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Effect of the Solvent Content in Solidifying and Plasticising Baths on the Properties of Polyimidoamide Fibres

Abstract

The effect of solvent content in solidification and plasticising baths on the deformability, absorption and strength properties of polyimidoamide fibres has been examined. The fibres obtained under selected conditions are characterised by improved absorption properties, high thermal stability and a tenacity high enough to allow textile processing.

Key words: *polyimidoamide fibres, fibre formation, absorption properties, strength properties.*

Introduction

The properties of fibres spun by the wet process depend on the solidification process, the fibre structure formed during this stage and its deformability during the plasticising drawing. The role of the as-spun draw ratio in the formation of new polyimidoamide fibres with improved porosity and good moisture absorption properties has been analysed in paper [1]. The modified polyimidoamide polymer has been obtained by the introduction of diamine in the course of the second synthesis stage, as stated in work [4].

The main parameter which allows one to either moderate or intensify the fibre solidification process is the solvent content in the coagulation bath. The solvent content affects the mass exchange process and the development of fibre solidification according to the diffusion mechanism (with a high solvent content in the bath) or the drop mechanism typical of solidification in intensified baths with a low solvent content.

The fibre drawing process in the plasticising bath depends on the solvent content and the bath temperature, as these parameters influence the stress value under which the deformation process proceeds. The increase in the solvent content as well as in the bath temperature is accompanied by an increase in the molecular mobility of macromolecules, facilitating the drawing process and allowing higher deformations to be obtained. At the same time, however, if the solvent content is too high it can cause the process to take place with lower tensile stresses, which adversely affects its effectiveness in terms of increased macromolecule orientation and fibre strength. Thus, it seems necessary to select proper levels of these parameters.

The aim of the present study was to determine the effect of the solvent (N-methypyrrolidone) content in the solidifying and plasticising bath on the deformability, moisture absorption and strength properties of polyimidoamide fibres.

Experimental

The characteristics of the spinning solution, fibre formation conditions and fibre property testing methods were the same as those given in paper [1].

Results and Discussion

The effect of the coagulation bath concentration was examined using variable N-methylpyrrolidone contents from 25% to 65%. This range comprised both intensified and mild solidification conditions. As it was assumed to obtain improved moisture absorption of the fibres and their tenacity at a level high enough to ensure good textile processing, the solidification process was carried out at a low temperature which was selected during preliminary experiments [2].

Fibres were spun at the negative value of the as-spun draw ratio -15%, which was shown [1] to provide higher values of deformation during the drawing stage, and also a higher fibre strength. The drawing process was carried out with deformation values close to the maximum possible for the fibres solidified in a coagulation bath with the given concentration.

From the analysis of moisture absorption properties, it follows that the changes in the moisture absorption at 100% RH (Figure 1) and the water retention (Figure 2) are similar and show their extreme course versus the coagulation bath concentration and the draw ratio value, assuming the maximum value for the low content of the solvent in the coagulation bath at a level of 35%. This is consistent with alternative conditions (to the general rule) of the formation of porous fibres with high moisture absorption [3]. On the other hand, lower values of both

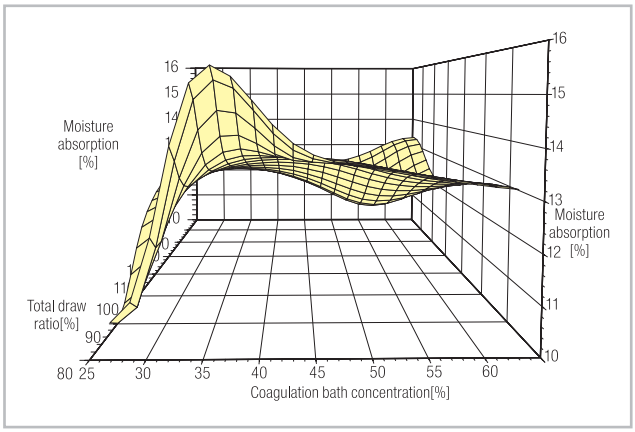

Figure 1. Dependence of moisture absorption at 100% RH on the coagulation bath concentration and the total draw ratio.

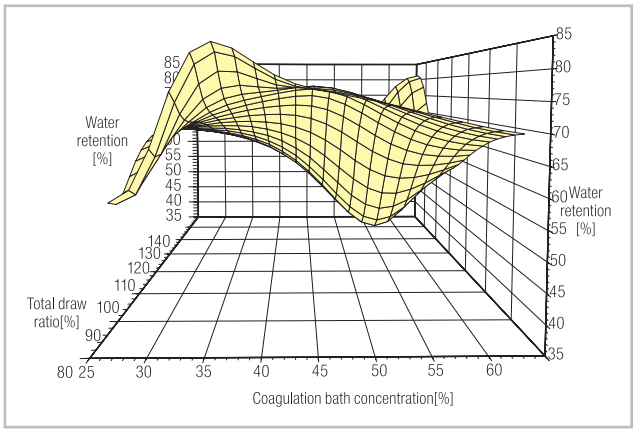

Figure 2. Dependence of water retention on the coagulation bath concentration and the total draw ratio.

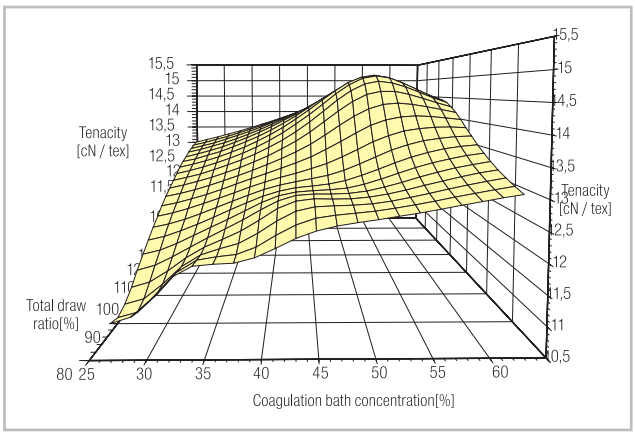

Figure 3. Dependence of fibre tenacity on the coagulation bath concentration and the total draw ratio.

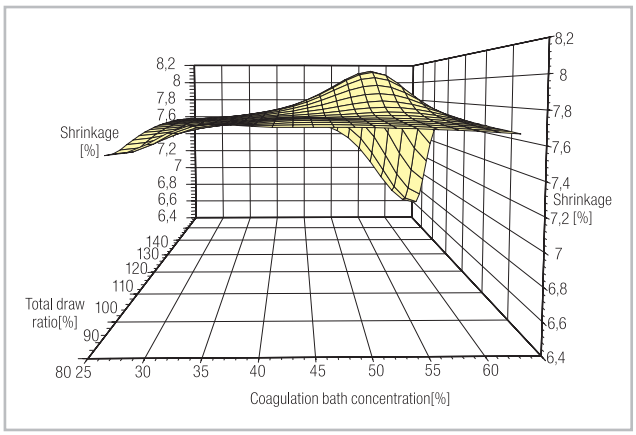

Figure 4. Dependence of fibre shrinkage at 240°C on the coagulation bath concentration and the total draw ratio.

parameters are shown by the fibres solidified in a mild bath with a solvent content of 55%. This is understandable as the process performed at low temperature requires no intensification of the solidification conditions in mild baths; the intensification is necessary to form fibres with an increased porosity and improved moisture absorption properties.

The moisture absorption at 65% RH changes within a narrow range of 4.2-5.3% with the change in the solvent content in the coagulation bath, assuming the highest values for extreme concentrations.

The increased solvent content in the solidification bath leads to the increase in fibre tenacity (Figure 3) from 10.6 cN/tex for intensified baths to 15.1 cN/tex for mild baths with solvent content increased to 55%. However the use of too mild baths with a solvent content of 65% is not recommended, despite the possible use of higher deformations of 148.4 - 150% during the plasticising drawing stage. The drawing is then executed under the influence of lower stresses, which provides no increase in the fibre strength. The value of fibre elongation at break for fibres solidified with variable solvent contents in the bath ranges from 12 to 14.7%. The highest value of this parameter is shown by the fibres solidified in the mildest bath, for which the drawing process could be carried out with the maximum value of the deformation being obtained.

The thermal shrinkage value (Figure 4) changes within a narrow range of 6.6-8.1%, and is associated with the deformation possible during drawing the fibres solidified with the given concentration of coagulation bath. The increase in the solvent content in the solidification bath of up to 55% is accompanied by the increased susceptibility of fibres to deformation during drawing, which results in increased fibre strength and internal stresses. This is shown by the highest value of thermal shrinkage at a level of 8%. An exception to this regularity is the fibres with the lowest shrinkage in the series at a level of 6.6%. They were drawn with the highest values of draw ratio reaching 150%. However due to the high solvent content in the solidification bath, and consequently an increased amount of solvent carried away by the fibres from the bath, the drawing process took place under the influence of lower stresses. This resulted in reduced strength properties (as compared to fibres solidified with 55% of the solvent in the bath). The internal stresses after drawing were also lower, which was shown by the considerably decreased value of fibre thermal shrinkage. This value is connected with the relaxation processes taking place in fibres at 240°C.

It is characteristic that the fibres solidified in the 55% bath show the highest strength properties but the lowest absorption properties in the series. The latter are, however, high if one considers this type of polymer. The moisture absorption at 100% RH is 11.5%, while the water retention is over 40%.

The solvent content in the solidification bath also influences the shape of the fibre cross-section. From the fibre cross-section micro-photograms, it fol-

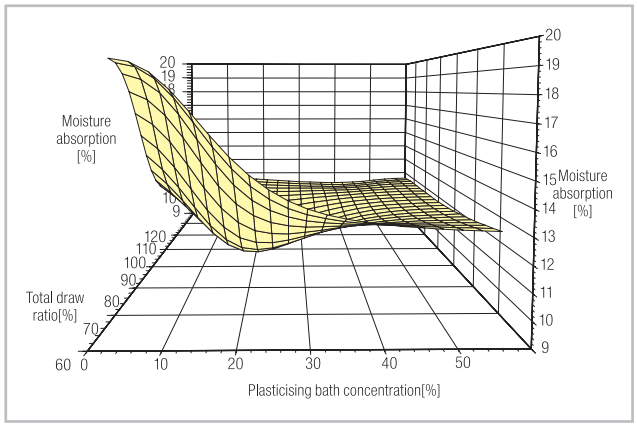

Figure 5. Dependence of moisture absorption at 100% RH on the plasticising bath concentration and the total draw ratio.

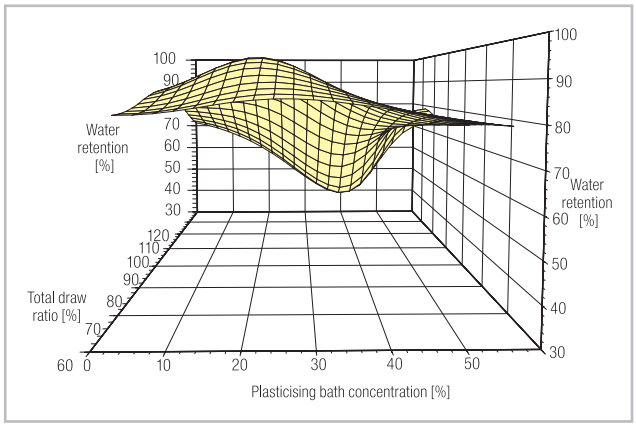

Figure 6. Dependence of water retention on the plasticising bath concentration and the total draw ratio.

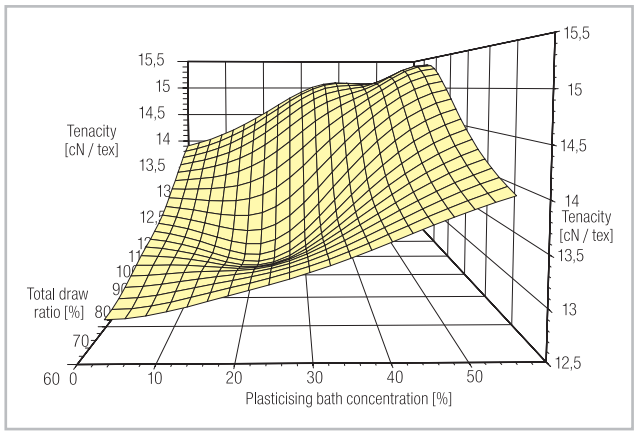

Figure 7. Dependence of fibre tenacity on the plasticising bath concentration and the total draw ratio.

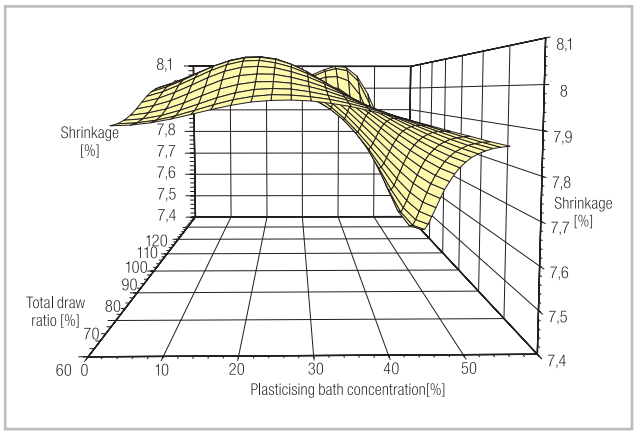

Figure 8. Dependence of fibre shrinkage at 240°C of the plasticising bath concentration and the total draw ratio.

lows that under intensified conditions of solidification (solvent content in the bath: 25%) the fibre cross-section obtained is close to circular. The increase in solvent content and the mild solidification conditions cause the cross-section to shift through an oval to typically bean-like cross-section at the concentration of 55%. In a bath with a high solvent content, the cross-section shape is again close to circular. This is associated with the change which takes place as the solidification conditions are moderated in the ratio of solvent streams to those of non-solvent, which affects the cross-section shape.

The effect of solvent content in the plasticising bath was examined within a wide range of changes in this parameter from 0 to 60%. Such a range of changes included extreme conditions of the process, i.e., drawing in hot water and in a bath with a higher solvent content than that in the solidification bath.

The plasticising bath temperature selected in preliminary experiments [2] was considerably elevated so as to create conditions for better fibre plasticising. From the analysis of absorption properties of the fibres versus the plasticising bath concentration and the fibre draw ratio, it follows that the moisture absorption at 100% RH (Figure 5) and water retention (Figure 6) show an extreme course with a downward trend of both parameters as the solvent content in the plasticising bath increases. The moisture absorption at 65% RH, with the same character of changes, amounts to 4-5.6%. The highest values of moisture absorption and water retention, amounting to 10-19% and 81-98%, respectively, are obtained when the drawing process is carried out in hot water and in a bath with a low solvent content.

Thus the solvent content in the plasticising bath influences not only the fibre deformability, but also the redevelopment of the fibre porous structure and the absorption properties associated with it.

The increase in solvent content in the plasticising bath results in the increased tenacity of fibres (Figure 7) to a level above 15 cN/tex. However too high a solvent content in the fibres is unfavourable to the drawing process, as its excessive content in the plasticising bath does not allow higher deformations to be obtained.

The solvent content in the plasticising bath should not considerably exceed that in the solidification bath (it can be higher by just several per cent).

The value of fibre elongation at break ranges from 16 to 20% depending on the deformation obtained during the drawing stage.

The thermal shrinkage changes within a narrow range from 8.1% to 7.4% (Figure 8), which is connected with the value of fibre internal stresses created during the drawing process executed with various deformations.

Generally, the increase in the solvent content in the plasticising bath to the level decreased in comparison to that in the solidification bath makes it possible to obtain fibres with improved absorption properties (the moisture

Table 1. Properties of polyimidoamide fibres.

Trial symbol	Moisture absorption at 65% RH	Moisture absorption at 100% RH	Water retention	Tenacity	Elongation	Shrinkage at 250°C	Tenacity after heating under stress	
	%	%	%	cN/tex	%	%	cN/tex	%
W_1	4.5	11.0	58.5	15.0	15.9	7.6	14.4	96.0
W_2	4.3	11.0	55.4	16.4	10.0	8.8	-	94.0

absorption at 100% RH of about 12% and the water retention at a level of 78%), with the fibre tenacity being over 15.5 cN/tex.

Under selected conditions, which were most favourable due to the obtained fibre properties, spinning runs were carried out with increased negative value of the as-spun draw ratio. At the same time, in the trial with symbol W_2, the drawing process was performed in two stages with the use of superheated steam in the second stage of drawing. The properties of the resultant fibres are given in Table 1.

The use of two-stage drawing makes it possible to obtain fibres with good absorption properties and a tenacity of over 16 cN/tex. After heating under stress at a temperature of 240°C for 5 h, the fibres maintain 94-96% of their initial tenacity, which indicates a high thermal stability of the fibres made of the new polyimidoamide material.

Conclusions

- The attenuation of solidification conditions makes it possible to obtain a deformable structure during the drawing process. With the extreme course of changes in absorption properties, this is shown by the increase in fibre tenacity.
- The increase in the solvent content in the plasticising bath makes it possible to obtain higher deformations, which results in an increase in the strength of fibres and a decrease in their absorption characteristics at the same time.
- The selection of conditions for spinning the modified polyimidoamide made it possible to prepare fibres with a high thermal resistance and good absorption properties with fibre tenacity at a suitable level for textile processing.

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Acknowledgement

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References

1. *T. Mikołajczyk, Fibres & Textiles in Eastern Europe, Vol. 10, No 1(36), 2002, p. 52-56.*
2. *T. Skwarski, T. Mikołajczyk, Report of the Research Project 7T08E05018 supported by the Committee of Scientific Research, Technical University of Łódź.*
3. *T. Mikołajczyk, Zeszyty Naukowe Politechniki Łódzkiej, No 781, Rozprawy Naukowe, Zeszyt 243, Łódź, 1997 (in Polish).*
4. *T. Skwarski, J. Ratajczyk, T. Mikołajczyk, Fibres & Textiles in Eastern Europe, Vol. 10, No 2(37), 2002, p. 35-38.*

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