.78 gm/den and an elongation of 16.4%, owing to its linear and crystalline polymer structure. The tenacity of dyed silk yarns varies with dye class due to pH, processing conditions, and material loss during degumming and dyeing. Results indicate that tub-dyed yarns retain higher tenacity than traditionally processed yarns, while elongation values are marginally higher in traditional methods, though the difference is not statistically significant Table 4.

Table 4 Tenacity and Elongation of Silk Yarn Samples

Sample Description	Traditional Treated Silk		Tub Dyeing	
	Tenacity (gm/den)	Elongation (%)	Tenacity (gm/den)	Elongation (%)
Degumming	3.40	17.1	3.62	17.2
Acid dyed yarn	3.32	16.8	3.45	17.0
Direct dyed yarn	3.28	16.8	3.39	17.1
Metal complex dyed yarn	3.35	16.7	3.42	16.9

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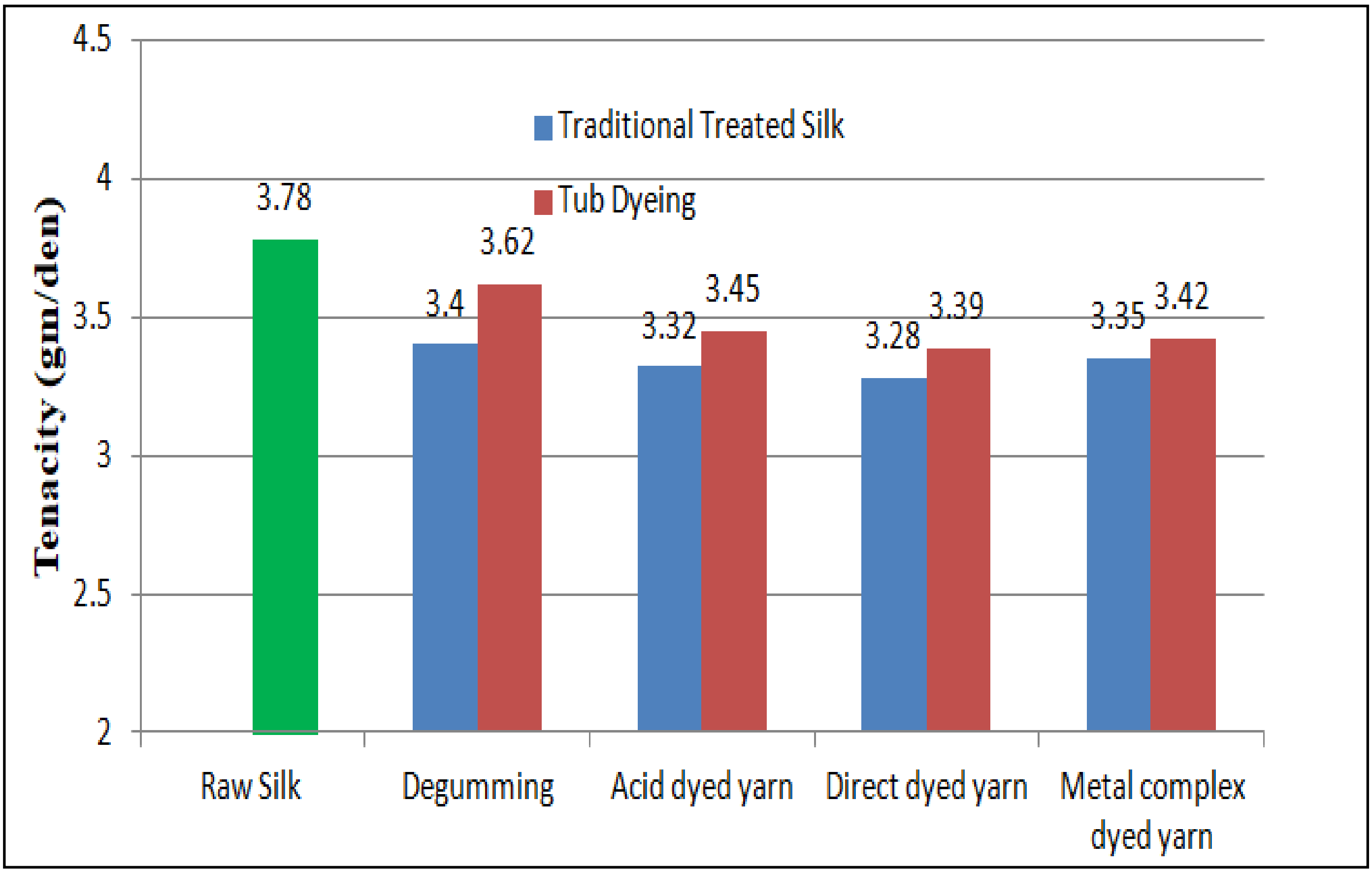

Fig 2 Strength of Silk Yarn Samples

➢ *K/S Values of Acid, Direct and Metal Complex Dyes on Silk:*

Colour strength values displayed in Table 5 shows that with all similar conditions silk yarn hanks dyed by contemporary tub dyeing method display higher colour strength (K/S) values which could be attributed to the better dyeing process parameters, process controls and improved dye exhaustion. The K/S Values of Acid, Direct and Metal Complex Dyes on Silk yarn for Traditional dyeing and Tub dyeing method is measured with 2% shade.

Table 5 Colour Values of Acid, Direct and Metal Complex Dyed Silk Yarn

Sample Description	Shade %	K/S value	
		Traditional dyeing method	Tub dyeing method
Acid dyed yarn (Acid Red RS)	2	12.51	13.42
Direct dyed yarn (Crysophenine G)	2	5.62	5.85
Metal complex dyed yarn (Violet 90)	2	13.20	15.53

➢ *Colour Fastness to Washing Test:*

Table 6 shows the Colour Fastness to Washing Test Results of Traditional dyed and Tub dyed Silk Yarn. The washing fastness results obtained from both the methods have moderate to poor results. The silk dyed with acid dyes yield poor fastness properties and direct dyes have moderate fastness properties in both the methods. Metal complex dyes have very good wash fastness results in both the methods. Overall Tub dyed silk yarn shows much better fastness results in acceptable (good to very good) range compared to Traditional dyeing method.

Table 6 Colour Fastness to Washing Test Results Dyed Silk Yarn

Class of Dye	Washing Fastness					
	Traditional dyeing method			Tub dyeing method		
	CC	SC	SS	CC	SC	SS
Acid dyed yarn (Acid Red RS)	2-3	1-2	2-3	2-3	2-3	2-3
Direct dyed yarn (Chrysophenine G)	3-4	3-4	4	4-5	3-4	4-5
Metal-complex dyed yarn (Violet 90)	4	4	4-5	4-5	4	4-5

*CC- Colour Change, *SC- Staining on Cotton, *SS- Staining on Silk

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➢ *Colour Fastness to Perspiration:*

Mulberry silk is majorly used for clothing especially in sarees. So, in this regard colour fastness against perspiration is a crucial consideration. Both an acidic solution and an alkaline solution were used in this test. The results for acid and alkaline perspiration are given in Table 7. From the table, it is clear that for all three classes of dyes—acid, direct, and metal complex dyes—perspiration fastness for alkaline solution was superior to that of acidic solution.

Table 7 Colour Fastness to Perspiration Test Results Dyed Silk Yarn

Class of Dye	Acid Perspiration						Alkaline Perspiration					
	Traditional dyeing method			Tub dyeing method			Traditional dyeing method			Tub dyeing method		
	CC	SC	SS	CC	SC	SS	CC	SC	SS	CC	SC	SS
Acid dyed yarn (Acid RedRS)	2-3	1-2	2-3	2-3	2-3	3	3-4	3-4	2-3	3-4	3-4	3
Direct dyed yarn (Chrysophenine G)	3-4	3-4	4	4	3-4	4-5	3-4	4	3-4	4-5	4	4-5
Metal-complex dyed yarn (Violet 90)	4-5	3-4	4	4	4-5	4-5	3-4	4-5	3-4	4-5	4	4-5

*CC- Colour Change, *SC- Staining on Cotton, *SS- Staining on Silk

➢ *Colour Fastness to Rubbing and Light:*

Silk dyed under Traditional dyeing and Tub dyeing methods using different class of dyes showed similar grading for dry rubbing and wet rubbing. Wet rubbing fastness results are poor in both the methods. For Light fastness test the dyed samples are exposed to Xenon Light for 16 hours. It is clear from Table 8 that tub dyed silk yarns have greater light fastness (on scale of 1-8) than conventional dyeing methods for all classes of colors evaluated. Acid dyes perform somewhat lower in terms of light fastness. Direct dyes and metal complexes both exhibit decent light fastness for indoor use.

Table 8 Colour Fastness to Rubbing and Light Fastness Test Results Dyed Silk Yarn

Class of Dye	Rubbing Fastness				Light Fastness	
	Traditional dyeing method		Tub dyeing method		Traditional dyeing method	Tub dyeing method
	Dry rubbing	Wet rubbing	Dry rubbing	Wet rubbing		
Acid dyed yarn (Acid Red RS)	4-5	4	4-5	4-5	3-4	4
Direct dyed yarn (Chrysophenine G)	4-5	3-4	4-5	3-4	4	4-5
Metal-complex dyed yarn (Violet 90)	4-5	3-4	4-5	3-4	5-6	6-7

➢ *Effluent Water Characters:*

Effluent characters from both the traditional and contemporary dyeing units show that volume of effluent is very low in traditional units due to standing bath used for higher silk lots. Due to low volume of water used and high unfixed dyes, all the effluent parameters from traditional dye house display significant higher values as evident from Figure 3.

The pH value of Tub dyed effluent water is within the acceptable limits whereas the Traditional dyed effluent has slightly higher. The effluent load generated in the Traditional dyeing activity found much higher level when compared with BOD and COD of Tub dyed effluent generated from the processing industry. With high values of BoD and CoD in the traditional dyeing units indicate poor effluent quality with presence of both biodegradable and non-biodegradable pollutants, requiring a broader treatment approach and suggesting potentially more complex, non-biodegradable contaminants are present and a greater risk to aquatic life. It is also to be noted that traditional silk dyeing units use far less water and release less effluent volume (around 700 litres for dyeing 100 kg silk yarn per day) compared to tub dyeing units which release around 1500 litres per day of effluent for dyeing 100 kg silk yarn (normalized). No proper weighing of chemicals during dyeing process, excessive dosage of chemicals to reduce treatment time, unfixed dyes, sourcing chemicals locally as ready treatment packs without knowing composition using standing baths for higher silk lot dyeing, washing, scrooping processes could also be some of the reasons for low volume and higher effluent load from traditional dyeing units.

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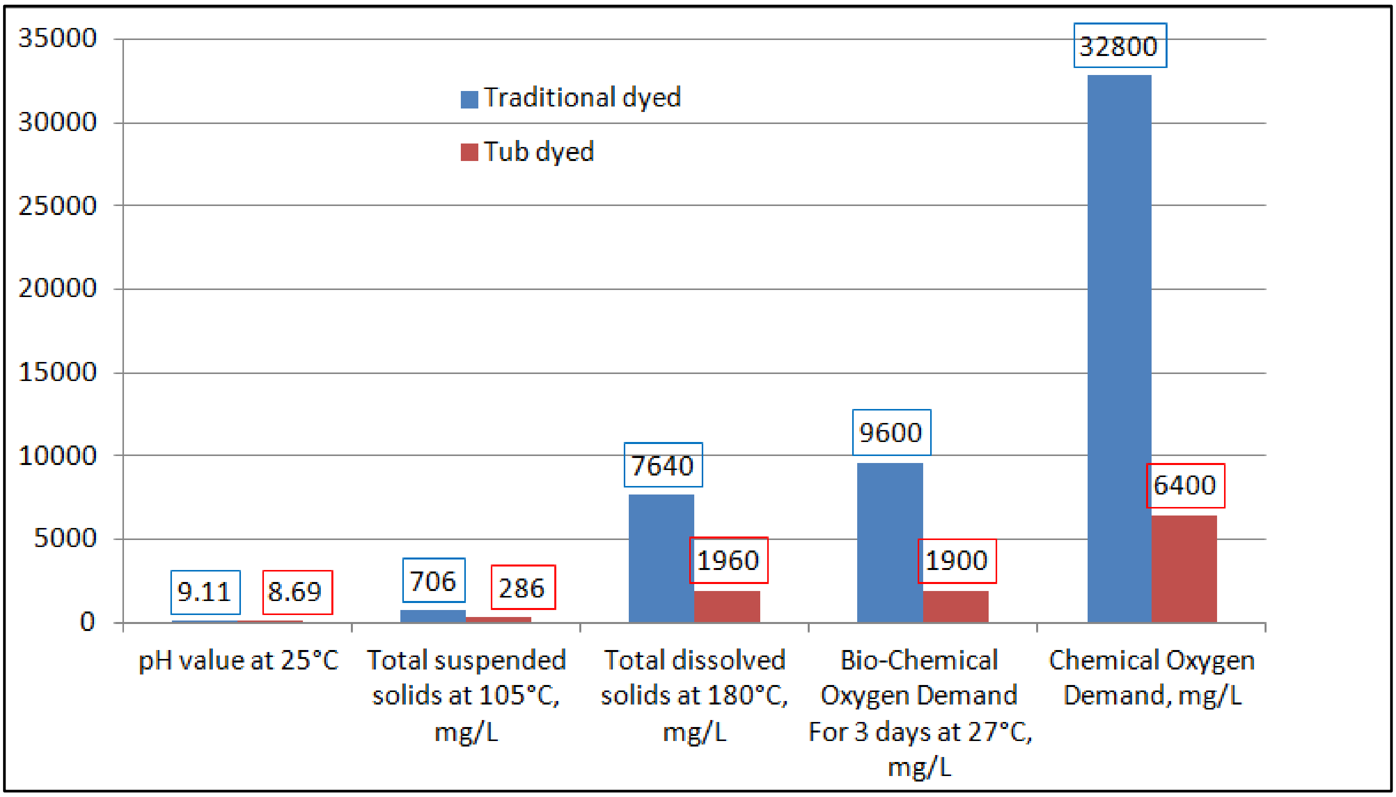

Fig 3 Effluent Characteristics from Silk Wet Processing Units

Table 9 Effluent Characteristic for Silk Wet Processing Unit

Parameter	Traditional dyed	Tub dyed
Effluent Quantity (100kgs Silk Dyeing)	700	1500
pH value at 25°C	9.11	8.69
Total suspended solids at 105°C, mg/L	706	286
Total dissolved solids at 180°C, mg/L	7640	1960
Bio-Chemical Oxygen Demand For 3 days at 27°C, mg/L	9600	1900
Chemical Oxygen Demand, mg/L	32800	6400

Table 10 Characteristics of Silk Wet Processing Effluent

Process	Composition	Effluent Nature
Degumming	Gum (sericin), waxes, grease, soda ash, sodium silicate, fibres, surfactants & soaps.	High in BOD, COD, suspended solids, dissolved solids, High pH
Bleaching	Soda, sodium silicate, hydrogen peroxide, surfactants, sodium phosphate	Alkaline, moderate suspended solids (SS)
Dyeing	Various dyes, mordants, reducing agents, acetic acid, soap	Strongly coloured, High COD, dissolved solids, low SS
Printing & Finishing	Pastes, starch, gums, oil, mordants, acids, soaps. Inorganic salts.	Highly-coloured, High COD, oily appearance, high SS

Overall characteristics of the effluent from different process stages and dyeing/printing activities have been summarized in the Tables 9 & 10. Compared to other textile processing mills, water consumption pattern in silk yarn dyeing units in silk clusters of southern India is very low. Main reason for this is standing bath usage in silk processing units which although has higher BOD and COD values but the overall effluent generated volume is very low. Also, silk is processed in mildly alkaline & acidic baths and single bath scrooping finish is imparted due to which use of water for neutralizing and repeated washing is also very low.

IV. CONCLUSIONS

Wet processing of silk plays a major role in the marketing of silk either in domestic or export market. It becomes very important that sufficient care is given to enhance the natural beauty of silk. The standard and optimum methodology for degumming and dyeing of silk ensures minimum loss of lustre and strength. Through the present study, it has been summarized that compared to traditional dyeing method, the dyeability, color yield, dye bath exhaustion, color fastness, and tensile properties of silk dyed with acid, direct and metal complex dyes using contemporary tub dyeing method was better which is mainly due to better control over the processing conditions.

Sustainability has become an essential attribute of today's textile and fashion industry. The process of transforming textile industry into more sustainable one is very sensitive, needs a lot of knowledge, skills and commitment. Use of silk as sustainable fibre in textile is a part of this process and a step towards this milestone. Silk has carved-out a niche brand since ages and demonstrated strength in fashion market; silk is much better and useful alternative among dominant and high impact natural fibres. Cotton has captured big market shares because of its properties but cotton growing is not favourable for the environment it requires lots of water, pesticides and fertilizers. Silk in contrast is much better in many aspects (Water, Employment Generation, Pesticides & Fertilizers). Sustainable practices in silk wet processing needs focus on the following:

- Use of ecological materials and/or processes for pre-treatment and dyeing;
- Minimization of water use, reuse of wastewater, and treatment of effluents;
- Incorporation of efficient and economically viable technologies;
- Development of partnerships between the different silk sectors i.e. Reeling, Wet Processing, Weaving, Garment production & Fashion; and
- Dissemination of information to all silk professionals, especially those professionals who are involved in the design process

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