

# Enrichment of silk using eco-friendly natural dye extracted from Lichen (*Flavoparmelia caperata*)

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■ **ABSTRACT** : Natural dyes are known for its soft and lustrous neutral colour. Now a day, it also gaining importance due to its eco-friendly nature with human being and environment. India has a rich sources of natural dyes and one of them are lichens. The lichens were traditionally used for dyeing the woollen yarn and fabric but after the invention of synthetic dyes, the use of lichen declines. In the present study among the different sources of Lichen, *Flavoparmelia caperata* was used to dye the silk fabric by standardise the dyeing recipe. Aqueous medium was used for the extraction of the dye. The dye was used for dyeing of degummed silk cloth and treated with four chemical auxiliaries *i.e.* Citric acid, Sodium sulphate, Oxalic acid and Tartaric acid. Study about fastness tests of dyed clothes was undertaken. Large range of shades was obtained because of varying chemical auxiliaries per cent and combination. It was observed that treatment with chemical auxiliaries improved the colourfastness properties of the dyed samples.

■ **KEY WORDS**: Natural dyes, Lichen, *Flavoparmelia caperata*, Chemical auxiliaries, Colourfastness properties

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India has a rich biodiversity and it is not only one of the world's twelve mega diversity countries, but also one of the eight major centres of origin and diversification of domesticated taxa. It has approximately 490,000 plant species of which about 17,500 are angiosperms; more than 400 are domesticated crop species and almost an equal number their wild relatives. Thus, India harbours a wealth of useful germplasm resources and there is no doubt that the plant kingdom is a treasure house of diverse natural products. One such product from nature is the dye (National Bureau of Plant Genetic Resources, 2000). There are different sources of natural dyes. Natural dyes of plant, minerals and

animal sources are fascinating beautiful and sometimes they challenge the wits of researchers and educators. Most of them produce very colourful effects that are so amazing to be hold. Natural colours are beautiful to be hold. Colouring matter extracted from the roots, stems, leaves or barriers and flowers of various plants have various expectations (Vankar, 2007). Discovery of man-made synthetic dyes in the mid-19<sup>th</sup> century triggered a long decline in the large-scale market for natural dyes. In last few decades, use of synthetic dyes is gradually receding due to an increased environmental awareness and harmful effects. Germany was the first to take initiative to put ban on numerous specific azo-dyes for

their manufacturing and applications. Netherlands, India and some other countries also followed the ban (Patel, 2011). Consumers now-a-days are becoming more concerned about environmental issues and hence are demanding for natural product incorporating natural ingredient.

Colour fastness is the resistance of a material to change any of its colour characteristics or extent of transfer of its colorants to adjacent white materials in touch (Samanta and Agarwal, 2009). Generally light fastness, wash fastness, rub fastness and perspiration fastness are considered for textile fabrics.

Australian National Botanical Garden (2014) stated that alichen is a composite organism that emerges from algae or cyanobacteria (or both) living among filaments of a fungus in a mutually beneficial (symbiotic) relationship. The whole combined life form has properties that are very different from properties of its component organisms. There are about 20,000 lichen species described all over the world so far, and India represents 10% (2305) of the lichens known (Singh and Sinha, 2010). In this study among the different sources of Lichen, *Parmotrema nilgherrensis* was used to standardise the dyeing recipe for silk. Four chemical auxiliaries used to study their effect on dye were: Citric acid, Sodium sulphate, Oxalic acid and Tartaric acid.

## ■ RESEARCH METHODS

### Selection of dye :

*Flavoparmelia caperata* was selected for the study. It was purchased from Ramnagar market, Nainital, Uttarakhand at the rate of Rs. 300/kg.

### Selection of fabric :

Mulberry silk fabric was purchased from Central Silk Board, Dehradun, Uttarakhand at a rate of Rs. 350/m.

### Degumming of silk :

Silk fabric degummed by standardised method recommended by Dantyagi (1983). A detergent solution containing 0.5 ml of genteel per 100 ml of water was prepared. It was heated by 50°C. Silk fabric was dipped into this solution and stirred gently for about 30 minutes. It was kneaded, squeezed in the soap solution and then raised under tap water till free from traces of detergent. After that the fabric was partially dried in shade and

ironed when half wet.

### Optimization of dyeing condition :

For optimizing the dye condition series of experiments were conducted for *Flavoparmelia caperata* such as medium of dye extraction, concentration of dye material, dye extraction time, dyeing time etc.

### Selection of medium of dye extraction :

Dye from the *Flavoparmelia caperata* was extracted using three dye solutions. These dye solutions were prepared by using 1.0 g of dye per 100 ml of water in acidic, alkaline and aqueous medium each. Acidic solution was prepared by adding 1 ml of hydrochloric acid in 100 ml of water while the alkaline solution was prepared by adding 1 g of sodium carbonate in 100 ml of water. Aqueous solutions were prepared by 100 ml of water without using any chemical. Boiled at 80°C for 60 minutes. After that, the solutions were filtered, pre soaked silk samples 2g was added to each beaker and dyed for 1 hour. Samples were then rinsed in cold and dried in shade. Each samples were analysed visually for the depth of the shade obtained and based on it extraction media was selected.

### Optimization of concentration of dyes material :

Four dye solutions were prepared by adding 2, 4, 6 and 8 g of dye material in 100 ml of water and boiled at 80°C for 60 minutes. After that, the solutions were filtered, pre soaked silk samples of 2 g were then added to each beaker and dyeing was carried out for 1 hour. Samples were then rinsed in cold water and dried in shade. Each samples were analysed visually for the depth of the shade obtained and based on it best concentration of dye material was selected.

### Optimization of time of extraction of dyes :

Four dye solutions were prepared by extracting the optimum concentration of the dye material in 100ml of water. Solutions were boiled at 80°C for 30, 60, 90 and 120 minutes. After that, the solutions were filtered, pre-soaked silk samples of 2 g were then added to each beaker and dyeing was carried out for 1 hour. Samples were then rinsed in cold water and dried in shade. Each samples were analysed visually for the depth of the shade obtained and based on it dye extraction time was selected.

### Optimization of dyeing time :

Four dye solutions were prepared by extracting the optimum concentration of the dyes material in 100ml of water at optimum extraction time. Two g per soaked silk samples were added to each beaker and dyed for 30, 60, 90 and 120 minutes, respectively. Samples were then rinsed in cold water and dried in shade. Each samples were analysed visually and through per cent absorption for the depth of the shade obtained and based on it dyeing time was selected.

### Optimization of concentration of chemical auxiliaries :

Chemical auxiliaries namely citric acid, sodium sulphate, oxalic acid and tartaric acid with four concentration of each were selected for the study. The concentration used for each chemical auxiliaries were 1, 2, 3 and 4 per cent (o.w.f.). For this, solution of each chemical auxiliaries concentration were prepared separately using optimized dye concentration and extracted at optimized extraction time. Samples were then rinsed in cold water and dried in shade. Each samples were analysed visually for the depth of the shade obtained and based on it concentration of chemical auxiliaries were selected.

### Colourfastness testing :

The best colour yielding samples from each optimising dyeing recipe was selected according to the visual evaluation percentage rating. The dyed samples were subjected to colourfastness testing against light, washing, rubbing and perspiration according to the standard test method, mentioned below.

Colour fastness to light: Test method AATCC-RR 92, 2013.

Colour fastness to washing: Test method IS: 3361-2003.

Colour fastness to rubbing: Test method AATCC-RA 38, 2005.

Colour fastness to perspiration: Test method AATCC-RA 52, 2006.

## RESEARCH FINDINGS AND DISCUSSION

The results obtained from the present investigation as well as relevant discussion have been summarized under following heads :

### Selection of medium of dye extraction :

Showed best results with aqueous medium and are shown in (Plate 1). Thus aqueous medium was selected as extraction medium of dye.

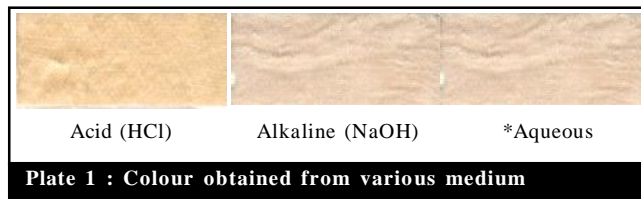






Plate 1 : Colour obtained from various medium





### Optimization of concentration of dye material :

Dye material in a different concentration of 1g, 2g, 3g and 4g were tried for optimizing the concentration of *Flavoparmelia caperata* for dye. The results and dyed samples are reported in Table 1. From the table it was cleared that 2g dye concentration per 100ml of water sample had given the darker shade than the rest of the samples dyed. Hence 2g dye concentration per 100ml of water was selected.

| Table 1: Results for optimization of dye concentration |                       |                   |   |
|--|-----------------------|-------------------|---|
| Dye material   | Dye concentration (g) | Percentage rating | Dyed samples  |
| <i>Flavoparmelia caperata</i>                          | 1                     | 38                |  |
|  | **2                   | *63               |  |
|  | 3                     | 52                |  |
|  | 4                     | 58                |  |

### Optimization of dye extraction time :





The dye was extracted for the different time period i.e. 60, 90, 120 and 150 minutes. The results and samples are reported in Table 2. From the table it was clear that as the timing of dye extraction increases colour shades

| Table 2 : Results for optimization of dye extraction time |                               |                   |   |
|---|-------------------------------|-------------------|---|
| Dye material  | Dye extraction time (minutes) | Percentage rating | Dyed samples  |
| <i>Flavoparmelia caperata</i>                             | **60                          | *63               |  |
|   | 90                            | 27                |  |
|   | 120                           | 35                |  |
|   | 150                           | 23                |  |

of the samples became lighter or less bright. Table also shows that out of four dye extraction time tried, maximum absorption per cent and percentage rating obtained with 60 minutes dye extraction time. 60 minutes dyeing time was selected.

**Optimization of dyeing time:**

Silk samples were dyed for 30, 60, 90 and 120 minutes with *Flavoparmelia caperata*. The dyed samples and results are shown in Table 3. From the table it was clear that the sample dyed at 60 minutes dyeing time gave the best shade.

| Dye material                  | Dyeing time (minutes) | Percentage rating | Dyed samples  |
|-------------------------------|-----------------------|-------------------|---|
| <i>Flavoparmelia caperata</i> | 30                    | 57                |  |
|                               | 60                    | 60                |  |
|                               | 90                    | 76                |  |
|                               | 120                   | 67.5              |  |

**Optimization of concentration of chemical auxiliaries :**





The optimized concentration of citric acid, sodium sulphate, tartaric acid and oxalic acid for *Flavoparmelia*

*caperata* were 2 per cent, 2 per cent, 3 per cent and 4 per cent, respectively. Results and samples shown in Table 4.

**Colour fastness test:**

Final samples were subjected to colourfastness test. Colourfastness to light, washing, crocking and perspiration was evaluated.

Colourfastness to light for the dyed samples with tartaric acid showed excellent colour fastness because it change the colour into darker shade when exposed to light, blank sample and sample dyed with sodium sulphate showed very good colour fastness. Washing fastness of the blank samples showed considerably good washing fastness as compared to samples treated with chemical auxiliaries. Crocking fastness of dyed samples showed good colour fastness for colour change in both dye and wet conditioning. Fastness to perspiration, in case of alkali perspiration blank samples and sample treated with citric acid dyed using *Flavoparmelia caperata* showed, negligible to slightly stained for silk samples and negligible stain for rest of the chemical auxiliary treated samples. Fabric dyed with *Flavoparmelia caperata* and treated with chemical auxiliaries showed good colourfastness property. Results are shown in Table 5. Boruah and Kalita (2016) also studied on natural printing paste for textile application.

| Dye                           | Chem. Auxiliary | Conc. | Percentage Rating | Dyed samples  |
|-------------------------------|-----------------|-------|-------------------|---|
| <i>Flavoparmelia caperata</i> | Citric acid     | 3%    | 76                |  |
|                               | Sodium sulphate | 2%    | 73                |  |
|                               | Oxalic acid     | 3%    | 77                |  |
|                               | Tartaric acid   | 1%    | 76                |  |

| Dye                           | Chemical Auxiliary | Conc. | Colour fastness grades |     |       |         |     |     |     |              |    |        |   |
|-------------------------------|--------------------|-------|------------------------|-----|-------|---------|-----|-----|-----|--------------|----|--------|---|
|                               |                    |       | Washing                |     | Light | Rubbing |     |     |     | Perspiration |    |        |   |
|                               |                    |       | CC                     | CS  |       | Dry     |     | Wet |     | Acidic       |    | Alkali |   |
|                               |                    |       |                        |     | CC    | CS      | CC  | CS  | CC  | CS           | CC | CS     |   |
| <i>Flavoparmelia caperata</i> | Control sample     | -     | 5                      | 4/5 | 3     | 5       | 5   | 4/5 | 4/5 | 5            | 5  | 5      | 5 |
|                               | Citric acid        | 2%    | 2/3                    | 4/5 | 6     | 4/5     | 4/5 | 4/5 | 4/5 | 5            | 5  | 5      | 5 |
|                               | Sodium sulphate    | 2%    | 2                      | 4/5 | 5     | 4/5     | 4/5 | 4/5 | 4/5 | 5            | 5  | 5      | 5 |
|                               | Oxalic acid        | 3%    | 1                      | 4/5 | 4     | 5       | 5   | 4   | 4/5 | 5            | 5  | 5      | 5 |
|                               | Tartaric acid      | 4%    | 2/3                    | 4/5 | 3     | 4/5     | 5   | 4   | 4/5 | 5            | 5  | 4/5    | 5 |

CC= Colour Change; CS= Colour Staining

## Conclusion:

Lichens are abundantly available in Western Ghats and Himalayan region of India that may be explored and used for dyeing of various textiles as these are natural source of dye and cheap and the processing does not involve any harmful chemicals thus making our environment safe. The study was conducted at the laboratory level, hence the optimized dyeing recipe can help the rural women to enhance their skills as well as motivate them to start their business enterprise at home level and cottage level that will generate employment opportunities and can give skill as well as economic empowerment. Further research can also be done on isolation of natural dyes to make easy packaging of these dyes for use in dyeing of various other textile materials.

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