

## Effect of various mordants in *Kum* dyed cotton, silk and woollen yarns

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The colour fastness properties, tensile strength and elongation percentage of various yarns such as cotton, wool and silk, naturally dyed with fermented *Kum* leaves [*Strobilanthes cusia* (Nees) Kuntze] using traditional technique of cold and heating process and chemical technique with metallic salts have been studied. The colour fastness in both traditional and chemical technique shows fair to excellent results. Except for some cases in silk and woollen yarns, the tensile strength and elongation percentage of all the dyed yarns are found to be higher as compared to that of the grey yarn. In cotton yarn, both traditional and chemical pre-mordanting techniques show improvement in colourfastness and high tensile strength and elongation percentage as compared to grey yarn.

**Keywords:** *Achyranthes aspera*, Colour fastness, Elongation percentage, *Garcinia pedunculata*, *Strobilanthes cusia* (Nees) Kuntze, Tensile strength

### 1 Introduction

Natural dyes have been part of man's life, since time immemorial. Natural dyes are currently undergoing a revival in craft and academic circles. The widely and commercially used synthetic dyes impart strong colours, but cause carcinogenicity and inhibition of benthic photosynthesis<sup>1</sup>. Despite the fact that the use of natural dyes decreases for over a century, the natural dyes have never been eroded completely and are still being used in diverse parts of the world<sup>2</sup>.

Indians have been considered as forerunners in the art of natural dyeing. The age-old art of dyeing with natural dyes is common in India. Among the Indians, the people of Manipur are well-known for their handlooms and handicrafts. The *Meitei* communities have been using the mature leaves and young shoots of *Strobilanthes cusia* (Nees) Kuntze (*Kum* in Manipuri) for producing a unique blue-black and indigo coloured *Kum* dye. *Kum* dye is used for dyeing *Kum phanek*, a formal dress worn by Meitei women woven on loom. The dyeing of *Kum* is an indigenous dyeing technology that has been passed from generation to generation. The knowledge about the extraction and dyeing methods dates back to the 11<sup>th</sup> century A.D. and still persists in a few valley localities<sup>3,4</sup>. *Kum* is the

most important plant used in the dyeing of clothes by various communities in Manipur<sup>3-10</sup>.

Although indigenous knowledge system has been practicing over the years in the past, the use of natural dyes has been discontinued over generations due to lack of documentation. The present study has been undertaken so as to revive the age-old art of dyeing with natural dyes. In this research, the mature leaves and young shoots of *Kum* are fermented and their effects on the dyeing process variables are study. The colour fastness properties (washing and rubbing) of dyed cotton, silk and woollen fabrics at optimal conditions, using different doses of both natural mordant and chemical mordants are evaluated. The tensile strength and elongation percentage of both the grey yarn and dyed yarn are also measured.

*S. cusia* of the family Acanthaceae is a wild perennial shrub growing to 13-18 cm, leaves are 10-18 cm long, membranous with 6-7 lateral nerves on either half, elliptic, ovate and acute at both ends. Flowers are purple in densely paniced spikes, usually opposite with ovate, deciduous bracts. Calyx segments are linear-spathulate and corollas are 4 cm long and glabrous<sup>11</sup>.

### 2 Materials and Methods

#### 2.1 Materials

Pure cotton, silk (Eri silk) and 100% pure woollen yarns were obtained from Deepak Departmental Store, Imphal, Manipur.

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Laboratory reagents (LR) grade aluminium sulphate, copper sulphate, ferrous sulphate, potassium dichromate, sodium carbonate and non-ionic soap (Monopol soap), obtained from Dealers of Scientific suppliers, were used.

## 2.2 Methods

### 2.2.1 Scouring of Cotton Yarns

Scouring of cotton yarn was done by washing the yarn in a solution containing 0.5g/L sodium carbonate and 1g/L non-ionic detergent (Monopol soap) at 80°C for 30 min, keeping the material-to-liquor ratio at 1:20 (w/v). The scoured cotton yarn was thoroughly washed with tap water and shade dried. The scoured yarn was soaked in clean tap water for 30 min prior to mordanting and dyeing.

### 2.2.2 Degumming and Bleaching of Silk Yarn

The silk yarn was degummed to remove sericin at 90°C for 30 min in an aqueous solution containing 1g/L non-ionic detergent (Monopol soap) and sodium carbonate (1g/L), keeping the material-to-liquor ratio at 1:30 (w/v). Degummed and bleached yarn was again cold washed with clean tap water for 30 min and finally shade dried.

### 2.2.3 Scouring and Bleaching of Woollen Yarn

Scouring and bleaching of woollen yarn was also performed using the same method as in case of cotton yarn, keeping the material-to-liquor ratio at 1:30 (w/v).

### 2.2.4 Preparation of Fermented Kum

The mature leaves and young shoots of *Kum* were collected and put inside an earthen pot. The pot was filled with water at material-to-liquor ratio 1:20 (w/v), kept under sunlight for 7 days and allowed to ferment. When the plant materials got fermented, a thick dark coloured liquid mass was formed. The fermented liquid mass was filtered through a thin muslin cloth and the filtrate was transferred to a new pot and kept for dyeing purpose.

### 2.2.5 Preparation of Kum-sunu (Lime Solution)

*Kum-sunu*, a lime solution prepared from oyster (*Unio* sp.) shells was used for traditional *Kum* dyeing. The oyster shells were sandwiched between dry cow dung cakes, spread over paddy straw bed and burned till the shell became brittle and dark grey in colour. The brittle oyster shells were powdered using mortar and pestle. The powdered shell was sieved through a muslin cloth and stored in a container for further use. For making the lime solution, lime powder was put in

an earthen pot, filled with water at material-to-liquor ratio 1:50 (w/v) and heated along with vigorous stirring until the desired thin lime paste was obtained.

### 2.2.6 Preparation of Alkaline Solution using *Achyranthes aspera* L.

Dry whole plants of *Achyranthes aspera* L. were burnt and ash were collected. The ash was dissolved in water at material-to-liquor ratio 1:50 (w/v) and filtered several times through a bamboo basket until an adequate alkaline solution of pH 10-12 was obtained. The obtained alkaline solution was stored in an earthen pot.

### 2.2.7 Preparation of Acidic Solution of *Garcinia pedunculata*

Fresh fruit of *G. pedunculata* was cut into small pieces, dried in shade and grounded into powdered form. The powdered form was mixed with water at material-to-liquor ratio 1:50 (w/v) and boil for 2 h. The cooled solution was filtered through a muslin cloth and the filtrate was stored in an earthen pot.

### 2.2.8 Mordanting of Cotton, Silk and Woollen Yarns with Metallic Salts

Scoured and bleached cotton and woollen yarns, degummed and bleached silk yarn were further mordanted prior to dyeing using 10% aqueous solution of aluminium sulphate [Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>], 2% aqueous solution of copper sulphate (CuSO<sub>4</sub>), ferrous sulphate (FeSO<sub>4</sub>) and potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) at 100°C (cotton and silk fabrics) and at 80°C (wool fabrics) for 30 min. The samples were then washed in cold water.

### 2.2.9 Dyeing of Pre-mordanted Cotton, Silk and Woollen Yarns

Dyeing was performed at 80°C ± 2°C for 1 h using fixed amount ( M:L 1:20, i.e. 10 g of cotton yarn in 200 mL of dyeing bath; and M:L 1:30 i.e. 10 g each of silk and woollen yarns in 300 mL of dyeing bath) of each fermented extracts in dyeing glass. Dyed samples were extensively washed in hot water containing 1 g/L non-ionic detergent for 30 min and again rinsed with cold water. Finally, the dyed samples were shade dried.

### 2.2.10 Dyeing of Cotton, Silk and Woollen Yarns in Traditional Method

Dyeing in traditional method was carried out at three different ratios of *Kum sunu* : alkaline solution : acidic solution, viz. 5:2:1, 2:3:3 and 3:3:2 in M:L 1:20 for cotton yarn and M:L 1:30 for silk and woollen yarns, maintaining the pH at 11. Yarn to be dyed was put in their respective dyeing glass for 30 min, then taken out, squeezed and air-dried. This process was

repeated for five times until the desired colour or shade was obtained. After the final dyeing, the yarns were washed thoroughly and allowed to shade dried.

### 2.2.11 Evaluation of Colour Fastness, Tensile Strength and Elongation

Colour fastness of the dyed fabric to rubbing (dry and wet) was assessed as per ISO 105/X-X12, 1984 using Crock meter<sup>12</sup>. The colour fastness to washing of the dyed fabric was determined as per ISO 105/C- 1982 methods using a Launder-O-meter following IS-1 wash fastness method<sup>12</sup>. The wash fastness rating was assessed using ISO-105-C01 (extent of staining)<sup>12</sup>.

The tensile strength and elongation percentage of the dyed and grey yarns were measured on an Instron tensile tester.

## 3 Results and Discussion

### 3.1 Effect of Metallic Salts

The evaluation of colour fastness to rubbing and washing, tensile strength and elongation % of all the pre-mordanted samples with aluminium sulphate, copper sulphate, ferrous sulphate and potassium dichromate is presented in Table 1. The colour fastness to rubbing in dry and wet grades and wash fastness in cotton and woollen yarns show medium to good (3-4) results in all the pre-mordanted samples. The colour fastness to rubbing and washing in silk show excellent to good (4-5) results for almost all the treated samples. There is no colour staining to adjacent cotton fabrics (4-5).

In cotton and woollen yarns, for metallic salts pre-mordanting technique the tensile strength is higher as compared to that in grey yarn. In case of silk, the tensile strength of 2% FeSO<sub>4</sub> treated sample is lower than that of grey yarn. Elongation percentage in dyed woollen yarn is higher than in grey yarn but there is less elongation % in cotton yarn. In 10% Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and 2% CuSO<sub>4</sub> treated silk samples the elongation % is higher than in grey yarn but it is lesser in 2% FeSO<sub>4</sub> and 2% K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> treated samples (Table 1).

### 3.2 Effect of Natural Mordants

In traditional hot dyeing technique, wash fastness shows medium to good result (3-4). Silk yarn shows good to excellent result (4-5). Rub fastness in wet grade shows poor result in cotton and woollen yarn but the overall rub fastness in dry grade shows medium to good result (3-4). Dyed cotton and woollen yarn in three different ratio of mordant show higher tensile strength as compared to grey yarn. In dyed silk yarn the value is lesser than in grey yarn for 2:3:3 and 3:3:2 ratio treatment. Elongation % of dyed cotton yarn is higher than that of grey yarn for 5:2:1 and 2:3:3 ratios and it is lesser than in grey silk and woollen yarns (Table 2).

In traditional cold technique, the rub fastness (dry and wet) shows poor to medium (2-3) result in cotton yarn and fair to good (3-4) result in wash fastness. Colour fastness to both rubbing and washing shows fair to good (3-4) result in silk and woollen yarn (Table 3).

Table 1—Fastness grades, tensile strength and elongation % of dyed cotton, silk and woollen yarns using different chemical mordants

Yarn	Mordant	Rub fastness		Wash fastness		Tensile strength, kgf		Elongation, %	
		Dry	Wet	CC	CS	Grey yarn	Dyed yarn	Grey yarn	Dyed yarn
Cotton	10%Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	4	3-4	3	4-5		4.939		6.45
	2% CuSO <sub>4</sub>	3	3	3-4	4-5	3.698	4.913	10.99	7.77
	2%FeSO <sub>4</sub>	3-4	3	4	4-5		4.403		7.67
	2% K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	3-4	3-4	3-4	4-5		4.859		10.81
Silk	10%Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	4-5	4	4	4		3.382		22.74
	2% CuSO <sub>4</sub>	4-5	4	4-5	4-5	2.201	2.738	19.64	22.60
	2%FeSO <sub>4</sub>	4	3	4	4		1.530		15.89
	2% K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	4-5	4	4-5	4-5		2.335		18.40
Wool	10% Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	4	4	4	4		8.134		18.60
	2%CuSO <sub>4</sub>	4	4	4	4	4.939	6.335	15.26	15.26
	2%FeSO <sub>4</sub>	3-4	3	4	4		8.751		21.42
	2%K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	3-4	3	3-4	4		6.899		18.12

CC—Colour change and CS—Staining to adjacent cotton fabric.

Table 2—Fastness grades, tensile strength and elongation % of dyed cotton, silk and woollen yarns dyed in hot condition using both alkaline and acidic mordant w fermented *kum* in different ratio

Yarn	KS:Al:Ac	Rub fastness		Wash fastness		Tensile strength, kgf		Elongation, %	
		Dry	Wet	CC	CS	Grey yarn	Dyed yarn	Grey yarn	Dyed yarn
Cotton	5:2:1	3-4	3	4	4		6.147		11.33
	2:3:3	3	2	4	4	3.698	6.201	10.99	11.03
	3:3:2	3-4	2-3	3-4	4		3.758		7.860
Silk	5:2:1	4	3-4	4	4		3.543		15.78
	2:3:3	4-5	4	4-5	4-5	2.201	2.094	19.64	12.28
	3:3:2	4	3-5	4	4-5		2.147		10.73
Wool	5:2:1	3-4	2-3	3-4	4		5.503		11.37
	2:3:3	3-4	2-3	3-4	4	4.939	5.798	15.26	12.06
	3:3:2	4	3	4	4		4.698		11.16

KS—*Kum sunu*, Al—Alkaline mordant, Ac—Acidic mordant, CC—Colour change and CS—Staining to adjacent cotton fabric.

 Table 3—Fastness grades, tensile strength and elongation % of cotton, silk and woollen yarns dyed in traditional cold method using *Kum sunu*, alkaline mordants, acidic mordant and fermented *Kum* in different ratio

Yarn	KS:Al:Ac	Rub fastness		Wash fastness		Tensile strength, kgf		Elongation, %	
		Dry	Wet	CC	CS	Grey yarn	Dyed yarn	Grey yarn	Dyed yarn
Cotton	5:2:1	2-3	2	3-4	4-5		5.664		10.86
	2:3:3	4	3	4	4-5	3.698	5.530	10.99	11.08
	3:3:2	3-4	3	4	4-5		5.959		9.88
Silk	5:2:1	4	3	4	4		3.490		19.26
	2:3:3	4	3	4-5	4-5	2.201	2.550	19.64	13.84
	3:3:2	4	3-4	4-5	4-5		2.631		16.62
Wool	5:2:1	4	3-4	4-5	4-5		5.181		12.42
	2:3:3	4	3	4	4	1.939	6.040	15.26	14.65
	3:3:2	3-4	3	4	4		8.268		21.13

KS—*Kum sunu*, Al—Alkaline mordant, Ac—Acidic mordant, CC—Colour change and CS—Staining to adjacent cotton fabric.

It can be conferred from Table 3 that as compared to the grey yarn the tensile strength of all the dyed yarns shows greater values. In case of elongation %, the cotton yarn have almost high value, and the silk yarn has lesser elongation break than the grey yarn. Except for 3:3:2 treatment the woollen yarn shows lower value of elongation break compared to that of grey yarn.

Good rub fastness (dry and wet rub fastness) is reported for silk dyed with *Acalypha* and other natural dyes<sup>13-15</sup>. Cutch and ratanjot show moderate to good dry rub fastness but the wet rub fastness is found to be average<sup>16,17</sup>.

Most of the natural dyes are highly soluble in water and therefore, the colour fastness to washing is low. In order to improve its colour fastness property the mordants are necessarily be used<sup>18</sup>. Mordants are metal salts which produce an affinity between the fabric and the dye<sup>19,20</sup>. Most mordants are mineral

salts, the most common being aluminium, iron, copper, tin and chrome. The influences of different mordants are found to play important role in fading of 18 yellow natural dyes<sup>21</sup>. Mordanting technology improves the development of shade and provides a link to colouring substrate for fixing on cloth.

Medium to good rub fastness properties in all these cases indicate that there are almost no superficial unfixed natural dyes left on the fibre surface after soaping and washing. Expectedly this natural dye is penetrated well inside the fibre voids and probably might have been fixed well by co-ordinated complex formation with the mordants and mordanting assistant.

Colour fastness to washing and rubbing for application of selected dye at higher alkaline pH, with or without treatment with dye fixing agents is always found to be better because higher alkali concentration causes better ionization of dye anion

and anion formation (preventing aggregation of dye molecule) to participate in complex formation among the dyes and mordants. It is reported earlier that alkaline pH of the dye bath improves the colour fastness of jute dyed with red sandalwood<sup>22</sup>. There are even changes in thermal transitions of some natural dyes when subjected to heating (equivalent to dye bath temperature or higher). However, both light fastness and wash fastness depend on the nature of pre-treatment (alkaline/acidic) on the fabric before dyeing<sup>23</sup>.

The extension of break i.e. the elongation % for the treated samples is higher than the untreated one due to the fact that the influence of mordanting with zirconium salt leads to produce more elastic wool fibres<sup>24</sup>.

High tensile strength of the yarns helps it to withstand the sewing stress. The combined property of strength and elongation of silk is a measure of toughness of materials which is related to weaving ability<sup>25</sup>. High tensile strength in silk yarn may be due to its proteinaceous nature of the fibre that easily gets bound with metal ions present in mordants. Increase in mechanical strength is due to the application of metal ions that improve fibre resistance. The influence of metal ions on silk varies with the type of mordant used and amount absorbed<sup>26</sup>. However, tenacity value after dyeing is decreased because the interaction of chemicals and heat treatment during pre-treatment and dyeing processes decreases the degree of polymerization of cotton fibre, although the strength of cotton fibre is higher when wet as compared in dry state<sup>27</sup>. Peptide bonds or salt linkages 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<sup>28</sup>.

Mordanted specimen shows an improvement in single yarn strength as compared to undyed non-mordanted yarn. The increase in breaking strength may be due to the increase in the size of dye molecule after using mordants, which penetrates in to the fibre core and might have, in turn, increased the strength<sup>29</sup>.

As the chitosan concentration increases, the breaking strength values of cotton yarns increases, the breaking strength values of cotton yarns increase and elongation-at-break of the yarn decreases. This is due to the binding of fibres in the yarn

by the chitosin, thereby offering better resistance to the axial load<sup>30</sup>.

#### 4 Conclusion

It is found that the fermented *Kum* dye, in regards to colour fastness to washing and rubbing, tensile strength and elongation percentage of cotton, silk and woollen yarn, shows good results which are helpful for textile industries. Good fastness properties exhibited by all the dyed yarns are because of mordant used. Poor fastness property of yarns following the cold traditional dyeing technique may be due to the cumbersome and long dyeing process in traditional technique. Thus, the long cold traditional technique can be replaced by heating process following the traditional technique.

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