Solvent texturing of partially oriented nylon-6 multifilament yarn

G Basu*, R K Singh & A K Samanta
The Technological Institute of Textiles, Bhiwani 125 021, India

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Nylon-6 POY was solvent textured at room temperature in a chemomechanical process with the aid of phenol, acetic acid and formic acid. The properties and fine structure of solvent-textured nylon-6 POY were studied and compared with the properties of the base POY yarn as well as with the conventional thermally draw-textured yarn. Yarns solvent-textured with phenol give highest bulk and crimp rigidity and lowest residual shrinkage. They also show high crystallinity but considerable loss in breaking tenacity. Acetic acid-textured sample shows significant shrinkage but unacceptable yarn properties. Formic acid shows results in between those obtained from acetic acid and phenol treatments. The extent of γ-form is reduced in all cases of solvent texturing; however, γ-form content of solvent-textured nylon-6 is found to be higher than that of thermal draw-textured nylon-6.

Keywords: Bulk, Crimp rigidity, Nylon-6, Partially oriented yarn, Residual shrinkage, Solvent texturing, Swelling

1 Introduction

Solvent texturing is a chemomechanical form of texturing process involving mechanical stress and solvent/substrate interaction. In this process of texturing, weakening of intermolecular forces is achieved by solvent/polymer interaction and the application of mechanical stress, followed by removal of solvent, facilitates bond reformation for setting the torque applied during texturing. This may be considered as an alternative to conventional thermomechanical texturing. Limited literature is available on solvent texturing. In the present study, the feasibility of nylon-6 POY as feeder yarn in solvent texturing has been explored.

The conventional thermal draw-texturing process involves high thermal input, high electrical power requirement, heat dissipation in air during cooling, low thermal conductivity of the yarn, thermal variation, irregular crimp set and mechanical instability, which can be avoided in solvent texturing.

Interactions of different polymers and solvents have been investigated by many workers. Ribnick et al. studied interactions of 26 solvents with different textile fibres. Subramanian et al. studied the effect of benzyl alcohol, aqueous solution of phenol and formic acid on nylon-6 filament drawn to different extents and POY yarn also. The mechanism involved in nylon/phenol interactions has been reported to be analogous to the mechanism of thermal setting.

Usenko reported a continuous method of texturing thermoplastic yarn with the aid of solvents and suggested that suitable solvents for nylon yarn might be phenol, sulphuric acid, muriatic acid and formic acid.

Sengupta et al. reported a discontinuous method of solvent texturing of fully drawn nylon-6 filament. They also studied the structural changes occurring on solvent texturing of fully drawn nylon-6 yarn with the aid of phenol as swelling agent.

Gurajani et al. have stated that the feeder yarn should have a relatively low crystallinity for improved crimpability as the coil structure is allowed to set easily. Thus, POY with insignificant crystallinity is a good feeder yarn for texturing. The solvents selected were 3% aqueous phenol, 30% formic acid and 99% acetic acid based on the reported data by earlier workers and also preliminary work done during the present investigations.

2 Materials and Methods

2.1 Materials

2.1.1 Multifilament Yarn

Fully drawn nylon-6 multifilament yarn (105
denier, 24 filaments) was used for study of swelling behaviour in different solvents and nylon-6 multifilament POY yarn (122 denier, 24 filaments) (supplied by L.M.L. Ltd Fibre Division, Kanpur) was taken as feeder yarn for solvent texturing and conventional thermal draw texturing.

2.1.2 Chemicals
Laboratory reagent quality of phenol, trichloroacetic acid, acetic acid, dimethylformamide, phosphoric acid, sulphuric acid, formic acid and hydrochloric acid were taken. Finally, 3% aqueous solution of phenol, 30% aqueous solution of formic acid and 99% (supplied strength) acetic acid were taken for solvent texturing of nylon-6 POY.

2.2 Methods
2.2.1 Swelling Treatment and Shrinkage Behaviour Study
Nylon-6 fully drawn multifilament yarns (105 denier, 24 filaments) of known length were treated with the different solvents or their aqueous solutions at the optimized concentration (where partial dissolution of nylon-6 ceases) at room temperature under completely relaxed condition for 2 min. The solvent-treated samples were neutralized with 5% sodium carbonate solution except the DMF-treated sample which was washed with carbon tetrachloride. After good washing the samples were dried and the final lengths were measured to estimate the percentage shrinkage in the usual way.

2.2.2 Solvent Texturing
Solvent texturing was achieved by the twist-solvent set-detwist process, a discontinuous chemomechanical method. For twisting, an ARCT-CD-8 twister with spindle speed of 7800 rpm was used. The speeds were 3.96 m/min during twisting and 3.64 m/min during detwisting. The twist level was 1960 per metre during twisting and 2144 per metre during detwisting (higher twist during detwisting was used to compensate the contraction in solvent setting) under yarn tension 0.16 g/den. Solvent setting after twisting was achieved by dipping the twisted yarns wound (at constant winding tension of 0.57 g/den) on perforated collapsible stainless steel bobbins in the selected swelling agents (solvents) for 2 min; this facilitates the twisted yarn to shrink during the swelling treatment which is achieved by collapse of the bobbin. After the above treatment, the samples were neutralized with 5% sodium carbonate and then washed and dried in air and detwisted in the same twister in the reverse direction.

2.2.3 Measurement of Crimp Rigidity
The crimp rigidity was measured by the HATRA method and involved relaxing the yarn hank in water at room temperature for 2 min.

2.2.4 Measurement of Bulk
The bulk of yarn was estimated from the following formula:

\[
\text{Bulk percentage} = \frac{\text{Specific volume of textured yarn}}{\text{Specific volume of parent yarn}} \times 100
\]

The specific volume of the yarn was calculated in the usual way from the diameter of the yarns measured under projection microscope under a constant tension of 0.00536 g/den.

2.2.5 Measurement of Denier
The denier of the textured yarn was measured after removing the crimp with suitable load as recommended by the Monsanto Company.

2.2.6 Measurement of Residual Shrinkage
Yarn samples of known length were boiled in water for 10 min in relaxed condition and then the samples were dried and the change in length was measured under a pretension of 0.25 g/den. Finally, the residual shrinkage was expressed in terms of per cent change in length.

2.2.7 Measurement of Mechanical Properties
For breaking load and breaking elongation, the Instron tensile tester (model-1112) was used with gauge length 50 mm, and cross-head speed and chart speed of 100 mm/min and 200 mm/min respectively. The pretension used for the textured yarn was 0.1 g/den, as recommended by the Monsanto Company.

2.2.8 Measurement of Birefringence
The birefringence, \( \Delta n \), was measured by using a Vickers polarizing microscope fitted with a Berek compensator and the following expression:

\[
\Delta n = \frac{6.18 \times \text{Phase difference}}{100 \times \text{Filament diam.}}
\]

2.2.9 X-ray Crystallinity
The wide angle X-ray diffraction pattern was obtained using a Philips X-ray diffractometer. Rotation powder method with identical setting for all samples was used for measuring crystallinity of the fibre. Nickel filtered CuK\(_\alpha\) radiation was used for this purpose. The recordings were taken for 20 values between 10° and 35°.

2.2.10 Estimation of Relative Amount of \( \gamma \)-form
The dependence of the weight fraction of the \( \alpha \)- and
γ-forms in nylon-6 on crystallization conditions has been investigated by Kyotani and Mitsuhashi using X-ray diffraction method. Their method involves the measurement of heights of the diffraction peaks due to the two forms in the X-ray scan. The relative amount of γ-form in the samples of the present work were estimated from the formula:

\[
A_y = \frac{I_y}{I_y + I_{x1} + I_{x2}}
\]

where \(I_y\) is the height of the γ-peak at \(2\theta = 22.7°\); and \(I_{x1}\) and \(I_{x2}\), the respective heights of the peak at \(2\theta = 20°\) and \(24°\) due to the α-form.

3 Results and Discussion

3.1 Effect of Different Solvents on Yarn Properties

3.1.1 Crimp Rigidity and Bulk

The shrinkage and strength loss data for multifilament nylon-6 yarns are shown in Table 1. Some properties like crimp rigidity, bulk percentage, etc. of solvent-textured nylon-6 POY are shown in Table 2. The latter data are in conformity with the shrinkage value of nylon-6 yarn as shown in Table 1 for the three solvents under study. The suitability of phenol for the solvent texturing of nylon-6 POY is apparent from the data. The slightly higher crimp rigidity value for thermal draw-textured yarn may be attributed to the application of higher twist in the latter method. With acetic acid, the values of both bulk and crimp rigidity of solvent-textured nylon-6 POY are much less. Similarly, formic acid is less effective than phenol. These effects may be attributed to the different extents of swelling action in the two cases.

3.1.2 Denier

The increase in denier is found to be the highest (Table 2) in phenol-aided textured nylon-6 POY. This is apparently due to the high bulk and the high shrinkage in phenol. Denier increase is relatively lower for acetic acid-aided and formic acid-aided solvent-textured samples.

3.1.3 Residual Shrinkage

Residual shrinkage provides a measure of dimensional stability of the textured yarn; the lower the value, the higher the stability of the yarn. In phenol- and formic acid-aided solvent-textured nylon-6 POY, residual shrinkage values are found to be lower than that of the thermal draw-textured yarn. Higher value of residual shrinkage of acetic acid-aided textured nylon-6 yarn may be due to acetic acid being a less effective swelling agent.

3.1.4 Initial Modulus

Table 2 shows that the initial modulus of thermal draw-textured nylon-6 POY is higher than the initial modulus of other textured nylon-6 POY samples. The lower value of initial modulus in the solvent-textured samples may be attributed to the formation of solvent-induced crystallites at the cost of a fraction of oriented non-crystalline part through chainfolding as was observed by Subramanian et al. in earlier work. This would contribute to a reduction in initial modulus. The higher initial modulus of formic

| Table 1—Shrinkage behaviour and strength loss of nylon-6 multifilament yarn in different solvents/solutions |
|-----------------|-----------------|-----------------|
| Solvent         | Conc. (wt/wt) % | Shrinkage after 2 min % | Loss in strength after 2 min % |
| Phenol          | 3               | 14.5             | 20.85             |
| Trichloro acetic acid | 7               | 3.55             | 6.95             |
| Acetic acid     | 99              | 9.27             | 2.37             |
| DMF             | 99              | 0.82             | Nil              |
| Phosphoric acid | 50              | 5.7              | 9.8              |
| Sulphuric acid  | 14              | 8.9              | 34.8             |
| Formic acid     | 30              | 10.4             | 18.4             |
| Hydrochloric acid | 8               | 12.6             | 32.0             |
| Water           | 100             | 2.03             | 2.03             |

| Table 2—Properties of untextured, solvent-textured & thermal draw-textured nylon-6 POY base yarn |
|---------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Parameter                      | Untextured POY (Base yarn) | Phenol-textured yarn | Formic acid-textured yarn | Acetic acid-textured yarn | Thermal draw-textured yarn |
| Crimp rigidity, %               | —               | 28.75            | 20.05            | 13.65            | 32.3            |
| Bulk, %                        | —               | 195              | 186              | 177              | 205             |
| Denier                         | 122             | 127.05           | 122.55           | 122.79           | 115.28          |
| Residual shrinkage, %          | 122             | 127.05           | 122.55           | 122.79           | 115.28          |
| Initial modulus, g/den         | 11.5            | 13.25            | 15.25            | 13.25            | 17.5            |
| Breaking tenacity, g/den       | 4.4             | 3.87             | 3.86             | 3.91             | 5.07            |
| Breaking elongation, %         | 75.16           | 43.3             | 50.52            | 37.87            | 52.09           |

1 g/den = 8.83 cN/tex
acid-aided textured nylon-6 POY may be due to its larger fraction of oriented noncrystalline phase. In the case of phenol-aided and acetic acid-aided textured samples, the increase in initial modulus compared to that of POY base yarn can be attributed to the stretching effect during texturing.

3.1.5 Breaking Tenacity

As shown in Table 2, for all the solvents used, a drop in breaking tenacity is observed. This is in line with other reported findings. The breaking tenacity results show an apparent increase in breaking tenacity in the case of thermal draw-textured nylon-6 POY filament; this is apparently because of the sequential thermal draw-texturing, the nylon-6 POY multifilament is first drawn.

The decrease in breaking tenacity of the base partially oriented filaments may be attributed to the polymer-solvent interactions which have a degradative effect, lowering the degree of polymerization of nylon-6 polymer.

3.1.6 Breaking Elongation

On solvent texturing, the breaking elongation decreases. This may be due to the further stretching that occurs during twisting. The increase in orientation is reflected in the corresponding birefringence data given in Table 3. In case of formic acid-aided textured nylon-6 POY filaments, the decrease in breaking elongation is minimum, apparently because of higher non-crystalline fraction.

3.1.7 Birefringence

During texturing there is enhancement of orientation due to the stretching of the yarn as a result of the twist applied. However, the presence of solvent can induce relaxation. As shown in Table 3, the birefringence data for the three solvent-textured yarns indicate an increase in total orientation, and interestingly the highest value is shown by the acetic acid-aided textured nylon-6 POY, apparently because it is less effective swelling agent. The general trend of increase in birefringence may be related to the further orientation that occurs during twisting. On detwisting after solvent setting this preferred orientation is not lost.

3.2 Effect of Solvent Texturing on Fine Structure of Nylon-6

3.2.1 Birefringence

During texturing there is an increase in crystallinity (%), crystallinity (γ-form) of solvent-textured nylon-6 POY is higher in all the cases except in the case of formic acid-textured nylon-6 POY filament. The increase in crystallinity is highest in the case of phenol-aided solvent-textured nylon-6 POY and even higher than that of the conventional thermal draw-textured filament. This may be due to solvent-induced crystalline formation.

3.2.2 Crystallinity

It is observed from Table 3 that crystallinity (%) of solvent-textured nylon-6 POY is higher in all the cases except in the case of formic acid-textured nylon-6 POY. The increase in crystallinity is highest in the case of phenol-aided solvent-textured nylon-6 POY and even higher than that of the conventional thermal draw-textured filament. This may be due to solvent-induced crystalline formation.

3.2.3 Amount of γ-form

As shown in Table 3, there is a decrease in the amount of γ-form during solvent texturing which suggests an increase in stability of structure of textured yarn probably by γ to α transition. However, the amount of γ-form in all the solvent-textured nylon-6 POY yarns is higher than in the case of thermal draw-textured yarn.

4 Conclusions

4.1 Out of the three solvents studied, phenol is found to be the most effective solvent for solvent-aided texturing of multifilament nylon-6 POY yarn. This is because phenol-aided solvent texturing is seen to result in the highest bulk, the highest crimp rigidity and the lowest residual shrinkage. Though the crimp rigidity is still less than that of thermal draw-textured yarn, their bulk values are comparable. Phenol, like other solvents/solutions, shows some loss in breaking tenacity of nylon-6 POY during solvent texturing.

4.2 Acetic acid results in significant shrinkage of nylon-6 filament; however, it gives the lowest bulk and crimp rigidity and highest residual shrinkage values. Thus, acetic acid is not a suitable solvent for texturing of nylon-6 POY yarn at room temperature. Its effectiveness at higher temperature needs to be investigated.

4.3 Formic acid shows properties which are in between those obtained by phenol- and acetic acid-aided solvent texturing of nylon-6 POY.

4.4 The study of fine structure of solvent-textured nylon-6 POY shows that there is an increase of birefringence compared to that of base POY yarn for all the solvents under study. Phenol- and acetic acid-aided solvent texturing show increase in crystallinity but use of formic acid shows more or less...
similar range of crystallinity and sometimes even slightly lower than that of the parent yarn. The crystallinity increase in phenol is highest and even higher than that of thermally draw-textured counterpart. Relative amount of γ-form reduces considerably in solvent-textured nylon-6 POY, but the value is still found to be less than that of thermal draw-textured nylon-6 yarn.

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