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Effect of different mordants, with mordanting methods on physical properties of silk yarn dyed with *Acacia nilotica* pods

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Abstract: The degummed mulberry silk yarn and dried *Acacia* nilotica pods were used for the study. The powdered *Acacia nilotica* (5, 10, 15 and 20g) was soaked overnight in different Material Liquor Ratio (1:20, 1:30 and 1:40) for optimize the dye-concentration and it was extracted by aqueous method. Based on the reflectance and K/S value, 10g dye-concentration, 1:30 Material Liquor Ratio, 30 min extraction-timewere optimised for dye-extraction. The pre treated silk mordanted with different metallic mordants *viz.,* alum (5%, 10%, 15%), stannous (0.5%, 1.0%, 1.5%), Copper and Ferrous (1%, 2%, 3%) in pre, simultaneous and post mordanting methods and dyed in optimized dye concentration in 1:40 Material Liquor Ratio for 30 min. The dyed silk assessed for yarn strength and elongation. Irrespective of mordants, mordant concentration and mordanting methods, pre mordanted dyed silk yarn with alum (15%), stannous (0.5%), copper and ferrous (3%) showed increased K/S value & colour co-ordinates and these mordants were used for mordant combinations. The silk pre mordanted with (15%) alum showed greater strength & elongation than the other samples. The various shades obtained from dyed silkmordanted with different mordants. Thus, natural dyeing of *Acacia nilotica* pods with silk yarn is unique and new venture for the dyers, painters and fashion designers. Rural folk can take up these activities as a enterprise to sustain their livelihood.

Key words: Colour strength, Elongation, Mordants, Mulberry silk, Yarn strength

Introduction

Acacia nilotica is a slow - growing species and is moderately long-lived. The species can withstand extremely dry environments and can also endure floods. The plant is good source for fodder especially leaves and pods feed to the cattle's. Pods are used as a supplement to poultry rations in India. It is considered as a very important economic plant since early times it was used as a source of tannins, gums, timber, fuel, fodder and medicine. Gum is widely used as industrial, food and medicinal purposes. The dark brown heartwood is widely used in constructions, for railway sleepers, mine props, tool handles and carts. They are important trees for lac insects in the Indian subcontinent. Gum collected from the trunk and branches was formerly used in paints and medicines. This is frequently used in calico printing and dyeing as a thickening agent (Abhishek, 2015). The species is also used for medicinal purposes. Bark of *Acacia Nilotica* has been used for treating hemorrhages, colds, diarrhea tuberculosis and leprosy while the roots have been used as an aphrodisiac and the flowers for treating syphilis lesions. It has an inspiring range of medicinal uses with potential anti-oxidant activity (Atif, *et al.,* 2012).

Natural colorants find the application in a wide range of colouration like dyeing and printing of textiles and they produce very uncommon, soothing and soft shades as compared to synthetic dyes and it has anti-allergic property and less harmful to human beings. Environmental issues are becoming more crucial all over the world. Natural dyes due to their eco friendly nature create superior value to the textile substrate with the advent of synthetic dyes stuff in abundance and a wide range of colours of remarkable fastness properties. Natural dyes produce special aesthetic qualities which combined with the ethical significance of a product that is environmentally friendly, gives added value to textile production as craftwork and as an industry (Wanyama, *et al.,* 2010). However, silk also being the natural protein fibre possessed lustrous, translucent, good drapability, crisp hand and pleasing appearance. It has a greater affinity for natural dyes and with good fastness properties and obtain vivid colour shades. The present study is designed following objectives; effect of mordants, mordanting methods and mordant concentration on single yarn strength (kgf) and elongation of single mordanted dyed silk yarn(%) and effect of different mordant combinations on single yarn strength (kgf) and yarn elongation (%) of dyed silk yarn.

Material and methods

The present study was carried out at University of Agricultural Sciences, Dharwad, Department of Textile and Apparel Designing during the year 2014-2016. A fresh *Acacia nilotica* pods (Fig. 1) were collected from local forest of molakalmuru taluk of Chitradurga district, Karnataka and pods were shade dried and crushed into fine powder by traditional pounding technique (Fig. 2). The multivoltine yellow race degummed mulberry silk was procured from Demonstration Cum Training Center (DCTC), Central Silk Board, Rayapur Dharwad for silk dyeing.

The powdered *Acacia nilotica* (5, 10, 15 and 20g) was soaked overnight in different Material Liquor Ratio (1:20, 1:30 and 1:40) for optimize the dye concentration and it was extracted by aqueous method. Optimisation of dyeing

Fig. 1. *Acacia nilotica* pods

conditions are essential to get the desired properties of dye source. The dyeing variables were selected based on the All India Co-ordinated Research Project (AICRP) on natural dyes, UAS, Dharwad.The dyeing variables *viz.,* method of extraction, time of extraction (for example 30, 45, 60 min), dyeing time (30, 45, 60 min) were optimized based on the spectral value at 430 (ë) wavelength of the dye source. Based on the reflectance and K/S value, 10g dye-concentration, 1:30 Material Liquor Ratio (MLR) and 30 min extraction time were optimised for dye extraction.

One natural mordant *viz.,* myrobolan and four metallic salts *viz.,* potash alum (5, 10 and 15%), Copper sulphate and Ferrous sulphate (1, 2 and 3%) and stannous chloride (0.5, 1 and 1.5%) mordanted in pre, simultaneous and post mordanting methods were used for the study.

The degummed silk yarn pre treated with myrobolan (20g owf for 11/2 hour) and dyed with 10% dye concentration (owf) in varied M.L.R (1:20, 1:30, 1:40) and dyeing time(30, 45 and 60 min) intervals. Based on the colour strength (K/S) and colour co-ordinates values one dyeing time (30min) and one M.L.R (1:40) was optimised and used for further dyeing.

The pre treated silk hanks were dyed in open dye bath with optimised 10g dye concentration, 1:40 Material Liquor Ratio and 30 min dyeing time (Fig 3). When the bath reached boiling

Fig 3 Dyeing silk yarn with *Acacia nilotica* dye

Fig. 2. Pounding technique of *Acacia nilotica* pods

temperature the pre treated silk hanks were slowly dipped in dye bath. The silk hank were continuously stirred with the help of steel rods to get an even dye absorption. Based on the mordanting methods, mordants were added to the dye bath. In pre mordanting method silk hanks were pre mordanted prior to dyeing, while in simultaneous and post mordanting mordants were added during and after dyeing. Irrespective of mordants, mordant concentration and mordanting methods, pre-mordanted dyed silk yarn with alum (15%), stannous (0.5%), copper and ferrous (3%) showed increased K/S value & colour co-ordinates and these mordant concentrations were further used for mordant combinations. The dyed silk assessed for single yarn strength and yarn elongation (Fig. 4).

Shades obtained on silk yarn dyed with *Acacia nilotica* pods in different mordants were assessed based on visual and coded by panthone colour system.

Fig. 4. Single yarn strength (kgf) and yarn elongation tested from unistretch

Results and discussion

Effect of mordants, mordanting methods and mordant concentrations on single yarn strength (kgf) of single mordanted dyed silk yarn

Table 1 showes that, the single yarn strength of silk yarn pre mordanted with 1.5 percent stannous chloride (0.20 kgf) showed significantly greater strength as compared to control (0.15 kgf) and other mordanted samples. The silk yarn pre mordanted with potash alum in varied concentrations (5, 10 and 15%) showed greater strength in 10 and 15 per cent (0.16 kgf) compared to silk yarn pre mordanted with 5 per cent (0.14kgf).As the copper sulphate concentration increased, the single yarn strength also increased in pre mordanted dyed silk (0.14 - 0.16kgf). The similar trend was observed in silk yarn pre mordanted with ferrous sulphate in all the concentrations (0.17 and 0.18 kgf). Pre mordanted silk yarn exhibited increased single yarn strength than the control sample. This may be due to the increase in the size of the molecule after using mordants, which penetrates into the fibre core which adds weight in turn, increase the strength of dyed silk yarn.

However, the influence of metal ions on silk varies with the type of mordant used and amount absorbed. The silk has Peptide bonds or salt linkages that have strong inter polymer forces of attraction and therefore contribute to the cohesion of appropriate fibre polymer system due to absorption of dye molecules. This in turn, enhances the tenacity, elastic nature and durability of textile fibre and ultimately textile material. This may be due to deposition and penetration of mordants on the surface of the fibre polymers and fibre core which inturn adds weight and strength to the dyed silk yarn (Saikam *et al.,* 2015).

In the simultaneous mordanting method the greater strength (0.20 kgf) was obtained in silk yarn mordanted with 10 per cent potash alum and 1 per cent copper sulphate than the other mordanted samples. As the mordant concentration increased there was increase in the single yarn strength of silk yarn mordanted with potash alum *i.e.,* 0.13 (5%), 0.16 (10%), 0.20 (15%) and for stannous chloride 0.14 (0.5%), 0.16 (1%), 0.16 (1.5%) respectively.

The similar trend was observed in silk yarn post mordanted with potash alum (0.16/5%, 0.17/10%, 0.18/15%) and stannous chloride (0.15/0.5%, 0.16/1%, 0.17/1.5%). Whereas, as the mordant concentration increased with decreased yarn strength was noticed in simultaneous mordanted silk yarn with copper sulphate (0.20/3%, 0.17/2% and 0.15/1%) and ferrous sulphate (0.18/3%, 0.17/2%, and 0.16/1%). However, strength loss was observed in case silk yarn post mordanted with copper sulphate (0.19/2%, 0.17/ 1%, 0.16/3%) and ferrous sulphate *i.e.,* 0.21(1%), 0.14(2 and 3%) respectively. This may be due to, heat application during dyeing may destroyed fibre polymers and dye particles.

ANOVA explained that, among the mordants, mordanting methods and mordant concentrations only mordants influenced significantly on physical properties of silk yarn mordanted with varied concentrations. However, combined interaction of mordants, mordant concentrations and mordanting methods on single yarn strength of dyed silk was found to be non significant.

Effect of different mordants, mordant concentrations and mordanting methods on elongation of single mordanted dyed silk yarn (%)

Table 2 reveals that, the elongation of silk yarn pre mordanted with 1.5 percent stannous chloride (11.17 %) showed maximum elongation compared to control (9.55) and other mordanted samples.The silk yarn pre mordanted with potash alum in varied concentration (5 to 15%) showed greater elongation in 15 per cent (10.61%) compared to silk yarn pre mordanted with 10 per cent (9.46%) and 5% (8.93%).The copper sulphate pre mordanted dyed silk yarn showed increasing trend in elongation *i.e.,* 10.58 (1%), 11.12 (2%) and 11.23 (3%) respectively, explained that, as the concentration increased per cent elongation also increased.

The yarn elongation percentage was found to be greater in silk yarn pre mordanted with ferrous sulphate in 2 per cent concentration (8.93%) followed by 3 per cent (8.32%) and 1 per cent (8.15%). In the simultaneous mordanting method, the greater elongation (12.43%) was obtained in silk yarn mordanted

Table 1. Effect of different mordants, mordant concentration and mordanting methods on single yarn strength (kgf)

Dye concentration -10g | Dyeing time- 30 min | M.L.R ratio -1:40

Mordanting method	Control	Potash alum			Stannous chloride			Copper sulphate			Ferrous sulphate		
		5%	10%	15%	0.5%	1%	1.5%	1%	2%	3%	1%	2%	3%
Pre-mordanting method	0.15	0.14	0.16	0.16	0.13	0.15	0.20	0.12	0.15	0.17	0.17	0.17	0.18
Simultaneous mordanting method		0.13	0.16	0.20	0.14	0.16	0.16	0.15	0.17	0.20	0.16	0.17	0.18
Post mordanting method		0.16	0.18	0.19	0.15	0.17	0.16	0.17	0.19	0.16	0.21	0.14	0.14

Two factorial CRD table

A	B	C	A x B	B x C	A x C	A x B x C	
S.Em ±	0.011	0.011	0.011	0.005	0.003	0.045	0.015
C.D. @5%	0.031*	0.024 NS	0.024 NS	0.014 NS	0.009 NS	0.126 NS	0.042 NS
CV(%)19.97							

*Significant @ 5% level NS –Non Significant

A - Mordants B -Mordanting methods C -Mordant concentration with copper sulphate (1%) than the other mordanted samples. In case of silk yarn mordanted with potash alum resulted that maximum elongation in 10 per cent concentration (9.72%) followed by 15 per cent (8.76%) and 5 per cent (8.54%). However, silk yarn mordanted with stannous chloride showed maximum elongation in 1.0 per cent concentration (11.45%) followed by 1.5 per cent (10.5%) and 0.5 per cent (7.87%).

The silk yarn mordanted with ferrous sulphate obtained better elongation in 3 per cent (9.18%) followed by 1 per cent (9.27%) and 2 per cent (8.2%).

The silk yarn post mordanted with ferrous sulphate showed maximum elongation in 1 per cent concentration (10.75%) followed by 3 per cent (9.94%) and 2 per cent (9.44%). The similar trend was noticed in silk yarn mordanted with copper sulphate (10.11/1%, 10.47/2% and 10.19/3%), stannous chloride (9.07/0.5%, 10.56/1.0% and 9.91/1.5%) and potash alum (9.20/5%, 9.27/10% and 9.91/15%) respectively.

In general, the increase in elongation of mordanted silk samples may be due to an increase in the crimp because of heat application during dyeing (Durah, 2013). Moreover, during dyeing of protein fibres hydrogen bond as well as vanderwalls forces which anchor the dye molecule to fibre polymer system not only prevent dye stripping but also increase the strength and elongation of dyed yarn (Gohl and Vilensky, 1987).

Only mordants influenced significantly on physical properties of silk yarn mordanted with varied concentration. However, the mordant concentration and mordanting methods had no influence on strength and elongation of single mordanted dyed silk yarn.

The various shades obtained from dyed silk mordanted with alum and copper (light brown to dark olive green), stannous (light brown to dark cream) and ferrous (dark coffee brown to light ash).

Effect of different mordant combinations on single yarn strength (kgf) and elongation(%) of dyed silk yarn

It is learnt from the Table 3 that, the control sample *i.e.*, myrobolan pre treated *Acacia nilotica* dyed silk yarn showed lower yarn strength and elongation than the silk pre-treated with different mordant combinations. Among the silk yarn mordanted with different mordant combinations, single yarn strength was found to be more in potash alum+ stannous chloride (0.32) followed by stannous chloride+ ferrous sulphate (0.28), potash alum+ ferrous sulphate (0.27, potash alum+ copper sulphate (0.25), copper sulphate + stannous

Table 2. Effect of different mordants, mordant concentration and mordanting methods on yarn elongation (%)

Dye concentration -10 g | Dyeing time 30 min | M.L.R ratio -1:40

Mordanting method	Control	Potash alum			Stannous chloride			Copper sulphate			Ferrous sulphate		
		5%	10%	15%	0.5%	1%	1.5%	1%	2%	3%	1%	2%	3%
Pre-mordanting method	9.55	8.93	9.46	10.61	8.96	8.74	11.17	10.58	11.12	11.23	8.15	8.93	8.32
Simultaneous mordanting method		8.54	9.72	8.76	7.87	11.45	10.5	12.43	9.46	10.14	9.27	8.2	9.18
Post mordanting method		9.20	9.27	9.91	9.07	10.56	9.91	10.11	10.47	10.19	10.75	9.44	9.94

Two factorial CRD table

	A	B	C	A X B	B X C	A X C	A X B X C
S.Em ±	0.216	0.167	0.167	0.096	0.058	0.871	0.290
C..D (5%)	0.604*	0.467^{NS}	0.467^{NS}	0.269^{NS}	0.162^{NS}	2.429^{NS}	0.808^{NS}
CV(%)	51.12						

*Significant @ 5% level NS –Non Significant
A - Mordants B - Mordanting methods C -Mordant concentration

Table 3. Effect of different mordant combination on single yarn strength (kgf) and yarn elongation (%) of dyed silk yarn

Mordant combination	Strength yarn strength (kgf)			Yarn elongation (%)		
	Mean	S.Em±	CV%	Mean	S.Em±	CV%
Control	0.15	-	-	9.55	-	-
SC + FS	0.28*	0.020	20.8	12.74*	0.738	14.7
CS+SC	0.24*	0.013	14.7	13.19*	0.468	9.2
PA+SC	0.32*	0.018	17.5	13.48*	0.479	9.3
PA+CS	0.25*	0.017	18.4	11.50*	0.513	10.8
PA+FS	0.27*	0.014	15.0	12.95*	0.574	11.4
CS+FS	0.21*	0.035	43.2	9.80*	1.673	38.6

SC + FS : Stannous chloride + Ferrous sulphate
CS + SC : Copper sulphate + Stannous chloride
PA + SC : Potash alum + Stannous chloride
PA + CS : Potash alum + Copper sulphate
PA + FS : Potash alum + Ferrous sulphate
CS + FS : Copper sulphate + Ferrous sulphate

chloride (0.24) and copper sulphate + ferrous sulphate (0.21) compared to control sample (0.15).However, the elongation *results showed* that, the mean elongation was found to be higher in potash alum + stannous chloride (13.48) than the other mordant combinations *i.e.,* copper sulphate + stannous chloride (13.19), potash alum + ferrous sulphate (12.95), stannous chloride + ferrous sulphate (12.74), potash alum + copper sulphate (11.50) and copper sulphate + ferrous sulphate (9.80) compared to control sample (9.55).This may be due to, potash alum known as double salt and it has one kind of anion (So_4^{2-} ion) balanced by two kinds K^+ and Al^{3+} anchored with silk -NH^2bonds which may strengthen the silk yarn. Further, silk yarn pre mordanted with higher amount of potash alum (15%) and very less amount of stannous chloride gave good additive property may contribute for improvement in strength and elongation of dyed silk yarn (Samanta and Konar, 2010).

In general, silk yarn pre treated with potash alum + stannous chloride obtained greater yarn strength (0.32 kgf) and elongation (13.48 %).The single yarn strength (kgf) and elongation (%) of the pre mordanted dyed silk yarn with different mordant combination showed significantly greater strength and higher elongation than the control. However, the least CV(%) was found in copper sulphate +stannous chloride mordant combinations than the other mordant combinations.

Conclusion

The control sample *i.e.,* myrobolan pre treated *Acacia nilotica* dyed silk yarn showed lower single yarn strength and elongation than the silk yarn pre-treated with different mordant combinations.

The silk yarn pre mordanted with potash alum in varied concentrations (5, 10 and 15%) showed greater strength and elongation in 15 per cent (0.16 kgf and 10.61%) compared to silk yarn pre mordanted with 5 & 10 per cent concentrations.The mordant concentration increased with decreased strength was noticed in silk yarn mordanted (simultaneous & post) with copper sulphate and ferrous sulphate. The greater elongation (12.43%) was noticed in silk yarn mordanted with copper sulphate in the simultaneous and post mordanting methods at 1per cent than the other mordanted samples.The various shades obtained from dyed silk mordanted with alum and copper (light brown to dark olive green), stannous (light brown to dark cream) and ferrous (dark coffee brown to light ash).Silk yarn dyed with combination mordanting methods resulting in to unique and eye catching shades with optimum physical properties.

Finally, it was concluded that, natural dyeing of *Acacia nilotica* pods with silk yarn was unique and new venture for the dyers, painters and fashion designers. Rural folk can take up these activities as a enterprise to sustain their livelihood.

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