Chemical Setting of Nylon 6 Multifilament Yarns in Twist and Knit-de-Knit Texturing

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ABSTRACT

Effects of different solvents on tensile mechanical properties of nylon 6 filament yarns are examined. Treatment with these solvents decreases the yarn strength and increases the breaking elongation. Aqueous solutions of phenol and chlorophenol are used to set the mechanical deformation in twisted yarn and knitted fabrics. Properties of solvents set and heat set yarns are compared. The results are explained with reference to current literature.

Key Words
solvent, texturing, tensile mechanical properties, setting.

INTRODUCTION

Textured yarn is a continuous filament yarn that has been processed to introduce durable crimps, coils and loops along the lengths of filaments. In the production of textured yarn from thermoplastic polymers heat is usually used for setting the mechanical deformations, however, chemical reagents may be used as an alternative for heat for setting the deformed state of the filaments. The yarn can simply be deformed by twisting or knitting. Following setting, the twisted yarn may be untwisted or the fabric may be unravelled, then the yarn is capable of a large amount of elastic deformation and will have higher bulk and flexibility.

According to Jacobs [1], as referred to by Sengupta, et al. [2] the general mechanism of thermostabilization involves breaking of intermolecular bonds, chain relaxation and subsequent bond reformation in an energetically more favorable state. Suitable solvents also break intermolecular bonds causing chain relaxation; and finally the deformed state is set.

Stabilizing twisted yarn by solvents was examined by several researchers both for polyamide [2-6] and polyester yarn [7-8]. According to Moore and Weigmann [9] and Subramanian, et al. [10] treatment of nylon 6 yarn with different solvents increase the yarn dye uptake thus lowering energy cost during dyeing. Hashemi Pour, et al. [7,8] examined these techniques for twist texturing.
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partially oriented and high speed spun polyester yarn. They believed that also for energy saving, thermal energy can be substituted by chemical energy for texturing polyester filaments.

The purpose of this work is to study the effects of solvents on the setting of nylon 6 yarn in both twist and knit-de-knit texturing processes.

EXPERIMENTS

Materials and Methods
Commercial nylon 6 (polyamide), drawn, filament yarns were kindly supplied by Parsilon Co., a producer of nylon fibers. Table 1 shows the specifications of the yarns used in this work. Chemicals used were of reagent grade from Merck. Solvents used were as follows: (1) aqueous solution of phenol (1 to 5%); (2) aqueous solution of formic acid (10 to 50%); (3) aqueous solution of acetic acid (40 to 90%); (4) aqueous solution of ethylcarbonate (3 to 15%); (5) aqueous solution of chlorophenol (0.5 and 1%); and (6) a mixture of methanol and ethanol, all having solubility parameter approximately that of nylon 6. The aqueous solutions were prepared in w/v ratio, unless otherwise stated. Distilled water was used for treatments and for washing the samples.

For determining the effectiveness of different solvents, first undeformed yarns were examined. Loops of yarn were made under constant tension and after treating in slack form in solvent at room temperature (25 °C), the yarns were washed thoroughly with cold water and dried at room temperature for subsequent tests. Yarn shrinkage was calculated by equation 1:

\[ \% \text{SH} = 100 \times \frac{(L_1 - L_2)}{L_1} \]  

(1)

Where \( L_1 \) is the length of a loop of yarn under 1 g (0.0142 g/tex) before treatment with solvents and \( L_2 \) is the length of the same yarn samples after treatment under the same load.

Samples of yarn were then textured by two different methods. In the first method the yarns were twisted in a ring twister with twist multiplier of 80 tex ½ tpc (twist/cm). The twisted yarn was treated with an aqueous solution of phenol or chlorophenol for different length of time at 24 °C. The samples were subsequently washed several times thoroughly with water. After drying at room temperature the twisted yarns were untwisted in the same ring twister. For comparison with heat setting process, false twist textured yarns were also produced by a single heater Minibulk Scragg texturing machine. Heater temperature was 200 °C, yarn twist multiplier in the heater was 80 tex ½ tpc, and the spindle speed was 250,000 rpm. These yarns samples will be referred to as twist-solvent-textured and twist-heat-textured. In the other method, the samples of yarn were knitted in tubing in a small gauge circular knitting machine. Then the tubings were treated with aqueous solutions of phenol or chlorophenol, washed thoroughly by water, dried at room temperature and unravelled. For comparison with heat setting process, samples of fabric were kept in an oven for 5 minutes at 150 °C. These samples will be called knit-de-knit solvent textured and knit-de-knit heat textured yarn, respectively.

Load-elongation curves of single filaments and yarns were measured by an Instron tensile tester. Nominal strength was calculated by dividing breaking load by original tex of yarn and true strength was calculated by dividing the breaking load by the yarn tex at the point of breakage. A Jeol scanning electron microscope (SEM) was used to obtain photomicrographs of the fiber surface. A Carl Zeise Jena polarizing microscope was used for the measurement of fiber diameters and birefringence. Textured yarn crimp rigidities were

<table>
<thead>
<tr>
<th>Table 1. Yarn characteristics.</th>
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<tbody>
<tr>
<td>Count (dtex)</td>
</tr>
<tr>
<td>No. of filaments</td>
</tr>
<tr>
<td>TiO₂ (%)</td>
</tr>
<tr>
<td>Birefringence</td>
</tr>
<tr>
<td>Nominal strength (g/tex)</td>
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<tr>
<td>Breaking elongation (%)</td>
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</tbody>
</table>

* 95% confidence limits.
measured by suspending a hank of yarn under 0.01 g/tex stress and measuring the length $L_1$. Then the hanks were immersed in water for 2 minutes and the length $L_2$ was measured under 0.001 g/tex. Crimp rigidity (C.R) was calculated by equation 2.

$$\text{C.R} = \frac{(L_1 - L_2)}{L_1} \times 100$$

(2)

RESULTS AND DISCUSSION

The results of the shrinkage of yarns after 10 minutes in different solvents are shown in Figures 1 and 2. Figure 3 shows the effect of treatment time on the shrinkage of yarn in aqueous solution of phenol, chlorophenol, formic acid and acetic acid, at 25±2 °C. These results are extracted from two experimental works [11,12]. Treatment with solvents causes the reduction of yarn length. Yarn shrinkage depends on the treatment time and concentration. The shrinkage of yarns in phenol, formic acid and acetic acid depends on the time of treatment and the concentration of solvents. For the mixture of ethanol and methanol and ethylcarbonate, after initial shrinkage, the concentration does not show a profound effect (Figure 2).

It is well established that solving an amorphous polymer in a low molecular weight liquid causes the random coiled polymer molecule to expand and occupy a greater volume than it would in dry amorphous state [13]. It is generally known that when the polymer and solvent have the same solubility parameters the maximum expansion will occur. But for crystalline polymer it seems that during swelling there is a change of molecular order responsible for yarn shrinkage. Subramanian, et al. [10] studied the dyeing and mechanical properties of nylon 6 filaments subjected to swelling treatments. Using benzyl alcohol and an aqueous solution of phenol as swelling reagents, it is concluded that void volume increases in treated filaments. It was stated that chain folding was accompanied by yarn shrinkage when treating nylon 6 with phenol. Moore and Weigmann [9] studied the effect of fiber structural modification
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on mechanical and dyeing behavior of nylon 6 and nylon 66 yarns. Using an aqueous solution of phenol and benzyl alcohol as solvents to modify the fiber structure, it was concluded that these treatments lead to increased crystallinity, the size of crystalline domain and improvement in chain packing.

The results of the yarn nominal strength and breaking elongation after treating in different solvents with different concentrations are shown in Figures 4 and 5 where the 95% confidence limit and averages of at least 15 tests are shown. Treating nylon 6 with solvents reduces the yarn nominal strength. Treatment with solvents increase the breaking elongations. Figures 6 and 7 show the correlation between breaking elongation, nominal and true strength with shrinkage of treated yarn. The correlation coefficient for breaking elongation versus shrinkage is 0.97 and for nominal strength versus shrinkage is 0.90, while for true strength versus shrinkage this value is 0.033. The close correlation of shrinkage versus nominal strength and breaking elongation indicates that there exists the possibility of a similar mechanism for the change in yarn properties by different solvents. The effect of solvent treatment on true strength, showing the inherent properties of filament, is not significant.

In treating nylon 6 with an aqueous solution of phenol, Subramanian, et al. [10] attributed the increased yarn elongation before break to the slippage of the folded chain crystalline units due to the action of deforming forces [10]. Studying the thermochemical and chemomechanical texturing of nylon 6 Sengupta, et al. [2] referred to the possibility of leaching out oligomers causing voids and surface etching due to phenol which may be responsible for the poor strength. The decrease of tenacity following chemical treatments is attributed to a loss in amorphous orientation by Moore and Weigmann [9]. Table 2 shows the effect of different solvents after 10 minutes on the yarn properties. This table indicates that first phenol and then chlorophenol probably interact much
Fig. 3. Effect of solvents concentrations on yarn shrinkage after 10 seconds, 1 minute and 10 minutes, A: formic acid, B: acetic acid, C: phenol, and D: chlorophenol.

Table 2. Properties of solvent treated nylon 6.

<table>
<thead>
<tr>
<th>Solvents</th>
<th>Shrinkage</th>
<th>Nominal strength (g/tex)</th>
<th>Breaking extension (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formic acid (50%)</td>
<td>24.3 ± 0.3*</td>
<td>30.5 ± 0.5*</td>
<td>73.0 ± 1.0*</td>
</tr>
<tr>
<td>Acetic acid (90%)</td>
<td>30.4 ± 0.8</td>
<td>25.8 ± 0.4</td>
<td>106.5 ± 1.2</td>
</tr>
<tr>
<td>Phenol (5%)</td>
<td>44.2 ± 2.4</td>
<td>17.4 ± 0.2</td>
<td>143.3 ± 9.4</td>
</tr>
<tr>
<td>Methanol-Ethanol (18-82%)</td>
<td>9.2 ± 0.2</td>
<td>39.1 ± 0.6</td>
<td>43.6 ± 1.0</td>
</tr>
<tr>
<td>Ethylcarbonate (15%)</td>
<td>7.6 ± 0.4</td>
<td>39.2 ± 0.7</td>
<td>41.4 ± 0.9</td>
</tr>
<tr>
<td>Chlorophenol (1%)</td>
<td>21.8 ± 0.8</td>
<td>30.6 ± 0.6</td>
<td>65.9 ± 1.2</td>
</tr>
<tr>
<td>Water</td>
<td>2.0</td>
<td>43.5 ± 0.5</td>
<td>36.9 ± 1.0</td>
</tr>
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</table>

* 95% confidence limit.
more strongly with nylon filaments and cause structural changes. Then for examining the setting process, only these two solvents were used.

Table 3 shows the properties of heat and solvent set twist textured yarns. The results of crimp rigidity of yarn textured using heat and solvents for setting are comparable. Nominal strength of solvent textured samples is approximately 36% higher than that of heat textured samples. Breaking elongation of heat textured samples is 41% higher than that of solvent set filaments. These results of strength and breaking elongations are not in agreement with those of Sengupta, et al. [4]. Considering that the properties of textured yarn depend on the texturing condition [15], then the disagreement probably is due to the difference in the texturing condition.

Table 4 shows the properties of heat and solvent knit textured yarns. The results of crimp rigidity, nominal strength and breaking elongation of solvent textured and heat textured samples are comparable. The difference between the results of twist texturing and knit-de-knit texturing is probably because the yarns are subjected to

**Table 3. Comparison of heat and solvent twist-textured yarn.**

<table>
<thead>
<tr>
<th>Phenol (3%)</th>
<th>Chlorophenol (1%)</th>
<th>Heat twist texturing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crimp rigidity (%)</td>
<td>25.8 ± 1.8</td>
<td>26.1 ± 1.7</td>
</tr>
<tr>
<td>Nominal strength (g/tex)</td>
<td>33.8 ± 1.3</td>
<td>34.2 ± 0.7</td>
</tr>
<tr>
<td>Breaking elongation (%)</td>
<td>36.9 ± 1.5</td>
<td>33.3 ± 1.7</td>
</tr>
</tbody>
</table>

* 95% confidence limit.
The effect of solvents concentrations on yarn nominal strength and% breaking elongation. A and D: formic acid, B and E: methanol/ethanol (v/v% ethanol), C and F: acetic acid, G and H: water and X shows 95% confidence limits.

Different amounts of deformation.

Figure 8 shows the appearance of untreated, twist-solvent-set and twist-head-set textured yarn. As shown in Figures 8-B to 8-D the crimpiness and waviness of fibers are very prominent in both heat set and solvent set textured yarn.

Figure 9 shows SEM photomicrographs of the surface of solvent set and untreated filaments. With the magnification of microscope used the surface of solvent textured filaments were oily whereas surfaces of heat textured and untreated samples were smooth. It seems that the resolving power of the microscope (approximately 0.2 micrometer) was not sufficient to observe the

Table 4. Comparison of heat and solvent knit-textured yarn.

<table>
<thead>
<tr>
<th></th>
<th>Phenol (3%)</th>
<th>Chlorophenol (1%)</th>
<th>Heat knit-texturing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crimp rigidity (%)</td>
<td>1.7 ± 0.1</td>
<td>1.6 ± 0.04</td>
<td>1.7 ± 0.07</td>
</tr>
<tr>
<td>Nominal strength (g/tex)</td>
<td>27.8 ± 0.7</td>
<td>27.4 ± 0.9</td>
<td>28.8 ± 0.9</td>
</tr>
<tr>
<td>Breaking elongation (%)</td>
<td>19.5 ± 0.6</td>
<td>20.1 ± 0.4</td>
<td>17.9 ± 0.6</td>
</tr>
</tbody>
</table>

* 95% confidence limit.
Detailed structure of the surface of textured filaments.

In addition, the cross-sections of the filaments processed by heat and solvent twist textured filaments were considerably different. While the cross-sections of solvent treated filaments were round and not greatly deformed, those of heat twist textured were polygonal. The cross-sections of those filaments textured by knit-de-knit process were circular, which indicates that the lateral pressures in yarns during solvent treatment were not significant. These differences between the shape of cross-sections have considerable effect on the flexibility and bending rigidities of textured yarn.

CONCLUSION

To examine the setting of the imparted mechanical deformations with reference to the current theories of polymer solubility several solvents were selected and used. Among different solvents, aqueous solutions of 3% phenol and 1% chlorophenol were found to have comparatively better results when used for setting twisted or knitted filament yarn. Between yarn shrinkages, after treatment with solvents, and nominal strength and breaking elongation a close correlation was found, which indicates a similar mechanism for the changes in yarn properties. Extending the views of Moore and Weigmann [9] and Subramanian, et al. [10], the change of yarn properties due to the effect of solvent, in general, could be attributed to the change of fibers structural parameters such as amorphous orientations, void volume, size of crystalline domains, etc. Further work is needed to determine the effectiveness of different solvents in plasticizing and reorganizing the fiber structure.

Tensile mechanical properties and crimp rigidities of the false twist and knit-de-knit textured yarns at comparable conditions were evaluated.
Fig. 7. Correlation between shrinkage and true and nominal strength of yarn treated with different solvents.

Fig. 8. Photomicrograph of twist solvent-set and heat-set textured. A; untreated filaments, B; twist-set 3% phenol, C: twist-set 1% chlorophenol, D: false twist textured yarn.
The results were compared with those textured by solvents setting treatments. It was found that the heat twist textured yarns have a similar crimp rigidity and a lower strength in comparison to solvent twist textured yarns. The results of properties of solvent set knit-de-knit textured yarn were comparable with those of knit-de-knit heat set textured yarn.

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