Simultaneous dyeing of silk and synthetic fibres with sulphatoethylsulphonyl reactive disperse dyes

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INTRODUCTION
Silk, owing to its characteristic lustre and excellent physical and chemical properties, has long found use as a textile fibre. Although in Japan the demand for the traditional kimono is decreasing, the import of raw silk for Western-style dressmaking is becoming increasingly important. Silk can also be blended with natural fibres (wool, cotton, etc.) or synthetic fibres (polyester, nylon, etc.) to produce a wide range of materials. These blends are most economically dyed in one bath, in order to save water and energy. In practice, one-bath dyeing is used for silk/wool and silk/cellulose fibres [1], but not for silk/synthetic fibres as many problems are encountered.

In the present study an attempt was made to overcome some of these problems by dyeing silk/polyester and silk/nylon 6 fibres with sulphatoethylsulphonyl reactive disperse dyes.

EXPERIMENTAL

Materials

Fabrics
Silk fabric (14 Metsuke Habutae) was used after scouring in a non-ionic surfactant Noygen HC (Daichi Industrial Chemical Co. Ltd) of 1.0 g/l for 30 min at 80°C. The polyester and nylon 6 fabrics used were washed in water for 30 min at 80°C.

Dyes
Two reactive disperse dyes were synthesised by coupling diazotised p-sulphatoethylsulphonyl anilino with NN-disubstituted aniline. The chemical structures and the molecular weights of these dyes are listed in Table 1.

Dyes used in the present study

<table>
<thead>
<tr>
<th>Dye</th>
<th>R1</th>
<th>Substituent</th>
<th>Molecular weight</th>
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<tbody>
<tr>
<td>I</td>
<td>CH₃</td>
<td>CH₃</td>
<td>414</td>
</tr>
<tr>
<td></td>
<td>CH₃</td>
<td>CH₃</td>
<td></td>
</tr>
<tr>
<td>II</td>
<td>CH₃CH₂OH</td>
<td>CH₃CH₂OH</td>
<td>488</td>
</tr>
</tbody>
</table>

Dyeing

Effect of pH
Dye was introduced into the dyeing vessel along with 200 ml buffer solution at various pH values. After the dyebath temperature reached 90°C, each 2 g portion of silk and synthetic fibre (polyester or nylon) was immersed in the liquor and kept there for 1 h. After this the dyed sample was removed and the unreacted dye extracted with methanol. The dyed silk and nylon were dissolved by calcium chloride/water/ethanol mixture (1:8:2 molar ratio) and 90% (by volume) formic acid respectively, cooled to room temperature and diluted to a total volume of 100 ml. The concentration of the solution was determined by colorimetry and the amount of dye fixed was calculated. The amount of dye removed from the bath determined by adding the amount of dye extracted (unfixed) to the amount of dye fixed on the fibre.

The reactive disperse dyes used gave negligible fixation on polyester fabric and so only the unfixed dye was determined by colorimetry of the residual solution.

Effects of temperature and total dye concentration
A constant amount of dye was placed in a dyeing vessel along with 200 ml buffer solution (0.01 mol/l acetic acid/sodium acetate solution at pH 6). After the dyebath temperature achieved a constant value (70, 80 or 90°C), both the silk and the nylon fabrics were immersed in the dyebath where they remained for 0-4 h. The silk and the synthetic fibre were also dyed in a dyebath (pH 6) containing various amounts of reactive disperse dye for 1 h at 90°C. Exhaustion and fixation of dye on the silk and synthetic fibres were determined as described previously.

RESULTS AND DISCUSSION
Silk and polyester fabrics were dyed by reactive disperse dyes 1 and 2 in the dyebath at various pH values. The effects of dyebath pH on the uptake of these dyes on silk and polyester is shown in Figures 1 and 2. Both dyes were exhausted and fixed over a pH range 6 to 8, but only a very small proportion of dye 1 was exhausted onto the polyester, and even less of dye 2. The more hydrophobic dye 1 was taken up more by silk and polyester than was dye 2.

The effect of dye concentration on the uptake of dye 1 by silk and polyester was examined and the results are shown in Figure 3. Exhaustion (on silk and polyester) and fixation (on silk) increased slowly at lower dye concentration and increased somewhat at higher dye concentration. This is due to the low solubility of dye 1 in water. The extent of exhaustion of dye 1 on silk was over 20 times that on polyester at 1.5 × 10⁻⁴ mol/l total dye concentration.

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The relationship between the uptake of dye 2 and dye concentration is portrayed in Figure 4. Exhaustion and fixation of dye 2 on silk increased linearly with dye concentration. At a total dye concentration of $1.5 \times 10^{-4}$ mol/l both parameters were greater with dye 2 than with dye 1. Dye 2 dissolved more readily in water and thus the reaction was promoted by an increase in dye concentration. Dye 2 failed to be adsorbed onto polyester owing to its high hydrophilic character, even at higher concentrations.

**Adsorption of reactive disperse dyes on silk/nylon 6**

The effect of dyebath pH on the uptake of reactive disperse dyes by silk and nylon 6 was examined and the results are shown in Figures 5 and 6. The dye uptake of dye 1 on silk and nylon 6 reached a maximum at pH 8 and 6–8 respectively. The ratio of fixation to exhaustion was found to be very high. At pH 6 the uptake of both dyes on silk was very similar to that on nylon. Dyeing experiments were thus carried out at pH 6 at constant dye concentration. Figures 7 and 8 indicate the variation in exhaustion and fixation with time respectively for dye 1, while Figures 9 and 10 give similar information for dye 2.
Exhaustion and fixation increased with temperature and time in all cases. Increases were noted even after 4 h. The dyeing rates observed when using reactive disperse dyes for silk were essentially the same as those for nylon; this similarity was also found in the relationship between dye uptake and concentration (Figures 11 and 12). The relationship between exhaustion or fixation and dye concentration in the dyeing of silk with reactive disperse dyes was virtually the same as in case of nylon.

The maximum degree of adsorption of dye 1 on nylon was about $2.6 \times 10^{-5}$ mol/g, this being less than the amount of amino end groups in nylon [2]. It is for this reason that nylon adsorbs dye 1 to the same degree as does silk.

REFERENCES
2. NKuroki, Theoretical chemistry of dyeing (Tokyo: Maki-shoten) 43.