Use of beet sugar as wool colourant

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Received 28 October 1999; revised received and accepted 12 June 2000

Beet sugar (Beta vulgaris) colourant was obtained by concentrating beet sugar juice under reduced pressure and evaporating it to dryness. It showed three absorption bands at 220, 280 and 530 nm. The optimum concentration of beet sugar colourant for dyeing of Bharat Merino wool was found to be 0.03 g per g of wool at pH 4.5 and temperature 97.5°C. The wool dyed as such did not meet the requirement of BIS colour fastness standards. However, when the wool treated with metal ions such as Al(III), Cr(VI), Cu(II), Fe(II) and Sn(II) and rare earths such as La(III) and Sm(III) was dyed with beet sugar colourant, it could withstand the requirement of BIS fastness standards. The chemistry of wool dyeing process and the tensile behaviour of treated yarns are discussed.

Keywords: Beet sugar, Beta vulgaris, Betanin, Vulgaxanthin, Wool

1 Introduction

Natural dyes are being used all over the world to impart colours to the textiles, especially in cottage industries. The reddish violet colouring matter of the beet sugar has been found suitable for printing but much less information is available regarding its use in dyeing of textiles1,2. The present paper reports the use of beet sugar as wool colourant, its extraction, optimum conditions for wool dyeing and the fastness properties and tensile behaviour of the dyed yarn. The commonly available metal ions such as aluminum (III), chromium (VI), copper (II), iron (II), tin (II), lanthanum (III) and samarium (III) were used as mordants.

2 Materials and Methods

2.1 Materials

2.1.1 Beet Sugar

The fresh beet sugar (1 kg), which contained nearly 83.5% moisture, was sliced and subjected to extraction on grinder-cum-juicer. The juice was concentrated under reduced pressure on water bath and then treated with 70:30 alcohol-water mixture to remove gum and mucilage. The filtrate was concentrated and dried under reduced pressure over water bath. The yield was found to be 43.16 g per kg of fresh beet sugar. This colourant (abbreviated as BS) consisted of betanin and vulgaxanthin as its constituents. The absorption spectrum of 10 ppm aqueous solution of BS was obtained on UV-visible spectrophotometer 160 (Hitechi). It showed three absorption peaks at 220, 280 and 530 nm.

2.1.2 Wool

Two-ply worsted yarn of Bharat Merino wool obtained from the Central Sheep & Wool Research Institute, Avikanagar, was used for dyeing.

2.2 Methods

2.2.1 Scouring

The worsted yarn was scoured as per the method detailed in IS: 1349:1964. The scoured yarn was subjected to ethanol extraction in soxhlet apparatus at the rate of six siphons/h for 3 h, rinsed with distilled water and finally dried at room temperature. This was done to ensure the removal of residual soap, etc. to facilitate the maximum penetration of dye molecules in the fibre.

2.2.2 Determination of the Optimum Concentration of BS

The stock solution was prepared by dissolving 1.0 g of BS per litre of the solution. Three samples of scoured wool (1.0 g each) were soaked in 2% acetic acid (100 ml, pH 4.5) for nearly 30 min and dyed at a particular concentration of BS prepared from stock solution as per standard procedures3. The process was repeated for six different concentrations of BS, viz. 5,
10, 20, 30, 40 and 50 ml of stock solution, while maintaining volume of the dye bath at 100 ml by using 2% acetic acid solution in each dye bath. The absorbance of the solution was recorded before and after dyeing of wool at 530 nm in each case. An average of three absorbance measurements at each concentration is given in Table 1. The optimum concentration of BS was found as 0.03 g per gram of yarn.

2.2.3 Dyeing of Yarn
The above dyeing treatment was repeated using 10 g wool yarn and 0.3 g BS at boil temperature and 4.5 pH for 60 min. The wool dyed as such did not meet the requirements of BIS colour fastness standards (Table 2). Hence, the treatment of metal ions (mordant) was given to the scoured yarns prior to dyeing.

2.2.4 Pre-mordanting of Yarn
Three samples of scoured yarn (3.0 g each) were subjected to mordanting as per standard procedures by using 2% (w/w) mordant at pH 4.5 and temperature 97.5 °C for 60 min. The mordants used in the present investigation were Cu(II), Fe(II), Sn(II), Al(III), Cr(VI), La(III) and Sm(III).

2.2.5 Dyeing of Pre-mordanted Yarn
The pre-mordanted yarn was subjected to dyeing treatment under optimum conditions as per standard procedure at boil for 60 min at pH 4.5 using the optimum concentration of BS. The absorbance of BS in the dye bath solution was recorded in each case before and after dyeing at 530 nm.

2.2.6 Colour Fastness Tests
The dyed yarns were subjected to testing for their ability to withstand light and wash fastness as per BIS methods IS: 2454-1967 and IS: 687-1979 respectively.

Shirley development light fastness tester SDL 237 was used to test the specimen of dyed yarn. The instrument was adjusted to expose the dyed samples and blue wool standards (rating 1-8) for 5, 10, 20 and 40 h at 65 ± 5 % RH. The exposed dyed samples and blue wool standards were compared after 5, 10, 20 and 40 h of exposures.

The dyed yarn was converted into knitted form measuring 100 mm x 4 mm. It was then subjected to wash fastness test in a Laundrometer using 5.0 g per litre of soap (Lisapol D paste) at 50 ± 2°C for 30 min. The sample was compared with gray scale rating (1-5) as per the BIS method.

### Table 1 — Absorbance of colourant by wool

<table>
<thead>
<tr>
<th>Volume of colourant ml</th>
<th>Absorbance of solution before dyeing</th>
<th>Absorbance of solution after dyeing</th>
<th>Absorption of colourant %</th>
</tr>
</thead>
<tbody>
<tr>
<td>05</td>
<td>0.148</td>
<td>0.760</td>
<td>49.0</td>
</tr>
<tr>
<td>10</td>
<td>0.286</td>
<td>0.075</td>
<td>52.1</td>
</tr>
<tr>
<td>20</td>
<td>0.570</td>
<td>0.137</td>
<td>53.9</td>
</tr>
<tr>
<td>30</td>
<td>0.885</td>
<td>0.263</td>
<td>54.9</td>
</tr>
<tr>
<td>40</td>
<td>1.160</td>
<td>0.399</td>
<td>52.3</td>
</tr>
<tr>
<td>50</td>
<td>1.580</td>
<td>0.553</td>
<td>51.9</td>
</tr>
</tbody>
</table>

### Table 2 — Colour fastness, tensile behaviour and colour of undyed and dyed yarns

<table>
<thead>
<tr>
<th>Yarn type</th>
<th>Light fastness</th>
<th>Wash fastness</th>
<th>Tenacity g/tex at-break</th>
<th>Extension-at-break %</th>
<th>Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scoured</td>
<td>—</td>
<td>—</td>
<td>7.23</td>
<td>11.3</td>
<td>Light yellow</td>
</tr>
<tr>
<td>Dyed</td>
<td>2</td>
<td>2-3</td>
<td>6.55</td>
<td>13.5</td>
<td>Light yellow</td>
</tr>
</tbody>
</table>

2.2.7 Determination of Tensile Behaviour
The experimental yarns were subjected to conditioning at 27 ± 2°C and 65 ± 5% RH for 48 h and the testing was carried out on Instron TM 1102 model. The crosshead speed was adjusted to 25 cm/min so as to break the yarn of 50 cm gauge length within 20 ± 2 s. The tex values were estimated for each tested yarn. An average of twenty-five measurements was taken for each tex, tenacity (g/tex) and extension-at-break values.

3 Results and Discussion
Beet sugar imparts its colour due to the presence of betanin and vulgaxanthin as its constituents. Both have 2,6- pyridine-dicarboxylic acid units in their molecules. The reddish violet colour of BS is stable in acid and neutral medium and changes to yellow in alkaline medium.

Betanin (reddish violet) $\xrightleftharpoons{\text{OH}^-}{\text{H}^+}$ Vulgaxanthin (yellow)

3.1 Dyeing of Wool
The wool fibre morphology and its complex histological structure play a major role in dyeing. When wool enters into the dye bath solution it gets wetted and swells in length and breadth. Its pore size increases approximately by 18% as the dye bath temperature is increased to boil. The BS migrates from the solution to the surface of the wool fibre, gets adsorbed on the surface and diffuses through swollen
pores into the interior of the fibre by means of thermal molecular motion at boil. The colourant molecules are held by weak forces in the wool fibre which carries a large number of functional groups (Fig. 1). Thus, there exist a loose chemical combination between colourant molecules and wool.

The reddish violet solution of BS imparts yellow colour to the wool fibre on dyeing. This is because of the increase in pH within the fibre during dyeing due to partial acid hydrolysis of wool keratin. The yellow colour on wool does not meet the requirements of BIS colour fastness standards (Table 2). However, the colour fastness properties improved when the scoured wool was subjected to mordanting prior to dyeing (Table 3).

### 3.2 Mordanting of Yarn

In pre-mordanting process at boil, the metal ion migrates from solution to the surface of the wool, gets adsorbed and diffuses through swollen pores into the interior of the fibre due to the thermal molecular motion. The size and charge of the metal ion influence the rate of diffusion and wool-metal ion interaction. The metal ion possibly gets attracted to the carboxylic anion of aspartic acid and glutamic acid residue in wool at pH 4.5 forming metal corboxylates (Fig. 2). Similar observations have been made by Hartlel in chrome mordanting. The anion of the metal salt gets attached to oppositely charged group on different protein chains of wool.

### 3.3 Dyeing of Mordanted Yarn

The BS (2,6- pyridine-dicarboxylic acid unit) may possibly act as bi- or tri-dentate ligand. The bi- or trivalent metal ions in the mordanted wool react with BS

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**Table 3 — Colour absorbance, colour fastness, tensile behaviour and colour of pre-mordanted dyed yarns**

<table>
<thead>
<tr>
<th>Mordant</th>
<th>Colour absorbance</th>
<th>Colour fastness</th>
<th>Tenacity (g/tex)</th>
<th>Extension at break (%)</th>
<th>Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before dyeing</td>
<td>After dyeing</td>
<td>Lightness</td>
<td>Wash fastness</td>
<td></td>
</tr>
<tr>
<td>Cu(II)</td>
<td>0.885</td>
<td>0.177</td>
<td>80.0</td>
<td>4-5</td>
<td>6.03</td>
</tr>
<tr>
<td>Sn(II)</td>
<td>0.885</td>
<td>0.141</td>
<td>84.7</td>
<td>3-4</td>
<td>5.30</td>
</tr>
<tr>
<td>Fe(II)</td>
<td>0.885</td>
<td>0.217</td>
<td>75.5</td>
<td>2-3</td>
<td>5.88</td>
</tr>
<tr>
<td>Al(III)</td>
<td>0.885</td>
<td>0.201</td>
<td>77.3</td>
<td>2-3</td>
<td>6.01</td>
</tr>
<tr>
<td>La(III)</td>
<td>0.885</td>
<td>0.138</td>
<td>84.1</td>
<td>4-5</td>
<td>6.12</td>
</tr>
<tr>
<td>Sm(III)</td>
<td>0.885</td>
<td>0.134</td>
<td>84.9</td>
<td>4-5</td>
<td>6.08</td>
</tr>
<tr>
<td>Cr(VI)</td>
<td>0.885</td>
<td>0.121</td>
<td>86.3</td>
<td>4-5</td>
<td>5.84</td>
</tr>
</tbody>
</table>
to form metal chelates I and II (Fig.3). On the basis of IR studies, the carboxylate ion of wool is reported to act as bidentate ligand in chrome mordanting of wool\(^7\). Here also, the carboxylate ion presumably acts as bidentate ligand.

The BS imparts yellow colour on wool mordanted with Sn(II), Al(III), La(III) and Sm(III). The mordant activity of metal ions is found to follow the sequence La(III)<Sn(III)<Al(III)<Sn(II). The different colours achieved on the wool yarn are due to the different mordants used in dyeing process. The wool, when mordanted with Cr(VI), Cu(II) and Fe(II), provides brownish green colour with BS. The colour intensity was found to be maximum when the wool was mordanted with Cr(VI) and least when mordanted with Fe(II). Similar observations have been reported in literature\(^3,10\). Table 3 shows that the pre-mordanted yarn absorbs more colour from dye bath as compared to non-mordanted yarn. Further, the colour absorbed by wool is more when Sn(II), La(III), Sm(III) and Cr(VI) are used as mordant as compared to Cu(II), Al(III) and Fe(II). This may possibly be due to the more availability of binding sites in mordants.

### 3.4 Colour Fastness

As stated earlier, the reddish violet solution of BS imparts yellow colour to the non-mordanted wool yarn. The yellow colour could not withstand the requirement of BIS colour fastness standards (Table 2). However, the yarn first mordanted and then dyed with BS enhances the light and wash fastness. Table 3 shows that the wash fastness of wool yarn first mordanted and then dyed with BS has considerably improved in comparison to that of the yarn dyed only with BS. This is due to the change in the nature of chemical bond and increase in molecular size of wool-metal-dye complex in comparison to wool-dye complex. The wool-metal-dye complex helps in the retention of the dye in the wool fibre.

### 3.5 Tensile Behaviour

The tenacity and extension-at-break do not show any significant change after dyeing the mordanted yarn (Tables 2 and 3). As compared to scoured yarn, the overall extension-at-break was found to increase by 18% on dyeing of yarn, while the tenacity (g/tex) was found to decrease by 20%. However, the wool mordanted with Sn(II) seems to be an exception where extension-at-break was found to improve by 4.85% only and tenacity decreased up to 27%. This may be due to the weakening of S-S linkage of cystine in wool, resulting in formation of tin-sulphur polymeric linkage. The increase in extension-at-break may be due to the rupture of ionic linkage and extraction of gelatin mass from wool fibre in mordanting and dyeing processes. Similar observations have been made by Leeder and Rippon\(^10\).

### 4 Conclusion

Dyeing of wool with beet sugar is possible but for adequate fastness to light and washing, pre-mordanting is necessary. With the use of different metal ions such as Cu(II), Sn(II), Al(III) and Cr(VI) and rare earths such as La(III) and Sm(III) as mordants, different colours like brownish green and yellow are obtained.

### Acknowledgement

The authors are thankful to the Head, Department of Chemistry, University of Rajasthan, for providing facilities for conducting research work. One of the authors (JPM) is grateful to the Director, Central Sheep & Wool Research Institute, Avikanagar, for granting study leave for research work leading to Ph.D. degree, extending facilities for physico-chemical testing and supplying Bharat Merino wool yarn.

### References