Improving the Colour Fastness of the Selected Natural Dyes on Cotton

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Abstract: This paper reports the improving the colourfastness of the natural dye with dye fixing agents, extraction of the colourants from natural sources; effects of different mordants and mordanting methods; selection of fixing agents; dyeing variables; post-treatment process; development of newer shades with post treatment and analysis of colour improvement parameters with fixing agents for cotton dyed with natural dye; assessed colour improvement with colourfastness test.

Key words: Dye fixing agents, colourfastness, shade variations with dye fixing agents, natural dye.

I. Introduction:
Natural dyes are considered to be very good for fair colour experimentation, quality, excellent, for their endurance and soft lustrous colouring. Even after a long period they retain great beauty and charm. They do not create any pollution problems as they are applied with sample chemical reactions. The wide varieties of exotic shades produced by natural dyes are parallel with the synthetic range and hence can replace them with ease. At present, meager information in this aspect is available. Natural dyes are being considered as a more environmentally friendly substitute for synthetic dyes but these have few draw backs low colourfastness property is one. But no study reported on improving the colourfastness properties of natural dyes with dye fixing agents. So in this article results were shown the improvement of colourfastness of natural dyes with dye fixing agents.

II. Materials And Methods:
1.1. Selection of materials:
1.1.1. Selection of dye source:
Annato, a potential orange dye source (known as zatropha in Telugu and Tamil or lotpan in Hindi) was used for this study. As per the available literature annatto is known as the direct natural dye which is fugitive and thus exhibit limited fastness properties. Hence, it was selected to see the effect of the fixing agent in improving the colorfastness.

1.1.2. Selection of fabric:
Light weight cotton fabric woven in plain weave was selected

1.1.3. Selection of mordants:
The three eco-friendly mordants, namely alum, stannous chloride and ferrous sulphate.

1.1.4. Selection of eco-friendly fixing agents:
Fixing agents such as vinegar, alum, ammonia, lime juice and calcium chloride were selected, as they eco-friendly and easily available.

III. Preparatory Processes For Fabrics:
1.2. Cotton scouring:
The cotton fabric contains dust, oil and other impurities that interfere with the absorption of the dye. Hence, cotton was scoured using mild alkali and a detergent.

1.3. Pre treatment of cotton with myrobalan:
Pre-treatment was required for cotton fabric as it had no affinity towards natural dyes. Myrobalan fruit was selected for giving pre-treatment to cotton as the tannin content present in it, aids in enhancement of dye uptake and fixing of dye on the fabric.
IV. Optimization Of Dye Extraction Methods:
Dye extraction methods such as aqueous, alkaline and acidic were tried for extraction of dye from the selected sources. The alkaline method was found suitable for extraction of the natural dye source from annato seeds.

1.4. Selection of suitable wave length:
A wave length of 468 nm was found to give maximum optical density for annato seeds.

1.5. Optimization of dye extraction time:
To find out the optimum time for extraction of dye, the selected dye material was added to each of the three beakers containing 100 ml of water. The temperature for boiling was raised 95°C. The optical density of the dye liquor was noted after 15, 30, 45 and 60 minutes of boiling.

V. Optimization Of Mordanting Procedures:
1.6. Selection of mordanting methods:
There are three methods of mordanting methods of mordanting viz., pre-mordanting, simultaneous and post mordanting. Among the three methods, pre-mordanting method was suitable for many natural dye sources especially for the selected dyes with all mordants (Devi et.al, .2002). Hence; pre-mordanting method was selected for improvement of colourfastness of natural dyes on cotton fabric with fixing agents.

1.7. Optimization of mordanting time:
To optimize the time for mordanting, pre-mordanting method was selected one gram of sample was placed in each of the three beakers containing 60 ml of alum solution. The samples were mordanted at 60 °C for 30, 45 and 60 minutes respectively in water bath. The mordanted samples were then dyed in 2 per cent annato seeds dye liquor for 30 minutes. The optical density of the dye liquor was recorded before and after dyeing. The dyed simples were dried under shade. Based on the percentage of dye uptake and visual appearance of the three samples, one sample was selected and the corresponding mordanting time was optimized.

1.8. Optimization of mordant concentrations:
To optimize the concentration of mordants, six concentrations of each mordant were tried with annato seeds. For alum 5, 10,15,20,25 and 30 per cent solutions were prepared. In case of ferrous sulphate and stannous chloride 1, 2,3,4,5 and 6 per cent solutions were prepared separately. Pre-mordanting method was used to optimize the concentration of mordants. These mordanted samples dyed in 4 per cent annato dye. The optical density values were recorded before and after dyeing and percent absorption was calculated. Based on the absorption and visual appearance, three concentrations of each mordant were selected.

VI. Optimization Of Dyeing Methods:
1.9. Dye uptake:
The dye uptake was estimated by taking difference between the concentration of dye liquor before and after dyeing. It was proportional to the dye up taken up by the fabric.

1.10. Optimization of dye material concentration:
To optimize dye concentration, separate containers with 100 ml of water in each were taken and the dye material 1g, 2g, 3g, 4g, 5g, 6g, 7g, 8g,9g, and 10g were placed in the container and boiled at 95°C. Fabric weighing 2g was then placed in the dye liquor and dyed for 30 to 45 minutes. The optical density values before and after dyeing were noted and the percent absorption was calculated. The percent dye absorption of the fabric was estimated by using the formula.

\[
\text{Per cent dye absorption} = \frac{\text{Optical density of the dye liquor before dyeing} - \text{Optical density of the dye liquor after dyeing}}{\text{Optical density of the dye liquor before dyeing}} \times 100
\]

1.11. Optimization of dyeing time:
The cotton fabric to be dyed was weighed. The extracted dye liquor was taken as per the material to liquor ratio of 1:50. The optical density of the liquor before dyeing was recorded. The pre-mordanted fabrics were then dyed at 95°C for 30, 45 and 60 minutes.
were placed in separate dye liquors and dyed for 30, 45 and 60 minutes respectively. The fabric was removed and the optical density of the dye liquor after dyeing was noted.

1.12. Post-treatment with fixing agents:
This is a post-treatment given to dyed fabrics to aid of fixing agents such as, alum, ammonia, lime juice and calcium chloride were selected for the treatment. These fixing agents were selected, as they are common fixing agents used for dyeing fabrics. As per pre Dedhia (1998) first 5 per cent solutions of each of the fixing agents were prepared 5 per cent of fixing agents produced noticeable changes in the dyed samples. Hence, 5 per cent fixing agent was selected. Later, the dyed fabric was placed in the solution for 30 minutes. Finally the fabric was removed, rinsed in warm soap solution and dried.

VII. Soaping-Off:
The dyed samples were washed in 2 per cent luke warm detergent solution to remove the loose dye on the fabric and then rinsed thoroughly in water and dried.

VIII. Evaluation Of Colourfastness Of Test Fabrics:
Colourfastness of any textile product is of considerable importance to the consumer as it directly affects the serviceability of the fabric. One colour fastness test was carried out on cotton fabric to evaluate the colours obtained from annato and also assess improvement in colour of the fabric treated with five fixing agents. The most common serviceable conditions such as the following was selected for evaluation of the colourfastness of fabrics.

- Colourfastness to sunlight
  The following standard procedures laid down by Bureau of Indian Standard Tests series IS-768-1956 for colour change and ARE 769-1956 for staining, using geometric grey scale.

IX. Atmospheric Conditions For Testing:
Prior to samples were conditioned as per bureau of Indian standards IS 6359-1977. The test specimens were kept in the atmospheric conditions 65 ± 2 per cent, relative humidity 27 ± 20 temperature for 24 hours before testing.

X. Statistical Analysis:
The colourfastness grades of the two colourfastness tests for each sample were judged by five respondents. The data was analyzed and one mean was assigned and tabulated.

XI. Results:
Natural dyes have emerged as prime colorants for textiles globally. The scientists in related fields are developing the technologies for extraction and dyeing with natural dyes. Very few studies were made to improve the colourfastness of these natural dye sources. Therefore, an attempt has been made in this study to improve the colourfastness of natural dye source i.e. Annato seeds on cotton, by using five fixing agents. The results of the study are presented under the following sub headings.

- Optimization of dyeing variables
- Colourfastness tests of annato dye

XII. Optimisation Of Dyeing Variables:
1.13. Optimization of method of dye extraction: The dye was extracted from natural sources using alkaline medium as it was found suitable for annato.

1.14. Optimization of alkali concentration: the optimum concentration of alkali required to produce high absorption of the dye pigment in cotton. An amount of 1g of due /100ml of water was found to give good shades as well as high absorption incase of annato seeds.

1.15. Optimization of dye extraction and dyeing time:
After boiling the dye material for 60 min. it was found to extract reasonably good amount of dye in both the sources selected. Around 45 min. dyeing time was noticed to produce good colour depth with both the source on cotton.
1.16. **Optimization of dye material concentration:**
The suitable concentration of dye material was found to be 3 g/100ml of water incase of annato.

1.17. **Optimization of mordant concentration:** mordant concentrations of 5, 10 and 15 for alum, 1, 2 and 3 for stannous chloride and for ferrous sulphate were selected based on the dye absorption and depth of the shade.

1.18. **Optimization myrobalan concentration:** it was found that the absorption of dye increased and reached maximum at 20 per cent concentration of myrobalan and decreased with increase in concentration there after. Hence, 20 per cent myrobalan concentration was selected.

XIII. **Shade Variations In Annato Dyed Cotton Due To Post-Treatment:**
After mordanting with eco-friendly mordants, cotton samples were dyed in annatto dye as per the optimized conditions to improve the colourfastness of shades developed. These dyed samples were post-treated with five fixing agents such as vinegar, alum, ammonia, lime juice and calcium chloride.

The effect of vinegar on the depth of the orange shades obtained by mordanting with alum, stannous chloride and ferrous sulphate was assessed. It was observed that alum and stannous chloride mordanted samples became deeper in orange shade than control. Ferrous sulphate mordanted cotton showed even bright brownish shades after treatment with vinegar. The depth of shade increased with increase in mordant concentration.

Alum post-treated samples showed dark orange shades. Alum post mordanted samples exhibited dark orange shade with slight reddish tinge. This darkened with the increase in mordanted concentration. Stannous chloride mordanted samples showed bright orange shade orange shade with yellowish tinge, after treatment with 5 per cent solution of alum. As the per cent of mordant increased, increase in depth of shade was noticed. Ferrous sulphate mordanted samples gained dark brown shade with a tint of yellow after treating with 5 per cent alum solution.

Ammonia which is considered as fixing agent was used for post-treatment on cotton mordanted with alum, stannous chloride and ferrous sulphate to fix annatto natural dye. Alum mordanted samples showed light orange shades than control. Ten 10 per cent and 15 per cent alum mordanted samples produced bright shades than 5 per cent alum mordanted samples. No colour change was observed in stannous chloride sample while ferrous sulphate mordanted samples produced greenish tinge along with natural brown shade. The increased depth in green shade was evident in the samples with increase in concentration of the mordant.

Lime juice is regarded as freshening agent to restore the original brightness of the shades in home laudering. Post-treatment with lime juice had brightened and darkened the orange shade of annatto on cotton. Alum mordanted samples displayed dark orange shades with reddish tinge. When mordant concentration was increased, the depth of the shade was also increased. Slight reddish tinge was added to bright brown shade obtained on ferrous sulphate mordanted samples treated with lime juice.

Alum mordanted samples post-treated with calcium chloride showed no colour change compared to control. In stannous chloride mordanted samples, slight yellowish tinge was observed. Ferrous sulphate mordanted samples became reddish brown following the treatment.

XIV. **Evaluation Of Colourfastness Tests:**

1.19. **Sunlight fastness of Annato dye on cotton:**

<table>
<thead>
<tr>
<th>Dye percentage: 3%</th>
<th>Mordants: Alum, stannous chloride, ferrous sulphate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extraction medium: alkaline alkali</td>
<td>conc: 1g/100ml.</td>
</tr>
<tr>
<td>Dye Extraction Time: 60 min.</td>
<td>Mordanting time: 30 min.</td>
</tr>
<tr>
<td>Dyeing time: 45 min.</td>
<td></td>
</tr>
</tbody>
</table>

**TABLE: SUNLIGHT FASTNESS PROPERTIES OF ANNATO (Bixa orellana) DYE ON COTTON**

<table>
<thead>
<tr>
<th>Mordant</th>
<th>Mordant conc. G/100g of fabric</th>
<th>Fastness Grades</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>control</td>
<td>T1</td>
</tr>
<tr>
<td>Alum</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>3</td>
</tr>
<tr>
<td>Stannous chloride</td>
<td>1</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>Ferrous sulphate</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>4</td>
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</tbody>
</table>

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Note: vinegar (CH$_3$COOH), T2-Alum (AlK(SO$_4$)$_2$), T3-ammonia (NH$_3$), T4- Lime juice, T5- calcium chloride (CaCl$_2$).

The sunlight fastness of annato dye on cotton mordanted with eco-friendly mordants and treated with various fixing agents. Fair to very fair fastness to sunlight was observed in control samples.

Post-treatment with vinegar had improved the sunlight fastness in case of alum and ferrous sulphate mordanted samples. Good and very good sunlight fastness was observed respectively in these mordanted cottons. Stannous chloride mordanted samples, however, did not show any improvement. Alum post-treated cottons showed mixed results registering lower and improved sunlight fastness compared to control. Alum pre-mordanted and post treated cottons had lowered sunlight fastness, graded as poor to fair. Stannous chloride and ferrous sulphate samples showed improvement in sunlight fastness from goods to very good. The fastness grades were lowered with the increase in the mordant concentration. Fastness was observed respectively in these mordanted cottons. Stannous chloride mordanted samples, however, did not show any improvement.

Post treatment with ammonia showed improvement in sunlight fastness both in alum and ferrous sulphate mordanted cottons. The fastness increased with increase in mordant concentration. Ferrous sulphate mordanted cottons showed very good to excellent sunlight fastness. Slight decrease in sunlight fastness was observed in case of 1% stannous chloride mordanted cottons. It is to be noted that post treatment with ammonia had decreased the depth of orange shades obtained with various mordants but, had contributed in improving sunlight fastness.

Treatment with lime juice had also registered improvement in sunlight fastness over the control. Very fair to good fastness was observed in all mordanted samples irrespective of the mordant used. This was one of the treatments; which had improved the sunlight fastness of the stannous chloride pre-mordanted samples besides, brightening the shades.

Post treatment with calcium chloride did not show any improvement in sunlight fastness of alum pre-mordanted cottons over control. Infact, the fastness was lowered by degree in case of stannous chloride mordanted samples.
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However, ferrous sulphate mordanted cottons had registered good fastness and showed improvement over control.

XV. Conclusion:

Among the mordanted and post-treated cottons, vinegar post-treated samples showed increased depth of the shade on cottons mordanted with alum, stannous chloride and ferrous sulphate, besides providing leveled shades over control. The sunlight fastness of alum and ferrous sulphate mordanted cottons improved and graded as good to very good.

The alum post-treated samples produced better shades than control. In case of alum and ferrous sulphate pre-mordanted cottons and increased level dyeing was observed in all mordanted samples. Stannous chloride and ferrous sulphate samples showed improvement in all sunlight fastness from good to very good fastness.

Post-treatment with ammonia did not register any improvement. However improvement in sunlight fastness both in alum and ferrous sulphate mordanted cottons observed over control.

The lime juice post-treated samples exhibited increase in depth of the shade and contributed for level dyeing in all mordanted samples. Improvement in sunlight fastness was observed in all mordanted samples. Improvement in sunlight fastness was observed in all mordanted cottons over control.

Calcium chloride post-treated cottons did not register any change in shade over control. Treatment did not help in improving the sunlight fastness of alum post-mordanted cottons over the control. Ferrous sulphate mordanted cottons had registered good sunlight fastness and showed improvement over control.

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