EXTRACTION OF NATURAL DYE 
POMEGRANATE RIND AND ITS FASTNESS PROPERTIES

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Abstract - Nowadays, every sector of Industry is turning towards an eco-friendly environment. In this sense in the Textile sector, the application of Natural Dyes is increasing due to awareness of ecology, environment and pollution control. This paper explains the Dyeing of the Cotton fabric using Natural Dye stuff extracted from the Pomegranate peel “Punica granatum”. There are several processes involved in the dyeing of Fabric using Natural Dye. First, the Dye extraction using from pomegranate using Aqueous Extraction method. The Mordants used were ferrous sulphate, Copper sulphate and Oxalic acid. Dyeing with Mordants was carried out with Pre Mordanting and Post Mordanting. Study of Fastness of Dyed fabrics are undergone.

KEYWORDS: Natural Dye, Pomegranate Peel Dye Extraction, Mordanting, Fastness.

1. INTRODUCTION

Dye is an aesthetic substance that is used to colour the fibre and fabrics that we use in our day-to-day life. Normally dyes are classified into natural and manmade dyes (i.e. synthetic dyes). The synthetic dyes are mainly used since 1856 to dye the fiber and fabric materials, due to its superior properties than the naturally extracted substances. The synthetic dyes are economical price and their excellent colour fastness properties. As these dyes are the by-products of the crude oil they are highly toxic and can cause inhibition of benthic photosynthesis and are highly carcinogenic. But the natural dyes are anti-allergic, nontoxic and easily biodegradable and also possess deodorizing properties. And these dyes are bio compatible and have a green approach and ecofriendly in nature so they do not affect the environment. These natural dyes are used for colouring the fabric material and other substances since the Bronze Age. In recent years their uses are extended to antimicrobial finishing of textiles, UV protective clothing, food colorations and pharmaceuticals etc. As they do not require any strong acids and alkalis for their application and production their demand is continuously increasing. The term "Natural dye" includes all the dyes which are derived from natural sources like plants, minerals and animals. But the dye content and colour yield of natural dye is comparatively lower than synthetic dyes.

Natural dyes also have various disadvantages in their properties. As already mentioned they do have colour fastness problems such as colour yield, reproducibility results, dying procedures are different and difficult and fastness properties. So the dyeing industries use mordants with natural dyes to make their affinity to textile materials and to produce different dye shades with various levels of colour fastness. Mordants are metallic or mineral salts when added to the natural dye bath either it intensifies the dye or changes the colour. They also play a large role in making the resultant shade to have prominent light and wash fastness. The dyes do not directly interact with the materials they are intended to colour. They are substantive and require a mordant to fix with the fabric. A mordant is an element which aids the chemical reaction that takes place between the dye and fibre, so that the dye is absorbed. The containers used for dyeing should be non-reactive, materials like stainless steel, enamel etc. can be used. Brass, iron materials should not be used as they do their own Mordanting.
2. MATERIAL AND METHODS

2.1 Materials

Raw material: Pomegranates

Pomegranate is a fruit from shrubs which contains many beneficial facts other than juices, meal garnishes, cocktails etc. In health aspects, it contains 3 times more antioxidants compared to Green Tea. Pomegranate vibrant red colour is due to the presence of polyphenols. Pomegranate is used for variety of purposes but its peel is almost used only for beauty aspects. The Peels can be used as Natural dye in Textile Dyeing aspect. Granatonine is the chemical compound responsible for colour in Dye which in alkaloid form N-methyl Granatonine

Scientific classification

Family: Lythraceae
Genus: Punica
Species: Punica Granatum
Kingdom: Plantae

Composition of Pomegranate Peel

<table>
<thead>
<tr>
<th>Components (g/100g Dry peel solids)</th>
<th>Fresh peel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>278.8(1.5)*</td>
</tr>
<tr>
<td>Crude Fiber</td>
<td>21.6(6.1)</td>
</tr>
<tr>
<td>Ash</td>
<td>3.3(0.3)</td>
</tr>
<tr>
<td>Protein</td>
<td>8.8(0.3)</td>
</tr>
<tr>
<td>Fat</td>
<td>1.3(0.5)</td>
</tr>
<tr>
<td>Total Carbohydrate</td>
<td>86.4</td>
</tr>
</tbody>
</table>

*Values in parentheses are standard deviations of three replicates.

Material to be dyed: Cotton Fabric which completes its Desizing, Scouring process.
Chemicals agents: Copper sulphate, Oxalic Acid and Ferrous sulphate.
Equipment Required:

- Physical balance
- Water Bath
2.2. Methods

2.2.1. Preparation of Raw Material:

The pomegranate peels are removed from the fruit and are dried under shade in sunlight for about 3-4 days until moisture gets dry. Once the peels are dried they are converted into powdered form by grinding it and the finely powdered peel is filtered before processing.

2.2.2. Extraction of Natural dye

The powdered pomegranate peels are soaked in water. After 12 hour, it is boiled at a simmering point for 30 minutes to 1 hour. Then the solution is filtered using cotton cloth. The Filtrate is the extracted liquid dye. The Extracted liquid dye is used for dyeing the Cotton Fabric.

2.3. Process parameter

Parameters for Mordanting
- Material : Liquor ratio - 1:10
- Concentration of Mordants - 4%, 10%
2.4. Mordanting
With Ferrous sulphate

In Iron compound only ferrous sulphate is used as a Mordant. Ferrous sulphate is a versatile mordant. They can be added to dye bath directly as mordant with iron or iron with alum. This mordant is very strong so a little amount gives dramatic colour results. This mordant with pomegranate peel gives dark olive green.

The Pre-Mordanting with Ferrous sulphate is carried out according to Material: Liquor ratio M:L=1:10. The Temperature is maintained at 80°C for 45 minutes with concentrations of 4% and 10%. Finally, the sample is taken out for the dyeing process without wash.

With Copper sulphate

The Copper compound creates an interaction between the fibres and dye. Copper sulphate mordant has an advantage over the iron compound is that it will not rough the surface. It tries to change the colour obtained from the natural dye. It gives good light fastness with greenish tinge colour.

The Pre-Mordanting with Copper sulphate is carried out according to Material: Liquor ratio M:L=1:10. The Temperature is maintained at 80°C for 45 minutes with concentrations of 4% and 10%. Finally, the sample is taken out for the dyeing process without wash.

With oxalic acid

The Mordant oxalic acid is used as a fixing agent for dyes to the fabric. The antimony salts of Oxalic acid is used as mordant. At high vacuum, it will be volatile state. Oxalic acid gives good rubbing fastness compared to ferrous sulphate and Copper sulphate.

The Pre-Mordanting with Oxalic Acid is carried out according to Material: Liquor ratio M:L=1:10. The Temperature is maintained at 80°C for 45 minutes with concentrations of 4% and 10%. Finally, the sample is taken out for the dyeing process without wash.
2.5. Dyeing procedure

For dyeing the textile material the materials required are taken according to the M:L ratio. For Dyeing the material with the natural dye the dye bath temperature is set to 40°-50°C. During dyeing from the total amount of salt taken for dyeing, half of the salt (i.e.50% salt) is added to the dye bath. The Pre Mordanted sample is now taken to the dye bath. Once the sample is introduced to the dye bath the temperature of Dye bath is raised to 90°C and maintained at the same for a period of half an hour. After 30 minutes, when the dyeing of the sample is completed the fabric samples are taken out from the dye bath and they are taken for cold wash treatment. After the cold wash treatment the sample is taken for further evaluation like wash fastness, light fastness and rubbing fastness etc.

3. EVALUATION OF COLOUR FASTNESS PROPERTIES

The Natural dyed Cotton fabric sample is tested for the following fastness types.

3.1. Wash fastness

Washing fastness is one of the important fastness property that has to be assessed in the dyed sample. Wash Fastness test was carried out by using an instrument called Laundrometer. The fabric sample is cut into a piece of 10x4 cm. The specimen ratio is 50:1. Then two pieces of white fabric of 5x4 cm is taken. The white fabrics are placed on the dyed sample such that half the portion of the dyed sample is exposed and the remaining half portion of the sample is hidden or unexposed. The Dyed sample is subjected to attachment with adjacent fabric by stitches. The fabric samples are then put into a stainless steel container with required amount of solvents. Then the samples are taken to the Laundrometer for soaping treatment. After the soaping treatment, the samples are removed from the steel chambers in the machine. The removed samples are then dried under a temperature not exceeding more than 60°C. The dried samples are then assessed by comparing them with the greyscale values.

<table>
<thead>
<tr>
<th>Grades</th>
<th>Fastness Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Poor</td>
</tr>
<tr>
<td>2</td>
<td>Moderate</td>
</tr>
<tr>
<td>3</td>
<td>Average</td>
</tr>
<tr>
<td>4</td>
<td>Good</td>
</tr>
<tr>
<td>5</td>
<td>Excellent</td>
</tr>
</tbody>
</table>

Table-2: Washing Fastness Grades

3.2. Light fastness

Light Fastness test is one of the fastness test. It is a measure of fabric resistance to fading. Light Fastness can be measured in Light Fastness Tester. The dyed fabric sample is attached of specimen size 10×5cm to the specimen holder and continued with exposed to Xenon light source for 24 hours and the sample
is removed from the specimen holder and assessed by comparison with Blue scale or Computer Colour Matching system.

<table>
<thead>
<tr>
<th>Fading Stage</th>
<th>Quality</th>
<th>Grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>Very Deep fading</td>
<td>Very poor</td>
<td>1</td>
</tr>
<tr>
<td>Voluminous fading</td>
<td>Poor</td>
<td>2</td>
</tr>
<tr>
<td>Considerable fading</td>
<td>Fair</td>
<td>3</td>
</tr>
<tr>
<td>Negligible fading</td>
<td>Moderate</td>
<td>4</td>
</tr>
<tr>
<td>Average fading</td>
<td>Good</td>
<td>5</td>
</tr>
<tr>
<td>Minimal fading</td>
<td>Very good</td>
<td>6</td>
</tr>
<tr>
<td>Very minimal fading</td>
<td>Excellent</td>
<td>7</td>
</tr>
<tr>
<td>No fading</td>
<td>Outstanding</td>
<td>8</td>
</tr>
</tbody>
</table>

Table-3: Light Fastness Grades

3.3. Rubbing fastness

Rubbing Fastness is another fastness that has to be assessed in the dyed fabric sample for better performance. The Rubbing Fastness was done in two conditions, Dry condition and Wet Condition. Dyed sample size-12×4 cm White test sample size -4×4 cm

During Dry Condition Rubbing Fastness test, in crock meter the Natural dyed samples are rubbed against white test sample which is placed along the grain lines. Around 10 cycle of were given to the sample by using the handle. The Rubbed sample is compared with grades to assess the degree of staining.

During Wet condition rubbing fastness test, in crock meter the Natural dyed samples in wet condition rubbed against white test sample which is placed along the grain lines. Around 10 cycle of rubs were given to the sample by using the handle. The Rubbed sample is compared with grades to assess the degree of staining.

<table>
<thead>
<tr>
<th>Grades</th>
<th>Fastness Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Poor</td>
</tr>
<tr>
<td>2</td>
<td>Moderate</td>
</tr>
<tr>
<td>3</td>
<td>Average</td>
</tr>
<tr>
<td>4</td>
<td>Good</td>
</tr>
<tr>
<td>5</td>
<td>Excellent</td>
</tr>
</tbody>
</table>

Table-4: Rubbing Fastness Grades
4. RESULT AND DISCUSSION

In the case of Natural Dyeing, Mordants are necessary for interaction with the materials. Ferrous sulphate is a versatile mordant. It is very strong so a little amount gives dramatic colour results. Copper sulphate mordant has an advantage over the iron compound is that it will not rough the surface. Using Oxalic acid as a mordant gives excellent rubbing fastness. When the concentration of ferrous sulphate is 4%, fastness properties like washing fastness rating is 3 according to the grades it gives good washing fastness. Then the rating of Light fastness is 3. The rating of washing and rubbing in which dry & wet condition is 4, then it gives very good fastness.

When ferrous sulphate concentration is 10%, according to grades ,the ratings are 4 for washing fastness and light fastness which evident good wash fastness in comparison with 4% ferrous sulphate concentration. It also gives good Rubbing fastness.

The dyed samples are analysed for various fastness properties and their values are tabulated.

<table>
<thead>
<tr>
<th>Chemicals</th>
<th>Concentration</th>
<th>Washing Fastness</th>
<th>Light Fastness</th>
<th>Rubbing Fastness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferrous sulphate</td>
<td>4%</td>
<td>3</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>10%</td>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Copper sulphate</td>
<td>4%</td>
<td>4</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>10%</td>
<td>3</td>
<td>3</td>
<td>5</td>
</tr>
<tr>
<td>Oxalic Acid</td>
<td>4%</td>
<td>1</td>
<td>3</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>10%</td>
<td>1</td>
<td>2</td>
<td>4</td>
</tr>
</tbody>
</table>

Table -5: Fastness Properties of Pomegranate rind dyed fabric

When the 4% concentration of copper sulphate is used, the fastness properties of Rubbing at dry condition was excellent. The rating of light fastness is 3 which indicates fair fastness. Where the Washing fastness has good fastness with the rating 4 as like the rubbing fastness in wet condition. At 10% concentration the rubbing fastness was good compared to washing and light fastness rating.

When the concentration of oxalic acid is 4% gives excellent rubbing fastness, but poor wash and light fastness. At 10% concentration, rubbing fastness was good compared to 10% concentration of copper sulphate but light and wash fastness was good.
From the above discussion regarding the mordant concentrations and its fastness properties, the mordant with 10% concentration of ferrous sulphate gave better fastness properties compared to the mordant copper sulphate and oxalic acid.

5. CONCLUSIONS

In this article, after performing the process of Dye extraction from pomegranate peel, Mordanting and Dyeing we studied the fastness properties of the dyed sample. We conclude that dyeing of Cotton fabric using pomegranate peel with different mordants to produce different shades, tones, hues. The overall fastness of dyed sample are:

- With Ferrous sulphate
  10% concentration gives Excellent Light fastness and 4% concentration gives good wash fastness.
- With Copper sulphate
  4% Concentration gives excellent Wash fastness and good Light fastness.
- With Oxalic Acid
  4% Concentration gives excellent Rubbing fastness.

6. REFERENCES


BIOGRAPHIES

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