Studies on mechanical properties and atmospheric dyeing of solvent pretreated polyester multifilament yarn

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Polyester multifilament yarns were subjected to swelling treatment with different solvents and subsequently dyed with a commercial disperse dye (Dispersol Red-B-3B; C.I. Disperse Red 11) at different temperatures in the range 70-100°C with a view to save thermal energy. It is observed that without much sacrificing the tensile and other mechanical properties of PET and some increase in breaking extension, disperse dye uptake reaches the desirable level at 100°C for pretreatments with m-cresol, dichloromethane, nitrobenzene and phenol at their optimum concentrations of either aqueous or non-aqueous solutions.

Keywords: Dyeing, HT-HP dyeing, Polyester multifilament yarn, Solvent pretreatment

1 Introduction
On commercial polyester fibre, having very compact structure and no dye sites, it is difficult to achieve satisfactory dye depth without the use of high temperature/pressure (HT-HP) or carrier chemicals. HT-HP dyeing is high energy expensive and the use of carrier chemicals has known disadvantages. Besides the use of carrier chemicals, low-temperature or atmospheric dyeing of polyester filaments through solvent pretreatment has potential to gain attention as a energy saving approach. With this in view, the present work was undertaken to study the atmospheric dyeing of PET yarn after different solvent pretreatments. Brennecke and Richter studied the shrinkage and tensile behaviour of fifteen different swelling agents on polyester yarn, but not the dyeing of polyester. Ribnick et al. studied interaction of 26 solvents with different textile fibres, but not the tensile properties and dyeing. The progressive swelling of continuous filament of polyester in phenol is explained by Bobeth. Reports are available in literature on solvent-assisted sequential drawing and its effect on structure of PET filament/fibre. Gulrajani studied the effect of phenol pretreatment on disperse dyeing of PET. Moore and Sheldon have shown a correlation between solubility parameters, shrinkage and swelling of unoriented amorphous polyester by different swelling agents. Dyeing of PET through solvent pretreatment has not been studied in detail and only a few reports are available in literature.

The solvents selected for the present investigation were phenol, m-cresol, nitrobenzene, dichloromethane and chloroform. The selection was made on the basis of shrinkage values and the results of a preliminary study on the shrinkage behaviour of PET in different solvent pretreatments carried out in our laboratory. Phenol and m-cresol were used in aqueous medium while the other three solvents were diluted in trichloroethylene.

2 Materials and Methods

2.1 Materials

Fully drawn, 50 denier, 24 filaments polyester multifilament yarn of M/s Petrofil Cooperative Ltd, India, was used. The yarn in small hank form wrapped in cotton cloth was soxhlet extracted in methanol for 4h, air dried and stored in CaCl₂ desiccator for 24h. All the chemicals, including the solvents, used were of LR grade (M/s S D Fine Chemicals).

2.2 Solvent Pretreatment

Methanol-extracted, dried PET yarns were subjected to solvent pretreatment in small hank form wrapped in cotton cloth. They were dipped in dif-
different solvents of the specified concentration at room temperature for 24h, keeping the material-to-liquor ratio at 1:100. After the solvent treatment, the yams were first neutralized and then prolong-washed with dil. NaOH solution in case of phenol and m-cresol pretreatments and with trichloroethylene in case of dichloromethane, chloroform and nitrobenzene pretreatments. Finally, the yarns were washed with hot water and dried at room temperature.

2.3 Measurement of Shrinkage
The length of the PET filament yarns in hank form was measured, with 0.2 g/den pretension, before and after the solvent pretreatment for calculating the shrinkage (%) consequent to change in length on given solvent treatment.

2.4 Measurement of Tensile Properties
Breaking load and breaking elongation for selected yarn samples were measured in Zwick 1445 CRT Universal Tensile Testing Machine using 100 mm test length and 0.1 N pretension. Tenacity was calculated on the basis of actual denier before and after the solvent pretreatment. Initial modulus was calculated using the stress values corresponding to 1% extension for each sample.

2.5 Dyeing
A commercial disperse dye, Dispersol Red-B3B (C.I. disperse Red 11; mol. wt, 268), obtained from M/s ATIC Industries (Atul), Valsad, was used without further purification to simulate commercial/industrial dyeing procedure. However, for experimental and scientific need, the purity of the dye was tested through repeated soxhlet extraction as suggested by Shenai and Parekh'.

Each dye bath contained: disperse dye, 1% (on the weight of goods, but considering the purity of the dye, the dye percentage in the bath was 0.6); dispersing agent (Setamol-WS), 0.1%; and a few drops of glacial acetic acid to obtain pH 4-4.5. The material-to-liquor ratio was kept at 1:100. The dyeing temperatures used were 70°, 80°, 90°, 100° and 130°C and the duration of dyeing in each case was 60 min. After the dyeing was over, the dyed yarns were washed with water and subjected to usual reduction clearing, soaping and washing and finally air drying at room temperature.

2.6 Measurement of Optical Density and Dye Uptake
The optical density of the dye solution (after extracting the dye from the dyed PET filament yarn using chlorobenzene extraction at 125°C) was measured using U-2000 Hitachi UV/VIS Spectrophotometer by the usual procedure and the dye uptake (g dye/kg of fibre) was noted.

3 Results and Discussion
The solvents act on polyester either by plastici- sising or swelling action with or without some degradation and lowering of glass transition temperature and, therefore, atmospheric dyeing of PET may become possible. The swelling agent diffuses into polymer matrix, swells the polymer and breaks and rebuilds some weak intermolecular forces between polymer chains, causing some structural rearrangement and permissible shrinkage with or without tensile strength loss. At the same time, there is an increase in accessibility in non-crystalline zone for better segregation of crystallites and non-crystallites as observed by Warwicker. Therefore, the effect of solvent pretreatment is somewhat analogous to thermal setting in polyester fibre. With the above background information, the results of the present investigations are discussed.

3.1 Mechanical Properties
Table 1 shows the tenacity, breaking extension, initial modulus and shrinkage values before and after the solvent pretreatment of polyester yarns with different concentrations of aqueous and non-aqueous solutions of the five selected solvents. The concentration ranges used were selected after a number of trial studies with a view to keep a balance among strength loss, breaking extension and moderate to high shrinkage values. For phenol and m-cresol, with increase in solvent concentration above 3%, strength loss becomes higher, breaking extension increase becomes marginal and rate of increase in shrinkage values becomes lesser. At about 3% concentration of the above two solvents, the initial modulus decreases to a large extent. Considering the above studies and the changes in mechanical properties with these two solvents, the concentration for the pretreatment before atmospheric disperse dyeing is selected as 3% for phenol and m-cresol.

The increase in breaking extension in all the pretreatments studied may be explained by high shrinkage with relaxation of the structure (stress relaxation) with chain folding and structural rearrangement on solvent swelling of PET, as reported earlier. In case of nitrobenzene, the strength loss is lowest at all the concentration levels used when compared with that for other solvents (at the comparable concentration level). At
100% concentration, nitrobenzene gives 16.33% shrinkage, marginal strength loss, about 7% increase in breaking extension and initial modulus value of 0.44 g/den. In case of dichloromethane and chloroform in trichloroethylene, the optimum concentration chosen is 50% considering and optimizing the strength retention level (at least 90%), increase in breaking extension (at least more than 35%) and loss of initial modulus (not more than 40-45%). The marginal loss in tenacity on controlled solvent pretreatment and some reduction in initial modulus were as expected from earlier reports.\textsuperscript{4,10-12}

### 3.2 Atmospheric Disperse Dyeing

Disperse dye uptake (g dye/kg of fibre) values of untreated and solvent pretreated PET at 70-100°C for atmospheric dyeing and at 130°C for HT-HP dyeing are shown in Table 2. A comparison of mechanical properties (Table 1) and disperse dye uptake (Table 2) indicates that there is minimum degradation of PET polymer pretreated with 3% aqueous solution of phenol or m-cresol and considerable increase in disperse dye uptake at 100°C (as compared to that at 100°C in the same process for untreated PET yarns). The increase in disperse dye uptake is 48.5% for 3% aqueous m-cresol pretreatment and 17.2% for 3% aqueous phenol pretreatment. But it is difficult to remove m-cresol from PET on washing, even after prolonged alkali wash, and it may degrade the polymer later on and leaves brownish colour on PET which is undesirable. Also, it is known that presence of m-cresol type of compound impairs light fastness for dyed fibres.

Increase in disperse dye uptake at 100°C for dichloromethane pretreatment is >32% and for nitrobenzene pretreatment, >26% (Table 2). This increase\textsuperscript{13} in dye uptake may be explained by lowering of $T_g$ and plasticizing effect of the swelling agents on PET and also by increase in accessibility by structural rearrangement on stress-relaxation and better segregation of crystalline and non-crystalline parts. Chloroform pretreatment not only has health hazards but also gives minimum increase in disperse dye uptake and, therefore, it can also be excluded from the possibility. A comparison of the results of conventional HT-HP dyeing of untreated PET filament at 130°C with those of dyeing at 100°C of PET filaments pretreated with 100% nitrobenzene, 50:50 dichloromethane/TCE and 3% aqueous m-cresol shows that disperse dye uptake is higher in case of dyeing of pretreated PET at 100°C. For 3% phenol pretreated PET, disperse dye uptake is almost similar to that for untreated PET dyed at 130°C by HT-HP process. However, it may be noted that the disperse dye exhaustion is apparently much at lower side in all the cases, probably due to the use of commercial dye directly without further purification. Determination of purity of the commercial dye used revealed that the dye
Table 2—Effect of solvent pretreatment on disperse dye uptake of polyester multifilament yarn

<table>
<thead>
<tr>
<th>Pretreatment</th>
<th>Dye uptake (g/kg of fibre)*</th>
<th>Increase in dye uptake (at 100°C) %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Atmospheric Lab-beaker dyeing at temperature(°C)</td>
<td>Lab HT-HP dyeing at 130°C</td>
</tr>
<tr>
<td>--------------------------------------------------</td>
<td>-----------------------------------------------</td>
<td>--------------------------</td>
</tr>
<tr>
<td>Nil</td>
<td>0.24 0.54 1.20 2.68</td>
<td>3.38 —</td>
</tr>
<tr>
<td>3% Aqueous phenol</td>
<td>0.57 0.97 1.67 3.19</td>
<td>— 17.2</td>
</tr>
<tr>
<td>3% Aqueous metacresol</td>
<td>1.72 1.87 2.75 3.98</td>
<td>— 48.5</td>
</tr>
<tr>
<td>100% Nitrobenzene</td>
<td>0.86 1.82 2.40 3.48.</td>
<td>— 26.1</td>
</tr>
<tr>
<td>50% Dichloromethane in TCE*</td>
<td>1.23 1.92 2.89 3.54</td>
<td>— 32.1</td>
</tr>
<tr>
<td>50% Chloroform in TCE</td>
<td>0.87 1.68 2.12 2.98</td>
<td>— 11.1</td>
</tr>
</tbody>
</table>

*Dye used was Dispersol Red-B-3B (C.I. disperse Red 11) from Atul, India. 1% disperse dye was used on the weight of fibre. The purity of dye was 60%. 100% exhaustion of bath will give 6 g dye/kg of fibre. Maximum exhaustion observed was 56%.

**Temperature used was within ±2°C.

*TCE—Trichloroethylene.

Purity was 60% and therefore the dye uptake results (Table 2) must be read keeping the per cent purity of the dye in mind.

4 Conclusions

Solvent pretreatment route of dyeing polyester (atmospheric dyeing of solvent pretreated PET) with disperse dye using 3% aqueous phenol, 3% aqueous m-cresol, 100% nitrobenzene and 50:50 dichloromethane/trichloroethylene (particularly the last two solvents) with success paves the way of effectively dyeing PET fibre in open bath at 100°C, without much sacrificing tensile and other mechanical properties on solvent pretreatment and ensuring substantial energy saving in polyester dyeing. However, m-cresol and chloroform pretreatments have shown some disadvantages. A detailed study involving some more solvents is needed to work out the commercial viability of this route.

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