Newer Dyeing Approach with Rose Anthocyanin
INTRODUCTION

• Dyes and pigments are substances that impart color to a material. Dyes are usually soluble in water, while pigments are generally not soluble in water. Most dyes are organic compounds, whereas pigments may be inorganic compounds. Pigments generally give brighter colors and may be dyes.

Advantages of Natural Dyes

- Obtained from renewable sources.
- Health and safety aspects.
- Biodegradable.
- Cost effective.
- Ecofriendly.
Classification of Natural Dyes

On the basis of Chemical Constituents
1. Indigoid Dyes
2. Anthraquinon
3. Alpha-Naphtha
4. Flavones
5. Anthocyanidins
6. Carotenoids

On the basis of Mineral Colorants
1. Red
2. Blue
3. Yellow
4. Black
5. Brown

On the basis of Application
1. Substantive Dye
2. Adjective Dyes
   (1) Direct Dye
   (2) Vat Dye
   (3) Mordant Dye
   (4) Acid Dye
   (5) Basic Dye
   (6) Dispersed Dye

Sources of Natural Dyes
- Plant/Vegetable Sources
- Animal/Insect Sources
- Mineral Sources

Types of Dyes
- Natural
- Synthetic
Anthocyanin pigment

Greek: *anthos* = flower + *kyanos* = blue
It is a Natural Pigment. More than 550 different types of anthocyanins are present in fruits and flowers of plants. It is the most important pigment of plants after Chlorophyll.

**Anthocyanidine** + **A Sugar Molecule** = **Anthocyanin**

**Benefits of Anthocyanin**

1. Attract insects for Pollination.
2. It possesses antioxidant, anti-inflammatory, antimicrobial and anti-cancer activities.
3. Protection from UV-Vis radiation.
4. pH indicator.
Dye Structure and Color

Dyes contain sequences of conjugated double bonds:
X=C−C=C−C=C− . . . ,

Where X is carbon, oxygen or nitrogen

Dyes contained conjugated systems of benzene rings bearing simple unsaturated groups (e.g., −NO₂, −N=N−, −C=O), called chromophores, and polar groups (e.g., −NH₂, −OH), named auxochromes. These Chromophore and Auxochrome are responsible for the color of dye.
Mordants

Mordant bind the natural dyes to the fabrics by the chemical reaction between the dye and the fiber.

Types of Mordants
- Metallic Mordants
  - Tannic Acid
  - Oil Mordants

Methods of Mordanting
- Pre mordanting
- Meta mordanting
- Post mordanting

Dyeing

The dyeing process involves three factors -

- **Dye bath**
  - Cotton, Silk, Wool.
  - Fabric
- **Mordant**
  - Alum, Copper sulphate, Potassium dichromate, Stannous chloride.
The literature review on my dissertation topic constitute about 95 references, which consider about Natural dyeing, it’s benefits, it’s future, Anthocyanin pigment, it’s advantages, it’s extraction from flowers and many more about anthocyanin pigment. Few references are given here-

- In 1930 one of the first chemists to analyze natural dyestuffs was French chemist Pfister, who used a micro chemical analysis.

- Dr. Conrad Hans Eugster (1990) gave an article on the Chemistry of Rose Pigments and presented a survey of the pigments found in the flowers and fruits of old and modern varieties of roses.

- David Hershey (2001) told the specific function of anthocyanin in plants. Anthocyanins are flavonoids. A major function of anthocyanins is to provide color to most flowers and fruits. The colors can help attract pollinating animals to flowers and animals that will help disperse seeds.

- Yoshiumi Kohno, Reina Kinoshita, Shuji Ikoma, Keiko Yoda et al. (2009) proposed the stabilization of natural anthocyanin by intercalation into montmorillonite.

- Lawrence W. J. C., Price J. R. suggested that the principal flower coloring matters are the anthocyanins, anthoxanthins and carotenoids.
MATERIALS AND METHODS

**Materials**
- Flowers of *Rosa rosa*
- Instruments
  - Rotavapor
  - UV-Vis Spectro-photometer
  - Colorsan Machine
- Fabrics
  - Cotton
  - Silk
  - Wool
- Chemicals
  - Tannic Acid
  - Alum
  - Copper sulphate
  - Potassium dichromate
  - Stannous chloride
  - Citric Acid
  - Methanol
Method

Washing and Preparation of Fabric

Tannic Acid Pretreatment of Cotton Fabric

Mordanting

Removal of Methanol from the Anthocyanin Extract

U-V Visible Testing of Anthocyanin

Extraction of Anthocyanin from Rose (Rosa rosa) flower

Dyeing with Anthocyanin Extract

L*a*b* values of dyed fabric (before and after Fixing)

Fixing of dye with Citric acid
RESULTS AND DISCUSSION

Absorption Band and Visible Spectra of Anthocyanin extracted from *Rosa rosa*

The Anthocyanin are characterized by two absorption bands-
Band I- 475-560 nm (Visible region)
Band II- 275-280 nm (UV region)

The broad peak of absorbance was observed at wavelength **526 nm** with the absorbance **0.942** as shown in figure

Yield of Anthocyanin Pigment - 4.074 %.
Results obtained by using Colorscan Machine

Table- 1. L*a*b* values for Cotton fabric dyed with Anthocyanin of *Rosa rosa* before fixing with Citric Acid

<table>
<thead>
<tr>
<th>Mordants</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>C</th>
<th>H</th>
<th>Color %</th>
<th>K/S values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Controlled</td>
<td>41.553</td>
<td>24.166</td>
<td>1.844</td>
<td>24.236</td>
<td>4.362</td>
<td>100</td>
<td>60.6401</td>
</tr>
<tr>
<td>Alum</td>
<td>41.631</td>
<td>23.763</td>
<td>1.601</td>
<td>23.817</td>
<td>3.853</td>
<td>83.484</td>
<td>50.6245</td>
</tr>
<tr>
<td>Copper sulphate</td>
<td>41.786</td>
<td>22.734</td>
<td>3.941</td>
<td>23.073</td>
<td>9.831</td>
<td>120.925</td>
<td>73.3293</td>
</tr>
<tr>
<td>Potassium dichromate</td>
<td>42.574</td>
<td>23.380</td>
<td>6.293</td>
<td>24.212</td>
<td>15.059</td>
<td>97.898</td>
<td>59.3653</td>
</tr>
<tr>
<td>Stannous chloride</td>
<td>40.508</td>
<td>22.795</td>
<td>-2.808</td>
<td>2.967</td>
<td>352.98</td>
<td>218.361</td>
<td>132.4141</td>
</tr>
</tbody>
</table>

Results obtained by using Colorscan Machine
<table>
<thead>
<tr>
<th>Mordants</th>
<th>L*</th>
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<th>C</th>
<th>H</th>
<th>Color %</th>
<th>K/S values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>44.004</td>
<td>7.609</td>
<td>5.851</td>
<td>9.598</td>
<td>37.544</td>
<td>22.452</td>
<td>13.6148</td>
</tr>
<tr>
<td>Alum</td>
<td>43.645</td>
<td>9.862</td>
<td>5.377</td>
<td>11.214</td>
<td>28.409</td>
<td>25.401</td>
<td>15.4033</td>
</tr>
<tr>
<td>Copper sulphate</td>
<td>43.975</td>
<td>13.861</td>
<td>7.901</td>
<td>15.955</td>
<td>29.672</td>
<td>31.118</td>
<td>18.8700</td>
</tr>
<tr>
<td>Potassium dichromate</td>
<td>44.891</td>
<td>12.105</td>
<td>12.207</td>
<td>17.191</td>
<td>45.22</td>
<td>36.328</td>
<td>22.0293</td>
</tr>
<tr>
<td>Stannous chloride</td>
<td>41.726</td>
<td>20.939</td>
<td>4.322</td>
<td>21.380</td>
<td>11.658</td>
<td>215.70</td>
<td>130.8014</td>
</tr>
</tbody>
</table>

Table- 2. L*a*b* values for Cotton fabric dyed with Anthocyanin of *Rosa rosa* after fixing with Citric Acid
Dyeing of Cotton from Anthocyanin
Extracted From *Rosa rosa* using Various Mordants

Controlled Tannic Acid treatment

Alum

Copper sulphate

Potassium dichromate

Stannous chloride
### Table 3. L*a*b* values for silk fabric dyed with Anthocyanin of *Rosa rosa* before fixing with Citric Acid

<table>
<thead>
<tr>
<th>Mordants</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>C</th>
<th>H</th>
<th>Color %</th>
<th>K/S values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>45.641</td>
<td>37.491</td>
<td>-0.941</td>
<td>37.503</td>
<td>358.563</td>
<td>100</td>
<td>48.5124</td>
</tr>
<tr>
<td>Alum</td>
<td>45.177</td>
<td>35.580</td>
<td>-1.567</td>
<td>35.614</td>
<td>357.479</td>
<td>113.067</td>
<td>54.8515</td>
</tr>
<tr>
<td>Copper sulphate</td>
<td>45.605</td>
<td>35.137</td>
<td>0.581</td>
<td>35.142</td>
<td>0.947</td>
<td>108.467</td>
<td>52.6197</td>
</tr>
<tr>
<td>Potassium dichromate</td>
<td>45.772</td>
<td>29.279</td>
<td>5.588</td>
<td>29.807</td>
<td>10.801</td>
<td>139.638</td>
<td>67.7418</td>
</tr>
<tr>
<td>Stannous chloride</td>
<td>43.515</td>
<td>18.853</td>
<td>-4.724</td>
<td>19.436</td>
<td>345.939</td>
<td>283.310</td>
<td>137.4406</td>
</tr>
</tbody>
</table>

**Diagram:**

Dyeing of Silk fabric with anthocyanin from *Rosa rosa* before fixing with citric acid
### Table-4. L*a*b* values for silk fabric dyed with Anthocyanin of *Rosa rosa* after fixing with Citric Acid

<table>
<thead>
<tr>
<th>Mordants</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>C</th>
<th>H</th>
<th>Color %</th>
<th>K/S values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>44.804</td>
<td>31.215</td>
<td>-0.654</td>
<td>31.222</td>
<td>358.800</td>
<td>51.623</td>
<td>25.0437</td>
</tr>
<tr>
<td>Alum</td>
<td>44.581</td>
<td>31.804</td>
<td>-1.757</td>
<td>31.852</td>
<td>356.839</td>
<td>74.217</td>
<td>36.0046</td>
</tr>
<tr>
<td>Copper sulphate</td>
<td>44.734</td>
<td>27.997</td>
<td>0.435</td>
<td>28.000</td>
<td>0.890</td>
<td>58.657</td>
<td>28.4558</td>
</tr>
<tr>
<td>Potassium dichromate</td>
<td>46.266</td>
<td>29.076</td>
<td>7.826</td>
<td>30.111</td>
<td>15.058</td>
<td>102.380</td>
<td>49.6671</td>
</tr>
<tr>
<td>Stannous chloride</td>
<td>44.345</td>
<td>20.189</td>
<td>1.198</td>
<td>20.225</td>
<td>3.395</td>
<td>229.395</td>
<td>111.2850</td>
</tr>
</tbody>
</table>

![Graph showing K/S values](image)
Dyeing of Silk from Anthocyanin
Extracted From *Rosa rosa* using Various Mordants

Controlled (washed)

Alum

Copper sulphate

Potassium dichromate

Stannous chloride
Table-6. L*a*b* values for Wool fabric dyed with Anthocyanin of *Rosa rosa* before fixing with Citric Acid

<table>
<thead>
<tr>
<th>Mordants</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>C</th>
<th>H</th>
<th>Color %</th>
<th>K/S values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>38.791</td>
<td>28.085</td>
<td>2.531</td>
<td>28.199</td>
<td>5.147</td>
<td>100</td>
<td>75.7111</td>
</tr>
<tr>
<td>Alum</td>
<td>38.935</td>
<td>25.902</td>
<td>3.612</td>
<td>26.153</td>
<td>7.935</td>
<td>84.040</td>
<td>63.6276</td>
</tr>
<tr>
<td>Copper sulphate</td>
<td>38.663</td>
<td>22.590</td>
<td>3.727</td>
<td>22.895</td>
<td>9.365</td>
<td>89.550</td>
<td>67.7994</td>
</tr>
<tr>
<td>Potassium dichromate</td>
<td>39.175</td>
<td>15.578</td>
<td>8.809</td>
<td>17.896</td>
<td>29.475</td>
<td>185.550</td>
<td>140.4820</td>
</tr>
<tr>
<td>Stannous chloride</td>
<td>37.015</td>
<td>14.508</td>
<td>-1.306</td>
<td>14.567</td>
<td>354.858</td>
<td>293.855</td>
<td>222.4810</td>
</tr>
</tbody>
</table>
Table-6. L*a*b* values for wool fabric dyed with Anthocyanin of *Rosa rosa* after fixing with Citric Acid

<table>
<thead>
<tr>
<th>Mordants</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>C</th>
<th>H</th>
<th>Color %</th>
<th>K/S values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potassium dichromate</td>
<td>40.405</td>
<td>14.894</td>
<td>12.275</td>
<td>19.300</td>
<td>39.478</td>
<td>133.856</td>
<td>101.3441</td>
</tr>
</tbody>
</table>

![Dyeing of wool Fabric with Anthocyanin from *Rosa rosa* after fixing with Citric Acid](image)
Dyeing of wool from Anthocyanin
Extracted from *Rosa rosa* using Various Mordants

- Controlled (washed)
- Alum
- Potassium dichromate
- Copper sulphate
- Stannous chloride
Comparison of K/S values

In each type of fabric K/S value is decreased after fixing with citric Acid. Wool give the best results among the three types of fabrics used in this study.

Effects of Mordants on K/S values

In **Cotton**, the order of K/s values after fixing was -

1. Control
2. Alum
3. Copper sulphate
4. Potassium dichromate
5. Stannous chloride

In **Silk**, the order of K/s values after fixing was -

1. Control
2. Copper sulphate
3. Alum
4. Potassium dichromate
5. Stannous chloride

In **Wool**, the order of K/s values after fixing was -

1. Alum
2. Control
3. Copper sulphate
4. Potassium dichromate
5. Stannous chloride
conclusion

Anthocyanin pigment was extracted from the petals of *Rosa rosa* and by using it pure natural fabric was dyed. Better results were obtained with few particular mordants but the best results were obtained with stannous chloride mordant with good washing fastness. Due to their non-toxic properties, low pollution and less side effects, there is increasing awareness among people towards natural dyes. Most of the flowers contain anthocyanin pigment and it can very well be used as dye material giving primarily different shades of red.

The wash fastness of metal-complex dyes is due to-

- the ability of dye molecules to associate into large aggregates in the fiber.
- Intermolecular Hydrogen-bonding