EFFECT OF AGING OF POLYCAPROAMIDE CRUMB
ON THE QUALITY OF VIDLON TEXTILE FIBRE

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In storage in the hoppers of spinning machines in nitrogen medium with a regulated oxygen content, oxidative processes inevitably take place in PCA crumb and significantly change the properties of the fibres. Spinning of fibres from PCA crumb stored for different times is an additional cause of their nonuniform properties. Installation of an additional smaller hopper with a day’s reserve of polymer under the machine’s hopper improves the quality of the fibres obtained.

Polycaproamide (PCA) is very sensitivity to thermooxidative degradation. Oxidation of PCA crumb basically takes place before spinning, during storage in two-section spinning machine hoppers, and aqueous extraction of low-molecular-weight compounds [1, 2]. In drying of PCA granules, thermooxidative degradation of PCA is intensified. Prolonged storage of PCA in the model PP-600-156 spinning machine hopper affects its quality [3]. This is due to the formation of stagnant zones in the machine’s hopper, so that crumb stored for different times — from one day to several months — is simultaneously fed in for spinning. Spinning from a mixture of crumb with different storage times is also one of the causes of nonuniform properties of the fibre.

In spinning, PCA crumb is stored in the hoppers of the machines in protective nitrogen medium containing no more than 0.0005% oxygen. The excess nitrogen pressure in the hoppers attains 0.5 atm, and the crumb temperature is maintained within 20-15°C [4]. Increasing the inert gas pressure even at low partial oxygen pressure accelerates oxidation [5]. Even an insignificant oxygen content in the nitrogen can thus initiate thermooxidative degradation.

In practice, the changes that take place in the polymer during storage in spinning machine hoppers are frequently not monitored, but the consequences of these changes are very pronounced. The need to avoid them led us to determine the possibility of altering the design of the hoppers on spinning machines.

Vidlon PCA fibre is spun at the polyamide fibre plant in Vidin on two types of spinning machines — models PP-600-156 and extruder model EVP-2030. It is necessary to note that the hoppers of the extruder machines are designed slightly differently than the PP-600-156 machines: there is a smaller hopper for each extruder directly under the main hopper. The amount of crumb in the smaller hopper is no greater than one day’s reserve. The last load is automatically produced only after the smaller hopper is free of crumb residues. This design solution is very important for attaining the required fibre quality.

We will examine the effect of the storage time of PCA crumb in spinning machine hoppers on the quality of textile fibres.

The experiments were conducted on the spinning machines indicated above. The hoppers of both machines were loaded with the same amounts of polymer. PCA with a relatively viscosity of 2.33 (1% solution on 95.6% sulfuric acid), 0.80% content of low-molecular-weight compounds, and 0.5% crumb moisture content was used. Spinning was conducted on the PP-600-156 in the following conditions: temperature in melting unit of 264±1°C; in the spinning head, 266±1°C; fibre winding speed of 11.17 m/sec. The process conditions on the extruder machine were different: the temperature by zones was 275, 270, 265, 260°C, 255±1°C in the spinning head, winding speed of 15.87 m/sec. The spun fibre with 76 dtex linear density was drawn on a Textima-3008 drawing machine. Due to different spinnneret drawing, the spun fibres obtained on the PP-600-156 machine were drawn at a draw ratio of 3.22 and the fibres from the extruder machine were drawn at a ratio of 3.11.


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Fig. 1. Density ($\rho$, 1, 1'), birefringence index ($\Delta n$, 2, 2'), and CC content (3, 3') in undrawn PCA fibres vs. duration ($\tau$) of storage of the polymer in nitrogen medium in the hoppers of PP-600-156 (1-3) and extruder (1'-3') spinning machines.

Fig. 2. Real breaking load ($\sigma$), elongation at break ($\varepsilon$), and coefficient of variation in elongation at break ($'V'$) of drawn PCA fibre vs. polymer storage time ($\tau$) in nitrogen medium in the hoppers of PP-600-156 (1-3) and extruder (1'-3') spinning machines.

As the time of storage of PCA crumb in the hoppers of the machines increased, the intrinsic viscosity ($\eta$) and degree of oxidation and branching of the polymer were analyzed during the studies.

The degree of oxidation was determined by the content of carbonyl compounds (CC) — one of the products of thermooxidative degradation of the polymer [6]. The CC content was indirectly established by polarography [7, 8]. The degree of branching of the polymer was found with the content of gel particles insoluble in concentrated formic acid [9] and with the Huggins constant [10].

The experiments showed that when the crumb was stored in the machine hoppers for three months, the intrinsic viscosity continuously decreased, although very slowly, and the CC and gel particle content in the polymer increased, and the Huggins constant also increased. Slightly more pronounced initially, this tendency indicates the simultaneous occurrence of thermooxidative degradation and cross linking of PCA.

Thermooxidative degradation of PCA takes place according to a radical chain mechanism and is initiated as a result of decomposition of an intermediate product — polycaproamide hydroperoxide. For this reason, even insignificant oxidation of the crumb during storage in the hopper subsequently becomes very important when the polymer is melted and spun. On the other hand, aging of PCA is accompanied by cross linking processes and the formation of cross links between the macromolecules. The changes in the structure of the polymer affect its fibre-forming ability. The density ($\rho$) and molecular orientation were determined to elucidate the structure of undrawn fibres. The density of the fibres was determined by the gradient tube method using immersion liquids. The molecular orientation was judged by the birefringence index ($\Delta n$) of as-spun fibres using a Amplival Pol D polarization microscope.

As the data in Fig. 1 indicate, the as-spun fibres from the extruder machine were characterized by a higher density than the fibres spun on the PP-600-156 (curve 1'). In our opinion, this is due to the greater spinneret drawing of the fibres spun on the extruder machine.

Spinning is conducted directly in severe temperature and pressure conditions at which specific destructive processes that have a different effect on the structure of the as-spun fibres take place in each of the two types of spinning machines. We previously showed [11] that thermooxidative processes primarily cause degradation in the polymer in spinning on the PP-600-156 machine and mechanical degradation processes predominate in extruder machines under the effect of a temperature-force field. The indexes we analyze are thus the resulting effects of the simultaneous occurrence of several processes.

It was previously shown in [12] that formation of cross links in PCA increases its density. Crystalization of cross-linked units is impeded and development of longitudinal ordering of the chains of the macromolecules is restricted [13].

Our studies showed that as the storage time of PCA crumb increases, the fibre density increases (curves 1 and 1') and the birefringence index decreases (curves 2 and 2'). The increase in the density of the fibres and the decrease in $\Delta n$ thus also...