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Standardization and optimization of different mordants on the dyeing efficiency of beetroot (*Beta vulgaris*) on silk fibre

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Abstract
In the current study, type of different mordants (Ferrous sulphate, alum, copper sulphate, acetic acid and citric acid) were standardized on silk fibre using beetroot dye. The dyed fibre samples were analyzed for absorbance, color coordinates and color strength. Standardization of different mordants was done while dye percentage was kept constant @ 10% (based on the outcome of preliminary testing). Based on the findings, it was determined that 10% beetroot dye combined with 3% alum produced better results on silk fibre.

Keywords: Mordants, dyeing efficiency, beetroot, *Beta vulgaris*, silk fibre

Introduction

Textile materials (natural and synthetic) used to be coloured for value addition, look and desire of the customers. Since ancient times, natural dyes have been used extensively for colouring food-grade materials, leather, and natural fibres like wool, silk, and cotton. This technique of dyeing was practiced by ancient people before thousands of years (Geelani *et al.*, 2015; Prabhu and Bhute, 2012) [8, 19]. Synthetic dyes, which are widely available for economical price and generate a wide range of colours, can occasionally cause skin allergies and other health problems for people, produce toxicity and other chemical hazards during their synthesis, undesirable/hazardous/toxic chemicals etc. Worldwide, growing consciousness about organic value of eco-friendly products has generated renewed interest of consumers towards use of textiles (preferably natural fibre product) dyed with eco-friendly natural dyes. Recently, the majority of industrial dyers and textile exporters have begun to relooking the maximum potential of using natural dyes for dyeing and printing various textiles in order to target specialized markets. India is a potent land to produce natural dyes industrially, as India is considered as one of the seventeen mega diversity countries globally (Singh and Srivastava, 2015) [23]. In recent past considerable research work started to explore new sources to fulfill demands of natural dyes in industrial level with low cost, more dye exhaustion quality and better fastness properties (Geelani *et al.*, 2015) [8].

Beetroot colour is widely used industrially, due to its red colour, from the pigment betalain mainly from betanin, betanidin, and betaxanthin (Singh *et al.*, 2017) [22]. Betalains are water – soluble nitrogen containing tyrosine derived vacuolar pigments (Hossain *et al.*, 2020) [12]. Betalains consist of betacyanins (red-violet) and betaxanthins (yellow-orange) (Stintzing *et al.*, 2004) [25]. The major betacyanin in beetroot is betanin and accounts for 75–95% of the red pigment (Jahan *et al.*, 2021 and Ninfali *et al.*, 2013) [14, 18]. Natural dyes are mostly substantive dye which requires mordants to prevent fading off the colour from the yarn due to wash and exposure to light (Siva, 2007) [24]. A chemical known as a mordant can successfully bond to yarn and create a ligand that dyes the yarn naturally. Mostly natural salts are used as mordants due to its easy availability. Common metal mordants are alum, ferrous sulphate, copper sulphate etc (Geelani *et al.*, 2016) [7]. However, different mordants can produce different shades with same dye (Ado *et al.*, 2014) [1]. Natural colour can adhere to natural fibres with ease. The proper and standardized dyeing processes for that specific fibre-natural dye system must be implemented for the successful commercial application of natural dyes for each given fibre. The purpose of this study is to create colour hues using various mordants and extract textile dye from beetroot peel.

Further, percent absorption, colour coordinates, colour strength (K/S), evaluation of light, wash and light properties of extracted dyes has been studied.

Materials and Methods

Collection of material

Silk fibre was procured from college of Temperate sericulture, SKUAST- K. Beetroot were obtained from local market, Srinagar. All the mordants used were of laboratory grade.

Preparation of dyeing material

The shade-dried plant material was washed with water to get rid of any remaining dirt, and it was then dried in a tray dryer at 80 °C for two hours before being ground into a fine powder. The substance was subsequently put through a standard test sieve (BSS-14). The powdered sample was used for further studies.

Extraction of dye from powdered beetroot Peel

The powdered plant material was soaked overnight and then boiled in distilled water for 4 hours. 1L of distilled water was used for 100 g plant material. Liquid extract was concentrated to 1/4th of its original volume at 80-85 °C to obtain the final concentrated dyeing extract (Chungkrang *et al.*, 2021) [5]. The extract was collected by filtration through muslin cloth.

Scouring

With the M: L ratio at 1: 50, test material was washed with 2% non-ionic soap (Labolene) at 50 °C for 20 min. The scoured material was then washed with lots of tap water and allowed to air dry. Prior to dyeing, the scoured material was soaked in distilled water for 30 minutes.

Dyeing of silk fibre

Process of dyeing was carried out in a water bath maintaining material to liquor ratio 1:100. The scoured samples were treated with different metal salt, only simultaneous mordanting with metal salts was carried out. Simultaneous mordanting method was considered as suitable mordanting method for each mordant. Similar observation has been obtained by Kumersan (2014) [16]. The mordant such as copper sulphate (3%), Alum (3%), citric acid (3%), acetic acid (3%), and Ferrous sulphate having (3%) were used. The scoured and washed test material were immersed in beaker containing dyeing solution at room temperature and raised to 38 °C with gentle stirring continued for 90 minutes. The material was taken out and washed 2-3 times with 1% of detergent and water. Dyed samples were squeezed and dried at room temperature (Geelani *et al.*, 2021) [10]. Dyeing of the samples was done at pH 6.5.

Standardization of mordant

Alum (3%), copper sulphate (3%), acetic acid (3%), citric acid (3%), ferrous sulphate (2%) has been screened

Determination of percent absorption of dye

The percent absorption of the natural dye by the test material was calculated by recording the optical density of the dye solution both before and after dyeing process using UV based absorption spectrophotometer over the range of 560 nm. The percent absorption of natural dye was calculated by using the following equation (Divya *et al.*, 2013) [6].

$$\text{Percent absoprtion} = \frac{\text{O.D before dyeing} - \text{O.D after dyeing}}{\text{O.D before dyeing}} \times 100$$

Determination of CIE colour coordinates (L*a*b*C*h° and ΔE)

The CIE Colour coordinates in terms of L*a*b*C*h° and ΔE values of the dyed and test samples were determined by computer colour matching system. The colour parameters L*(depth of colour), a*(positive value redness and negative greenness) and b* (positive value yellowness and negative value blueness) were recorded. The said values were ascertained for all mordants used in study.

Determination of Colour strength (K/S) value

The colour strength (K/S values) of the dyed test samples was evaluated using computer colour matching system (Geelani *et al.*, 2015) [8].

Evaluation of the fastness properties of the dyed fibres

It was done as per ISO standard test methods by measuring light, washing and rubbing fastness values. The colour fastness is usually rated either by loss of depth of colour in original sample or is expressed by staining scale, Samanta and Agarwal, (2009) [20]. The fastness rating was assessed using grey scale as per ISO-105-A02 (loss of shade depth) and ISO-105-A03 (extent of staining). Methods for evaluating these properties are as follows:

Fastness to light (ISO 105-B02)

Samples of the dyed material were exposed simultaneously to a light source of Xenon arc lamp using a Xenon test chamber (Model Xe-1-S, Q-SUN) under specified conditions for 10 hours (Geelani *et al.*, 2015) [8].

Fastness to Rubbing (ISO 105 X-12)

Colour fastness to rubbing (dry and wet) was assessed as per ISO-105-X12 method using a manually operated PROLIFIC Crock Meter with finger of 1.6 cm diameter moving to and fro in a straight line over a 10±0.30 cm track on specimen, with downward force of 9 N (Geelani *et al.*, 2015) [8].

Fastness to washing (ISO 105-C01)

The dyed fibre was taken sandwiched between two adjacent undyed white fabric and stitched round the edges. The soap solution contained 5 gm of soap per litre of distilled water. The solution was heated at 60 °C for 30 minutes. The liquor ratio was 1:50 i.e. 0.5 g/25 ml. After soaping treatment, the specimen was removed, rinsed in cold and hot water respectively. Squeezed and dried at room temperature (Geelani *et al.*, 2015) [8].

In this experiment level of different mordants were optimized. The following mordants were used. The details are tubulated as under:

Table 1: Optimization of different types of mordants

Type of mordants	Concentration (%)
Alum	3
Citric acid	3
Ferrous sulphate	3
Copper sulphate	3
Acetic acid	3
Dye Used	Concentrations
Beetroot (*Beta vulagris*)	10%

The dye percentage was kept constant @ 10%. The samples were analyzed for absorbance at 560 nm, color coordinates, colour strength (K/S) and fastness properties.

Results and Discussion

Table 2: Colour coordinates values of silk fibre dyed with beetroot using different mordants

Mordant type	Concentration (%)	Percent absorption dye (%)	L*	a*	b*	C	h⁰	ΔE	K/S
Copper sulphate	3	52.1	54.575	1.525	9.431	9.554	80.782	42.075	1.528
Ferrous sulphate	3	59.4	58.593	3.262	10.454	10.951	72.641	38.298	1.177
Acetic Acid	3	54.7	61.440	2.786	9.178	9.592	73.085	25.407	0.493
Citric acid	3	53.6	54.285	4.184	11.340	12.087	69.720	32.934	0.805
Alum	3	68.9	52.310	4.199	14.913	13.548	74.287	46.874	1.184

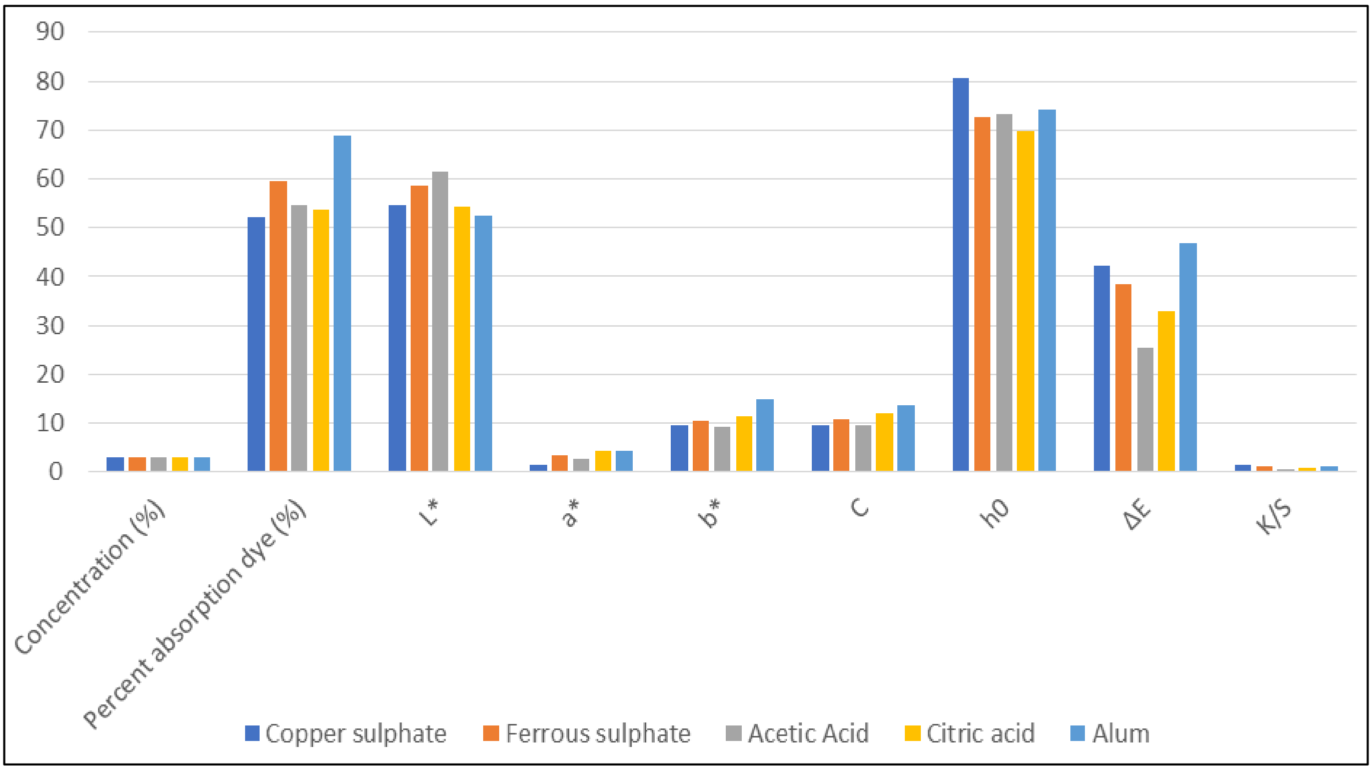

Fig 1: Percent absorption, color coordinates and colour strength of beetroot dyed silk fibre

Percent absorption of beetroot dye (10%) by the silk fibre using different mordants

Silk fibre recorded highest absorption of dye (68.9%) in alum followed by ferrous sulphate, acetic acid, citric acid and copper sulphate with values of 59.4, 54.7, 53.6 and 52.1 respectively. Our results are in agreement with Geelani *et al.*, 2016 [7] who reported that Silk fabric recorded the highest value with *Populus deltoids* adopting simultaneous mordanting; and found the best absorption results of the natural dyes when used in combination with mordants. Since the dye used is a water-soluble dye containing anionic groups would interact ionically with the protonated terminal amino groups of the silk fibres at acidic pH via ion exchange reaction. This ion attraction would increase the dye ability of the fibre. Our results are supported by Alam *et al.*, 2007 [2] who reported that in the absence of mordant (Alum), dye has poor affinity towards silk fibre due to the presence of similar charge group between the fibre and dye while used henna leaves dye and alum mordant on silk. He further reported that the absorption reaches saturation when silk fibre is dyed in the presence of alum as mordant. Our results are supported by Sharma *et al.*, 2005 [21] who reported that the simultaneous method gave good results for hamelia dye using alum, ferrous sulphate and copper sulphate mordants while dyeing silk. He further reported that in case of kilmora dye simultaneous mordanting was found to be good for alum mordant.

Colour coordinates of fibres dyed with beetroot using different mordant

Lowest L*(52.310) was recorded in alum followed by citric acid (54.285), copper sulphate (54.575), ferrous sulphate (58.593), and acetic acid (61.44). Highest values to the tune of 4.199, 4.184, 3.262, 2.786 and 1.525 were recorded for alum, citric acid, ferrous sulphate, acetic acid and copper sulphate for a* respectively. In case of b* values to the tune of 14.913, 11.340, 10.454, 9.431 and 9.178 were recorded for alum, citric acid, ferrous sulphate, copper sulphate, acetic acid respectively. In C* values to the tune of 13.548, 12.087, 10.951, 9.592 and 9.554 were recorded for alum, citric acid, ferrous sulphate, copper sulphate and acetic acid respectively. In case of hº values to the tune of 80.782 74.287, 73.085, 72.641, 69.72, were recorded for copper sulphate, Alum, acetic acid, ferrous sulphate, and citric acid respectively. Total colour change values to the tune of 46.874, 42.075, 38.298, 32.934, and 25.407 were recorded for Alum, copper sulphate, ferrous sulphate, citric acid, and acetic acid respectively. Our results are in concurrent with Narayana swamy *et al.*, 2013 [17] who reported that the decreased L* values of the dyed samples indicate darker shades while used *Psidium guajuva* dye on silk. Our results are supported by Narayanaswamy *et al.*, 2013 [17] who reported that the a* and b* values were positive for all the dyed samples indicating redder and yellower tones while they used *Psidium guajuva* dye on silk. Gias Uddin 2014 reported that while dying silk fabric with onion extract dye significant yellowness of silk fabric was increased using alum mordant over the reference dyed sample. Our results are in concurrent with Swamy 2020 who reported that the hue angle of the dyed samples ranged from 60.65 (Alum) to 75.54 (tannic acid), while the control samples showed 46.74. Narayanaswamy *et al.*, 2013 [17] reported L*, a*, b*, C* and h° to the tune of 46.50, 11.02, 5.78, 57.92 and 59.75 of silk fabric dyed with *Casuarina equisetifolia* bark extract and alum mordant. Geelani. (2014) [9] recorded colour change (ΔE) (42.59) using *Quercus robura* (fruit cups) dye with *Salix alba* (wood extract) mordant on silk fabric, while as lowest colour change (ΔE) 16.99 was recorded in silk fabric using *Tagetes patula* (petals) dye and Salix alba (wood extract) mordant.

Colour strength (K/S) of dyed fibres

The K/S values were observed with the values to the tune of 1.184, 1.528, 1.177, 0.805, and 0.493 for alum, copper sulphate, ferrous sulphate, citric acid, and acetic acid respectively. Jihad 2014 [15] reported that among the different fibre-mordanting systems studied, 3% of aluminum sulphate for subsequent dyeing on silk with 5% extract of *Rumex abisinicuss* jaq and show maximum k/s value. Baishya *et al.* (2012) [4] reported significant changes in the K/S values by using mordants. The higher colour strength of the mordanted fabrics may be due to their ability of forming coordination complexes with dye molecules (Baishya *et al.*, 2012) [4].

Table 3: Colour fastness grades of silk fibre dyed with beetroot using different mordants

Mordant type	Concentrations (%)	Washing		Lightness	Rubbing	
		Fading	staining		Dry	Wet
Citric acid	3	5	4/5	4/5	5	4/5
Ferrous sulphate	3	4/5	4/5	4/5	5	4/5
Acetic Acid	3	5	5	4/5	5	4/5
Alum	3	5	5	5	5	4/5
Copper sulphate	3	4/5	4	5	4/5	4

Excellent (5) grade washing fastness for colour change was recorded in alum, citric acid and acetic acid. Good to Excellent (4/5) colour change grade were recorded in ferrous sulphate and copper sulphate. The washing fastness for colour staining recorded Excellent (5) grade in alum and acetic acid. Good to Excellent (4/5) colour staining grade were recorded in ferrous sulphate and citric acid. Good (4) colour staining grade were recorded in copper sulphate. Excellent (5) grade for colour change in light fastness was recorded in alum and copper sulphate. Good to Excellent (4/5) grade for colour change was in citric acid, ferrous sulphate and acetic acid. Dry fading recorded Excellent (5) grade in citric acid, ferrous sulphate, acetic acid and alum. Good to Excellent (4/5) grade for wet staining were recorded in copper sulphate. Wet staining recorded Good to Excellent (4/5) grade in citric acid, ferrous sulphate, acetic acid and alum. Good (4) grade for wet staining was recorded in copper sulphate. Our results are supported by Geelani *et al*., 2016 [7] who reported lightness fastness recorded good to excellent and excellent grades while used *Populus deltoides* as mordant and *Quercus robur* as dye on natural fibres. Iqbal *et al.,* 2018 reported change in shade of silk fabric dyed with tannic acid gave best (4-5) results while dyed silk with *Beta vulgaris* peel dye. He further reported that change in shade of wool dyed with the presence of mordant like alum, citric acid and tartaric acid also showed good (4) results. Iqbal *et al.,* 2018 reported that colour fastness to water mostly showed rating from (4-5) to 5 of change in shade and stain of silk fabrics dyed with and without acid natural mordants with 2% and 4% beetroot peel dye concentrations. Our results are in agreement with Geelani *et al.*, 2016 [7] who reported rubbing fastness of the dyed fabrics recorded good to excellent-to-excellent grades in both staining and fading in dry and wet conditions while used mordant *Salix alba* with dye *Quercus robur*. Geelani *et al.,* 2016 [7] also reported the rubbing fastness recorded good to excellent to excellent in wool, silk and cotton fabrics while used *Quercus robur* and *Populus detoides* as dye and mordant respectively. Our results are in accordance with Arora and Rastogi, 2011 who reported that the silk exhibits excellent color fastness to rubbing with the rating of 4/5- 5 while used *Arnebia nobilis* dye on different fabrics. Jihad. (2014) [15] reported the washing fastness and rubbing fastness to dry and wet was found in the range of good to excellent while used *Rumex abyssinicus* dye and Alum mordant on silk fabric.

Conclusion

Utilizing natural dyes for colouring fibres in the textile industry is a good alternative to synthetic dyes because they can be harmful to the environment. *Beta vulagris*, a dye extract, showed high dye intake and the ability to produce brilliant hues when combined with 3% alum as a mordant. It is valuable that this combination may offer the silk industry a viable commercial option.

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