Application of Eco-Friendly Natural Dye on Cotton Obtained from the Flower of Opuntia ficus-indica Using Combination of Mordants

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ABSTRACT
The fastness properties of the flower of Opuntia ficus-indica dyed cotton fabric have been studied using different combinations (1:3, 1:1 and 3:1) of various mordants, such as myrobolan-nickel sulphate, myrobolan-aluminium sulphate, myrobolan-potassium dichromate, myrobolan-ferrous sulphate and myrobolan-stannous chloride. The wash, rub, light and perspiration fastness of the dyed samples have been evaluated. It is found that the flower of Opuntia ficus-indica dye can be successfully used for the dyeing of cotton to obtain a wide range of colours by using various combinations of mordants.

INTRODUCTION
Environmental pollution due to the discharge of dyeing industry effluents is the matter of major concern nowadays. For many years, people have been using all types of natural substances, derived mainly from plants and animals. The use of natural dyes for textile dyeing purposes decreased to a large extent after the discovery of synthetic dyes in 1856. Synthetic dyestuffs produce hazardous by-products (Gulrajani et al. 1992), some of which possess carcinogenic intermediates, and hence a ban has been imposed by Germany and some other European countries on the use of benzidine dyes in textile garments exported into their countries (Anderson 1971 & Kumaresan et al. 2010). Hence, due to the current eco-consciousness, the attention of researchers has been shifted to the use of natural dyes for dyeing textile materials (Kumaresan et al. 2012, Kumaresan et al. 2015). Dyes derived from natural sources have emerged as an important alternative to synthetic dyes.

MATERIALS AND METHODS
In the present work, the flower of Opuntia ficus-indica (Fig. 1) growing in all warm and damp parts of India, have been used. Opuntia ficus-indica (prickly pear) is a species of cactus that has long been a domesticated crop plant grown in agricultural economies throughout arid and semiarid parts of the world. Opuntia is grown primarily as a fruit crop, and also for the vegetable nopales and other uses. The dye was used to dye cotton at optimized dyeing conditions, using a combination of mordants and to evaluate the resultant colourfastness of the dyed samples to washing, rubbing, perspiration and light.

Bleached plain weave cotton fabric, obtained from Gandhi-gram Rural University, Dindigal, was used for the study. AR grade ferrous sulphate, aluminium sulphate, nickel sulphate, potassium dichromate, stannous chloride, and commercial grade acetic acid, common salt and sodium carbonate were used. A natural mordant myrobolan (Terminalia chebula) powder (Kumaresan 2016) was also used for the study. The ethanol extract of the flower of Opuntia ficus-indica was used to get pale brown colour for dyeing fabrics. Depending upon the mordant used, the colour obtained on textiles may give different shades.

A known quantity of flowers of Opuntia Ficus-Indica was dried, powdered and soaked in warm water overnight. The flower of Opuntia ficus-indica extract was obtained by boiling it in the same water. The dye extract was allowed to cool, filtered and used for dyeing. The dyeing was carried out at optimized conditions namely dye extraction time 60 min, material to liquor ratio 1:20, and dyeing time 50 min.

The mordant combinations viz., myrobolan-nickel sulphate, myrobolan-aluminium sulphate, myrobolan-potassium dichromate, myrobolan-ferrous sulphate, and myrobolan-stannous chloride were used in the ratio of 1:3, 1:1 and 3:1. The total amount of two mordants used in each
The dye was used to dye cotton at optimized dyeing conditions, using a combination of mordants and were estimated by using Hitachi-U-2000 UVVIS absorbance spectrometer. Bleached plain weave cotton fabric, obtained from Gandhigram Rural University, Dindigal to evaluate the resultant colour fastness of the dyed samples to washing, rubbing, perspiration and light. Colour fastness to the washing of the dyed fabric samples was determined as per IS: 764-1984 method. AR grade ferrous sulphate, aluminium sulphate, nickel sulphate, potassium dichromate, using a Sasmira launder-O-meter following IS-3 wash fastness method (Samanta et al. 2003, Bains et al. 2003). The wash fastness rating was assessed using greyscale as per ISO-05-A02 (loss of shade depth) and ISO-105-AO3 (extent of staining) and the same was cross-checked by measuring the loss of depth of colour and staining using Macbeth 2020 plus computer-aided colour measurement system attached with relevant software.

Colour fastness to rubbing (dry and wet) was assessed (Senthilkumar et al. 2002) as per IS: 766-1984 method using a manually operated crock meter and greyscale as per ISO-105-AO3 (extent of staining). Colour fastness to exposure to light was determined as per IS: 2454-1984 method. The sample was exposed to UV light in a Shirley MBTF Microsal fade-O-meter (having 500 Watt Philips mercury bulb tungsten filament lamp simulating daylight) along with the eight blue wool standards (BS1006: BOI: 1978).

Colour fastness to perspiration, assessed according to IS: 971-1983 composite specimen, was prepared by placing the test specimen between two adjacent pieces of fabrics of silk and cotton and stitched all among four sides. The sample was soaked in the test solution (acidic/alkaline) separately with MLR 1:50 for 30 minutes at room temperature. The sample was then placed between two glass plates of perspirometer under the load of 4.5 kg. The apparatus was kept in the oven for four hours at 37 ± 2°C. At the end of this period, the specimen was removed and dried in air at a temperature not exceeding 60°C. The test samples were graded for change in colour and staining using grey scales.

Table 1: Fastness grades of the flower of Opuntia ficus-indica dye, dried on cotton at optimum dyeing conditions (wavelength 440 nm, dye extraction time 60min, material to liquor ratio 1:20, dyeing time 50 min) using Mb-NS mordant combination.

| Mordanting Method | Mordant Properties | Light Fastness Grade | Wash Fastness Grades | Rub Fastness Grades | Perspiration Fastness Grades |
|-------------------|--------------------|----------------------|----------------------|---------------------|-----------------------------|
|                   | CC | CS | CC | CS | CC | Dry | Wet | Acidic | Alkaline |
| Pre Mordanting    | 1:3 | 3-4 | 4 | 5 | 5 | 5 | 5 | 4 | 5 | 4 | 5 |
|                   | 1:1 | 3-4 | 4-5 | 5 | 5 | 4-5 | 5 | 4 | 5 | 4-5 | 5 |
|                   | 3:1 | 3-4 | 4 | 5 | 4-5 | 5 | 5 | 5 | 5 | 5 |
| Simultaneous Mordanting | 1:3 | 3-4 | 4-5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
|                   | 1:1 | 3-4 | 4 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
|                   | 3:1 | 3-4 | 4-5 | 5 | 5 | 4-5 | 5 | 4 | 5 | 5 | 5 |
| Post Mordanting   | 1:3 | 3-4 | 4 | 4 | 5 | 5 | 5 | 4 | 5 | 5 | 4-5 |
|                   | 1:1 | 3-4 | 4-5 | 5 | 5 | 4-5 | 5 | 4 | 5 | 4-5 | 4-5 |
|                   | 3:1 | 3-4 | 4 | 5 | 5 | 5 | 5 | 4 | 5 | 5 | 5 |

Mb-NS = Myrobalan-Nickel sulphate, CC = Colour change, CS = Colour staining
RESULTS AND DISCUSSION

Mordant Combination: Myrobolan-Nickel Sulphate

The evaluation of colour fastness to light, washing, rubbing and perspiration of flower of *Opuntia ficus-indica* dyed cotton samples treated with myrobolan-nickel sulphate combination in the aqueous medium is presented in Table 1. All the treated samples subjected to light showed fairly good (3-4) light fastness for all ratio mordant combinations. The washing fastness grades ranged between 4 and 5-4 for all the treated samples and there was no colour staining. The colour change to dry and wet rubbing for all the treated samples was excellent (5).

Mordant Combination: Myrobolan-Aluminium Sulphate

The evaluation of colour fastness to light, washing, rubbing and perspiration of flower of *Opuntia ficus-indica* dyed cotton samples treated with myrobolan-aluminium sulphate combination in the aqueous medium is presented in Table 2. All the treated samples subjected to light showed fairly good (3-4) light fastness for all ratios of mordant combinations. The treated samples for pre mordanting showed fair (3 to 2-3) washing fastness grades, but they ranged from excellent to good (4-5 to 4) for all the treated samples for simultaneous and post mordanting. There was no colour staining. The colour change to dry and wet rubbing for all the treated samples was excellent (5). There was no colour staining ranged from no staining to negligible staining (5 to 4-5) in dry rubbing.

| Mordanting Method     | Mordant Properties | Light Fastness Grade | Wash Fastness Grades | Rub Fastness Grades | Perspiration Fastness Grades |
|-----------------------|--------------------|----------------------|----------------------|---------------------|-----------------------------|
|                       |                    |                      | CC       | CS       | CC       | CS       | CC       | CC       | CC       | CC       |
| Pre Mordanting        | 1:3                | 3-4                  | 4        | 5        | 4        | 5        | 4-5      | 4        | 5        | 4        |
|                       | 1:1                | 3                    | 5        | 5        | 4        | 4-5      | 4-5      | 4        | 5        | 4-5      |
|                       | 3:1                | 4                    | 4        | 5        | 4        | 5        | 5        | 5        | 5        | 4-5      |
| Simultaneous Mordanting | 1:3             | 4                    | 5        | 4-5      | 5        | 5        | 5        | 5        | 5        | 4-5      |
|                       | 1:1                | 4                    | 5        | 4-5      | 5        | 5        | 5        | 5        | 5        | 5        |
|                       | 3:1                | 4                    | 4-5      | 5        | 5        | 4-5      | 4        | 5        | 5        | 5        |
| Post Mordanting       | 1:3                | 3-4                  | 4        | 4        | 5        | 5        | 4        | 4        | 4-5      | 4        |
|                       | 1:1                | 3-4                  | 4        | 4        | 4-5      | 5        | 4        | 4        | 4-5      | 4        |
|                       | 3:1                | 3-4                  | 4        | 4        | 5        | 5        | 4        | 4        | 4-5      | 5        |

Mb-AS = Myrobolan-Aluminium sulphate, CC = Colour change, CS = Colour staining
Table 3: Fastness grades of the flower of *Opuntia ficus-indica* dye, dyed on cotton at optimum dyeing conditions (wavelength 440 nm, dye extraction time 60min, material to liquor ratio 1:20, dyeing time 50 min.) using Mb-PD mordant combination.

| Mordanting Method | Mordant Properties | Light Fastness Grade | Wash Fastness | Rub Fastness | Perspiration Fastness |
|-------------------|--------------------|----------------------|----------------|--------------|------------------------|
|                   |                    |                      | Grades         | Grades        | Acidic | Alkaline |
|                   |                    |                      | CC | CS | Dry | Wet | CC | CS | CC | CC |
| Pre Mordanting    | 1:3                | 3-4                  | 4 | 4 | 3 | 4 | 3 | 4 | 4 | 4 | 4 | 4 |
|                   | 1:1                | 3-4                  | 4-5 | 4 | 3 | 4-5 | 3 | 4 | 4 | 4 | 4 | 4 |
|                   | 3:1                | 3-4                  | 4 | 4 | 3 | 4 | 3 | 4-5 | 3-4 | 3-4 | 3-4 | 3-4 |
| Simultaneous      | 1:3                | 3-4                  | 3-4 | 4 | 3-4 | 4 | 4 | 3-4 | 3-4 | 3-4 | 3-4 | 3-4 |
| Mordanting        | 1:1                | 3-4                  | 3-4 | 4 | 3-4 | 4 | 4 | 3-4 | 3-4 | 3-4 | 4 | 4 |
|                   | 3:1                | 3-4                  | 3-4 | 4 | 3-4 | 4 | 4 | 3-4 | 3-4 | 3-4 | 3-4 | 4 |
| Post Mordanting   | 1:3                | 3-4                  | 4 | 4 | 4 | 4 | 4 | 4 | 3 | 3-4 | 3-4 |
|                   | 1:1                | 3-4                  | 4-5 | 3 | 3 | 3 | 4 | 4 | 3 | 3 |
|                   | 3:1                | 3-4                  | 4 | 3 | 3 | 3 | 4 | 4 | 4 | 3-4 | 3-4 |

Mb-PD = Myrobolan-Potassium dichromate, CC = Colour change, CS = Colour staining

Table 4: Fastness grades of the flower of *Opuntia ficus-indica* dye, dyed on cotton at optimum dyeing conditions (wavelength 440 nm, dye extraction time 60min, material to liquor ratio 1:20, dyeing time 50 min.) using Mb:FS mordant combination.

| Mordanting Method | Mordant Properties | Light Fastness Grade | Wash Fastness | Rub Fastness | Perspiration Fastness |
|-------------------|--------------------|----------------------|----------------|--------------|------------------------|
|                   |                    |                      | Grades         | Grades        | Acidic | Alkaline |
|                   |                    |                      | CC | CS | Dry | Wet | CC | CS | CC | CC |
| Pre Mordanting    | 1:3                | 3-4                  | 4 | 4 | 4-5 | 5 | 5 | 4 | 4-5 | 4 | 4-5 |
|                   | 1:1                | 3-4                  | 4-5 | 4 | 4-5 | 5 | 5 | 4 | 4-5 | 4-5 | 4-5 |
|                   | 3:1                | 3-4                  | 4 | 4 | 4-5 | 5 | 5 | 4 | 4-5 | 5 | 4-5 |
| Simultaneous      | 1:3                | 3-4                  | 3-4 | 4 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Mordanting        | 1:1                | 3-4                  | 3-4 | 4 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
|                   | 3:1                | 3-4                  | 3-4 | 4 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Post Mordanting   | 1:3                | 3-4                  | 4 | 4 | 5 | 4-5 | 4-5 | 4 | 4 | 5 | 4-5 |
|                   | 1:1                | 3-4                  | 4-5 | 3 | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 |
|                   | 3:1                | 3-4                  | 4 | 3 | 4-5 | 4-5 | 4 | 4 | 4 | 4 |

Mb-FS: Myrobolan-Ferrous sulphate, CC = Colour change, CS = Colour staining

Table 5: Fastness grades of the flower of *Opuntia ficus-indica* dye, dyed on cotton at optimum dyeing conditions (wavelength 440 nm, dye extraction time 60min, material to liquor ratio 1:20, dyeing time 50 min.) using Mb-SC mordant combination.

| Mordanting Method | Mordant Properties | Light Fastness Grade | Wash Fastness | Rub Fastness | Perspiration Fastness |
|-------------------|--------------------|----------------------|----------------|--------------|------------------------|
|                   |                    |                      | Grades         | Grades        | Acidic | Alkaline |
|                   |                    |                      | CC | CS | Dry | Wet | CC | CS | CC | CC |
| Pre Mordanting    | 1:3                | 3-4                  | 4 | 4 | 3-4 | 4 | 3-4 | 4 | 3-4 | 3-4 | 4 |
|                   | 1:1                | 3-4                  | 4-5 | 4 | 3-4 | 4 | 4 | 4 | 4 | 4 | 4 |
|                   | 3:1                | 3-4                  | 4 | 4 | 4 | 3-4 | 4 | 4 | 4-5 | 3-4 | 3-4 |
| Simultaneous      | 1:3                | 3-4                  | 4-5 | 4 | 4 | 4 | 4 | 4 | 3-4 | 3-4 | 3-4 |
| Mordanting        | 1:1                | 3-4                  | 4-5 | 4 | 4 | 4 | 4 | 4 | 3-4 | 3-4 | 4 |
|                   | 3:1                | 3-4                  | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 |
| Post Mordanting   | 1:3                | 3-4                  | 4-5 | 4 | 3-4 | 3-4 | 3-4 | 4 | 4 | 4 | 4 |
|                   | 1:1                | 3-4                  | 4-5 | 4 | 3-4 | 3-4 | 3-4 | 4 | 4 | 4 | 4 |
|                   | 3:1                | 3-4                  | 4 | 4 | 3-4 | 3-4 | 3-4 | 4 | 4 | 4 | 4 |

Mb-SC = Myrobolan-Stannous chloride, CC = Colour change, CS = Colour staining
combinations. The washing fastness grades ranged between excellent to good (4-5 to 3-4) for all the treated samples and there was no colour staining. The colour change to dry and wet rubbing for all the treated samples was excellent (5). The colour staining ranged from negligible to slight staining (4-5) in both dry and wet rubbing.

**CONCLUSION**

It was found from the study that flower of *Opuntia ficus-indica* dye can be successfully used for dyeing cotton to obtain a wide range of soft, pastel and light colours by using a combination of mordants. With regards to colour fastness, test samples exhibited excellent fastness to washing (except for pre-mordanting using myrobolan-potassium dichromate combination); excellent fastness to rubbing (except for pre-mordanting using myrobolan-potassium dichromate combination); and good to excellent fastness to perspiration in both acidic and alkaline media and fairly good fastness to light.

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