A Comparative Study on the Dyeability of Stabraq (Milkweed) Fibers with Reactive Dyes

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ABSTRACT

In this work, raw fibers of a local species of stabraq (milkweed) were scoured, bleached and blended with cotton fibers with a ratio of 75/25 (cotton/stabraq). The improved stabraq fibers, scoured and bleached cotton and the blended fibers were spun into fine yarns and then they were subjected to dyeing with two types of reactive dyes namely, cold brand and high exhaustion brand reactive dye at 1% omf in a liquor ratio of 20:1, in the exhaustion dyeing method. The extent of exhaustion, colour strength and other colorimetric parameters of the dyeing were determined and the results were compared. The results suggested that the dyeability of stabraq fibers with two reactive dyes used, resembles that of cotton yarns more closely, and the blended fibers exhibited superior dyeing behavior than the alone fibers. Prog. Color Colorants Coat. 1(2008) 19-26. © Institute for Colorants, Paint and Coatings.

1. Introduction

Currently, one of the major challenges, in textile industries, is the related environmental problem. Textile industries are facing great pressure to reduce pollutant emissions. This drives textile manufacturers to seek new approaches to produce environmentally friendly products. More and more attention has been drawn to agricultural products, wastes, and derivatives, because of their renewability [1].

Since, the majority of these natural fibers are ligno-cellulosic in nature, they are considered as low value industrial fibers. But the improvements in fiber and yarn production technologies through constant and serious research over the years and ecological considerations, have created a renewed research interest on the lingo-cellulosic fibers to explore their potentials in textile and allied fields. In this respect, many well-known fibers, such as: jute, ramie, sisal, hemp, coir and etc., have been studied and well documented [1, 2]. However, there are few vegetable fibers which still remain unutilized, and have limited usages, or going as natural wastes. One of the least investigated lingo-cellulosic fiber is the seed fiber obtained from stabraq (milkweed). Hence, for this current study, this seed fiber has been chosen to evaluate its dyeability for exploiting the relevant potential applications in textiles.

Stabraq (milkweed) plant belongs to the genus Calotropis Giganteae of the family Asclepiadaceae.

The giant stabraq (milkweed) is native to west and east of Africa, the Arabian peninsula, the South Asia,
Indochina, south of Iran and the Central South America [3].

The above plant is also known as Sodom apple, Calotrope and French cotton [4]. Its native Persian name is stabraq. It is a soft-wooded, evergreen, perennial shrub. It has one or a few stems, few branches, and relatively few leaves, mostly concentrated near the growing tip. The bark is corky, furrowed, and light gray. A copious giant stabraq (milkweed) has a very deep, stout taproot with few or no near-surface lateral roots and reaches depths of 1.3-3.0 meter in sandy desert soils [4, 5].

The giant stabraq (milkweed) usually reaches height of about 2 m, but may occasionally reaches 5 m in height and stem’s diameter of 25 cm [6]. Tissues of this plant, especially the root bark, are used to treat a variety of illness including: leprosy, fever, menstruation, malaria, and snake bites [6-9].

Stabraq (milkweed) fibers are cellulose seed fibers which are growing on single cells in a large seed of the plant. The diameter of the milkweed fibers ranges from 30 to 50 μm and the fiber length ranges from 9.5 to 30 mm, like cotton, it is a single-cell fiber, but unlike cotton, it is without convolutions. Stabraq (milkweed) fibers contain approximately 55% cellulose, 24% hemicellulose, 18% lignin and the remainings are minerals, waxes, fats and other compositions [9, 10].

The fibers are hollow, with a thin wall in relation to their diameter, and are, therefore, light weight. Owing to their structure, they are used in items where good insulation or buoyancy properties are needed, as in: filling fibers for comforters, life vests and winter jackets, and pillows' stuffing. These fibers are filled with tiny hollow tube like structures that act as insulators. The floss obtained from the seed of plant is used for the purpose of stuffing. Blending of stabraq (milkweed) fibers with other fibers causes more cohesion in the blended yarn [9-11].

In the available literature, though there have been some works on characterization of physical properties and spinning of these fibers [9, 10, 12, 13], but there was no attempt to dye them. In view of the above situation, an attempt was undertaken to carry out a research to evaluate the dyeability of these fibers.

This paper examines the dyeability of the stabraq fiber with reactive dyes and compares the dyeing behavior of the fiber with those of both cotton and cotton/stabraq blended fibers.

2. Experimental

2.1. Materials and chemicals

Materials and chemicals used in the study were: raw cotton fiber (scoured and bleached commercial), raw stabraq (milkweed) fiber, sodium hydroxide pellet (analytical grade, Merck, purity > 99.5%) for scouring, hydrogen peroxide (pure, Merck, 50% w/v) for bleaching, sodium carbonate (analytical, Merck), sodium silicate (technical Merck) a stabilizer for hydrogen peroxide, benzene (analytical merck) and methanol (analytical) for defatting and dewaxing of the fiber, distilled water, hydrochloric acid (pure, 35% solution Merck), for washing the scoured fiber to remove the last traces of alkali existed in it, Turkey red oil (sulphonated Castor oil, anionic wetting agent, commercial).

Two reactive dyes, namely C.I. reactive Red 120 and C.I. Reactive Yellow 3 were obtained from commercial sources. All other chemicals used were of analytical grade.

2.2. Methods

Dewaxing and defatting

The fatty and waxy materials present in the bone dry seed fibers were removed by Soxhlet extraction, continuously for 10 hrs using a mixture of benzene and methanol (1:1 v/v). The resulting fiber was dried under vacuum at 45 °C for about 3 hrs [14-24].

Delignification and bleaching treatment

The experimental fiber was subjected to partial delignification by boiling with 1.5% (w/v) NaOH for 1 hr. The material: liquor ratio was 1:40. A wetting agent (Turkey red oil, 1.0 g/l) was used to facilitate wetting and, hence, massive delignification. This solution was decanted and the resultant fiber was digested for 30 mins. at 75 °C, and then followed by washing thoroughly with distilled water to remove the traces of alkali. Final traces of alkali, existed in the fiber, were removed by washing with 1% (v/v) solution of 35% (w/v) HCl at room temperature and lastly, with distilled water until the washing became neutral to litmus paper and then dried under vacuum at 45 °C. Bleaching of the alkali treated fiber was also carried out using alkaline hydrogen peroxide at 70 °C for 20 mins. The fiber based weight percentages of additives, used in the bleaching experiment, were 96-97 water, 0.05 sodium hydroxide, 0.1 sodium carbonate, 0.02 Turkey red oil, and 1:20
sodium silicate. The stabilizer, sodium silicate was added to slow the rate of peroxide decomposition under alkaline conditions. It was combined with or neutralized by metal impurities that might catalyze the decomposition of hydrogen peroxide and induce fiber damaging. It also buffered the peroxide solution by increasing the pH. Sodium hydroxide/sodium carbonate was an activator for hydrogen peroxide. In H₂O₂ bleaching of the fiber, oxidative delignification could also occur [14-24].

**Yarn production**
Cotton and chemically treated stabraq (milkweed) fibers were manually blended in a ratio of 75/25 (cotton/stabraq) and then the samples including: blended one, pure cotton and pure milkweed fibers were carded and spun to fine yarns. Some of the physical properties of the stabraq fibers compared to those of cotton fibers which are given in Table 1.

**Dyeing**
Three specimen including: scoured and bleached cotton, improved stabraq and the blended cotton/stabraq yarns (75/25) were dyed with two types of reactive dyes namely, cold brand reactive dye (C.I. reactive yellow 3) and high exhaustion brand reactive dye (C.I. reactive red 120) in exhaust dyeing method (Figure 1). All dyeing were carried out by using yarns which had been wetted out in cold tap water, employing a 20:1 liquor ratio and 1% omf. The dyeing procedures used for the two dyes are depicted in Figures 2 and 3. Finally, the dyed samples were rinsed thoroughly with cold and hot water and then were dried in air.

![C. I. Reactive Yellow 3](image)

**Figure 1:** Chemical structures of applied dyes.
**Figure 2:** Dyeing of fibers with cold brand dye (C.I. Reactive Yellow 3) [25].

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Time (min)</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>30 °C</td>
<td>10'</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

* pH of dyebath below 7

A. Reactive dye 1% omf  
B. NaCl 2.0 gl⁻¹  
C. NaCl 5.0 gl⁻¹  
D. NaCl 15 gl⁻¹  
E. Na₂CO₃ 0.125 gl⁻¹  
F. Na₂CO₃ 0.125 gl⁻¹  

pH 8.8-9.3  
G. NaOH (flake) 0.125 gl⁻¹  
H. NaOH (flake) 0.125 gl⁻¹  

pH 10.5-11.0  
L. R. = 20:1

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**Figure 3:** Dyeing of fibers with high exhaustion brand dye (C.I. Reactive Red 120) [25].

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Time (min)</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>25-30 °C</td>
<td>5'</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1° min⁻¹</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* pH of dyebath below 7

A. Reactive dye 1% omf  
B. NaCl 2.0 gl⁻¹  
C. NaCl 10 gl⁻¹  
D. NaCl 20 gl⁻¹  
E. Na₂CO₃ 10 gl⁻¹ over 15'
2.6. Dye bath exhaustion

For each of the two dyes used, the extent of exhaustion achieved for a 1% omf. dyeing on each of the three types of yarns was determined by using absorbance spectroscopic analysis of the dyebath before and after dyeing.

The percentage of dyebath exhaustion (%E) achieved for each dye was calculated using equation 
\[ \frac{(A_1-A_2)}{A_1} \times 100 \]

Where \( A_1 \) and \( A_2 \) are, respectively the absorbance of the dye bath prior to and after dyeing at \( \lambda_{\text{max}} \) of the corresponding dye (i.e. 360 nm for C.I. reactive yellow 3 and 510 nm for C.I. reactive red 120).

2.7. Color measurements

The reflectance value (R) of the dyed materials was measured at different wavelengths in the visible region (400-700 nm). The K/S value at a \( \lambda_{\text{max}} \) was calculated, using the Kubelka-Munk equation K/S = (1-R)^2/2R.

The corresponding CIE L*, a*, b*, C*, and h° of the dyed samples were measured, employing a Macbeth Color-eye 7000 spectrophotometer under illuminant D65, and using a 10° standard observer.

3. Results and discussion

The main purpose of this investigation was to compare the dyeability of stabraq (milkweed) fibers, which are cellulosic in nature and structurally comparable with cotton fibers. As it is seen from Table 1, the physical properties of chemically treated stabraq fibers have made them suitable for blending and spinning. We, therefore, attempt dyeing these fibers with two types of reactive dyes, namely, cold brand and high exhaust brand dyes.

The depth of shades (expressed as K/S), the percentage of exhausted (%E), and the other colorimetric data obtained for dyeing of three types of yarns are shown in Table 2.

The results show that the extents of exhaustion (%E) for pure cotton fiber, pure stabraq (milkweed) fiber and the blended one, dyed with the high exhaust brand dye (C.I. Reactive Red 120) are 70, 76 and 72 respectively. The %E values for the same type of fibers dyed with cold brand reactive dye (C.I. Reactive Yellow 3) are 62, 67 and 64. Therefore, the %E values of three dyed fibers have the order: stabraq > blended cotton/stabraq > cotton, which clearly indicates higher exhaustion values for stabraq (milkweed) fibers.

It is well known, that in reactive dyeing, the effective factors include: substantivity of dyes, liquor ratio, temperature, pH, electrolyte concentration, geometry and the structure of both the dye and the fiber. Some of these factors, such as fiber and dye structure are out of the dyer control, whilst some of the other relevant variables, e.g. temperature, pH, etc., are within the dyer control.

The amount of the dye uptake on the fibers in the dyeing conditions could be attributed to structural feature of both the dyes and fibers. In this case, the highest %E of dyes achieved for stabraq (milkweed) fibers, is probably due to its higher content of lignin and hemicellulose, which offer good affinity of dyes toward the fiber. Further the bulky molecular size and planar structure of C.I. Reactive Red 120 have caused more substantivity and exhaustion between the dye and fibers, thereby the %E values for this dye is higher than that of C.I. Reactive yellow 3.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Stabraq Fiber</th>
<th>Cotton Fiber</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean length</td>
<td>21.3 mm</td>
<td>25.9 mm</td>
</tr>
<tr>
<td>Strength</td>
<td>23.0 g/tex</td>
<td>21.0 g/tex</td>
</tr>
<tr>
<td>Fineness</td>
<td>2.4</td>
<td>3.6</td>
</tr>
<tr>
<td>Elongation</td>
<td>6.0%</td>
<td>6.7%</td>
</tr>
<tr>
<td>Diameter</td>
<td>10-18 μm</td>
<td>12-23 μm</td>
</tr>
<tr>
<td>Density</td>
<td>0.97 g/cc</td>
<td>1.54 g/cc</td>
</tr>
</tbody>
</table>
Table 2: The dyeing data related to cotton, stabraq (milkweed) and blended fibers

<table>
<thead>
<tr>
<th>Dye</th>
<th>Fiber</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>C*</th>
<th>h*</th>
<th>k/s</th>
<th>%E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cotton</td>
<td>53.97</td>
<td>53.97</td>
<td>-1.88</td>
<td>46.93</td>
<td>357.70</td>
<td>4.42</td>
<td>70</td>
<td></td>
</tr>
<tr>
<td>R.R.120</td>
<td>53.39</td>
<td>49.03</td>
<td>-0.76</td>
<td>49.03</td>
<td>359.11</td>
<td>4.00</td>
<td>76</td>
<td></td>
</tr>
<tr>
<td>Stabraq/Cotton</td>
<td>57.93</td>
<td>57.93</td>
<td>-2.87</td>
<td>48.08</td>
<td>356.57</td>
<td>5.60</td>
<td>72</td>
<td></td>
</tr>
<tr>
<td>Cotton</td>
<td>74.37</td>
<td>74.37</td>
<td>53.59</td>
<td>55.40</td>
<td>75.35</td>
<td>5.87</td>
<td>62</td>
<td></td>
</tr>
<tr>
<td>R.Y.3</td>
<td>71.86</td>
<td>75.80</td>
<td>51.91</td>
<td>54.56</td>
<td>72.06</td>
<td>5.23</td>
<td>67</td>
<td></td>
</tr>
<tr>
<td>Stabraq/Cotton</td>
<td>76.39</td>
<td>76.39</td>
<td>54.87</td>
<td>57.03</td>
<td>73.24</td>
<td>7.00</td>
<td>64</td>
<td></td>
</tr>
</tbody>
</table>

Table 2 shows the K/S values (depth of shades) for three types of fibers (cotton, stabraq and blended fibers) dyed with C.I. Reactive Red 120 to be 4.42, 4.0 and 5.6 respectively. The K/S values for the same fibers dyed with C.I. Reactive Yellow 3 are 5.87, 5.23 and 7.00. Here, the order obtained from K/S values for the fibers, is presented as: blended cotton/ stabraq> cotton > stabraq.

Because of the rigid structure and the low concentration of accessible sites in stabraq fibers, the lower diffusion and reaction for bulky dyes expected to occur. The relative open structure of the cotton fibers, eases the diffusion of the dye into amorphous region of cotton fibers and finally, more accessibility of sites, yields more fixations.

So, the combined effect of higher exhaustion property of stabraq fibers, amorphous structure of cotton and easier diffusion of small molecular size C.I. reactive Yellow 3 into the cotton fibers has resulted in the highest K/S value (7.00) for cotton/stabraq blend fibers. Despite the high substantivity of C.I. Reactive Red 120, due to its bulky molecular size; it has poor migration properties and thereby the K/S value belonging to the fibers dyed with the above mentioned dye, is lower than those dyed with C.I. Reactive Yellow 3.

In the case of lightness (L*) and chroma (C*), although the values for fibers dyed with C.I. Reactive Yellow 3 is a little higher than those dyed with C.I. Reactive Red 120, it appears that L* and C* values are almost independent of the fiber type and mostly are dependent to the dyes used. Other colorimetric parameters such as: a*, b* and h° obtained for the three types of fibers, are in proper agreement with the expected hue characters of the applied dyes.

However, according to the results, it has been found that stabraq (milkwed) fibers show dyeing behavior similar to that of cotton fibers and the blended fibers exhibit acceptable dyeability when comparing to that of cotton fibers.
4. Conclusions
A comparative study on the dyeability of stabraq fibers has been proceeded, using high exhaust and cold brand reactive dyes.

The present investigation on the seed fiber of stabraq (milkweed) has revealed that the raw milkweed fibers and the blended cotton/stabraq fibers becomes spinable after chemical treatments. The color strength of pure milkweed fibers is lower and its exhaustion value is higher than those of pure cotton. The color, yielded on the blended fibers, has been found to be higher than those of both pure stabraq or cotton fibers. Overall, the obtained results, suggest that the dyeability of stabraq fibers with the two types of reactive dyes used, resembles that of cotton more closely and the blended fibers have gained superior dyeing properties over the two other fiber types used.

5. References
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