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Use of D-Optimal Design to Model and the Analysis of the Effect of the Draw Ratio on Some Physical Properties of Hot Multistage Drawn Nylon 6 Fibers

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ABSTRACT: In this article, we present an experimental design methodology for studying the effect of the draw ratio on the physical properties of nylon 6 fibers on hot multistage drawing. A response surface methodology involving D-optimal design was used for the modeling and optimization. According to the analysis of variance results, the proposed models could be used to navigate the design space. We found that the responses of the tenacity and initial modulus were very sensitive to the factor of the second-stage draw ratio, and the shrinkage response was governed by the factor of the third-stage draw ratio. The results show a good agreement between the experimental and model predictions with high correlation coefficients. The operation conditions for obtaining the drawn yarn with the highest tenacity and initial modulus and low shrinkage are proposed. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 130: 1337–1344, 2013

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INTRODUCTION

Nylon 6 is one of the most important synthetic fibers; it has been investigated extensively because of its long history in a large number of applications. One of the key processes in the course of the post-processing of synthetic fibers is drawing. The drawing process is necessary to improve the tenacity, initial modulus, and dimensional stability of nylon 6 fibers to make them acceptable for use in textile and industrial applications. There are various parameters that are influential in the synthetic fiber drawing process and that play significant roles in determining the final properties of the drawn fibers. The as-spun yarn properties, drawing temperature, number of the drawing steps, drawing speed, and total draw ratio are the main factors to consider.[1,2]

A better control and understanding of synthetic fiber processing are essential requirements for obtaining a desired range of mechanical and physical properties. In this regard, the statistical approach is well suited for the development of the synthetic fiber process technology; it efficiently leads to more reliable and accurate results in a small number of experiments.[3–7]

Although a considerable amount of work has been conducted on the field hot-drawing process,[8–13] there is a lack of literature about the use of a thorough statistical approach for optimizing the process and achieving specific targets. The control of the structure of drawn nylon 6 fibers will be more effective if quantitative relationships can be established between the fiber properties and drawing conditions.

The traditional optimization approach, which usually examines one variable at a time and is used for the optimization of multivariable systems, not only is demanding in terms of time and work but also completely lacks representation of the effect of interactions between different factors. Therefore, an alternate strategy involving a statistical approach should be adopted to determine this complexity involved in the multistage drawing process.

Response surface methodology (RSM) is essentially a particular set of mathematical and statistical methods for designing experiments, building models, evaluating the effects of variables, and determining the optimum conditions of the variables to predict targeted responses.[14,15] In most RSM designs, the relationship between the response and the independent variables is unknown. Thus, the first step in RSM is to fit the appropriate function (response) through the analysis of factors (independent variables). Usually, this process employs a low-order polynomial

Table I. Applied Drawing Conditions for the Multifilament Nylon 6 Yarns

Temperature (°C)						
Feeding roller	First godet roller	Hot plate	Second godet roller	Third godet roller	Drawing speed (m/min)	Spindle speed (rpm)
Ambient temperature	100	170	170	Ambient temperature	400	4000

equation in a predetermined region of the independent variables. The eventual objective of RSM is to determine the optimum operating conditions for the system or to determine the region that satisfies the operating specifications.[15–17]

This study was performed to determine a suitable approximating function to predict and determine future responses and to investigate the operating conditions in a region for the factors at under certain operating specifications. The statistical design was based on three factors [first-stage draw ratio (DR1), second-stage draw ratio (DR2), and third-stage draw ratio (DR3)] and three responses (shrinkage, tenacity, and initial modulus), which were monitored throughout the experiments.

EXPERIMENTAL

Materials and Methods

For the drawing process, a low-oriented polyamide 6 yarn was kindly supplied by Alyaf Co. (Iran). This yarn was melt-spun from nylon 6 chips with a relative viscosity of 2.5. The drawing was done on an industrial Zinser type 520-2 draw-twisting machine (Germany). A three-step drawing process was carried out on heated cylinders (godet roller) and a hot plate through the variation of the draw ratios in three stages. The applied drawing conditions are listed in Table I.

The yarn linear density (expressed in decitex) was determined in accordance with ASTM D 1577-96. The mean values were the average of five measurements.

The stress–strain curves were obtained with an EMT-3050 tensile tester (Elima Co., Iran). A crosshead speed of 500 mm/min and a gauge length of 300 mm were fixed for all of the measurements. From the stress–strain plots, the initial modulus and tenacity were evaluated. The reported values of all of the mechanical properties were averaged over at least 20 independent measurements.

The yarn shrinkages were measured after heating a freely hanging length of yarn in a circulating air oven controlled by an internal thermostat and monitored by an independent thermometer at 130°C for 10 min according to DIN 53840. The initial and final lengths were measured at room temperature, and the total shrinkage was defined as the fraction of the initial sample length remaining after it was exposed to the elevated temperature. The average of five measurements is reported as the shrinkage.

Experimental Design and Data Analysis

Design Expert software (version 7.0) was used for the statistical design of experiments and data analysis. In this study, the RSM and D-optimal design were applied to optimize the three important operating variables in the multistage drawing process. The D-optimal criterion can be used to select points for a mixture design in a constrained region. The experiments were initiated as a preliminary study for determining a narrower and practically feasible range of each step of the draw ratio before the experimental runs were designed. In addition, the sum of the three-stage draw ratios equaled the total draw ratio, and this parameter was considered a constraint for the applied draw ratios. As a result, the study ranges were chosen as DR1 (1.1–2.8), DR2 (1.5–4.2), and DR3 (1.3–3.4). The maximum total draw ratio was fixed at 5.9. To insert constraint into the software, the draw ratio values were converted to the logarithmic form, and then, the following constraint was applied:

$$A+B+C \leq 0.780852$$

where A, B, and C represent log DR1, log DR2, and log DR3, respectively, as shown in Table II.

The D-optimal designed experiments were augmented with five replications to evaluate the pure error and were carried out in a randomized order as required in the design procedure.

Model terms were selected or rejected based on the p value with a 95% confidence level. The results were analyzed completely with analysis of variance (ANOVA) by Design Expert software. The quality of the fit polynomial model was expressed by the coefficient of determination (R^2), and its statistical significance was checked with an adequate precision ratio and by the F test. Three-dimensional (3D) plots and their respective contour plots were obtained on the basis of the effect of the levels of three factors. From these 3D plots, the simultaneous interaction of the two factors on the responses was studied. The optimum region was also identified on the basis of the main parameters. The experimental conditions and the mechanical property results are shown in Table III.

RESULTS AND DISCUSSION

Model Fitting and Statistical Analysis

D-optimal design and RSM were applied to visualize the effects of the independent factors on the responses, and statistical

Table II. Drawing Parameters and Their Levels

Parameter	Code	Low level	High level
log DR1	A	0.04	0.45
log DR2	B	0.18	0.62
log DR3	C	0.11	0.53

Table III. Drawing Conditions and Responses

	Variable			Response		
Run	*A*	*B*	*C*	Shrinkage (%)	Tenacity (cN/tex)	Initial modulus (cN/tex)
1	0.04	0.62	0.11	6.8	63.08	734.3
2	0.32	0.21	0.21	7.3	57.43	722.4
3	0.45	0.21	0.11	7.8	60.48	746
4	0.24	0.18	0.11	4.4	27.05	402.8
5	0.45	0.21	0.11	7.2	57.64	734.9
6	0.04	0.18	0.11	3.2	15.68	229.4
7	0.24	0.41	0.11	6.7	64.31	800.5
8	0.04	0.62	0.11	6.4	62.2	780.4
9	0.04	0.18	0.53	6.5	44.5	654.4
10	0.21	0.28	0.29	7.3	59.63	729.2
11	0.04	0.4	0.11	4.5	28.94	426.1
12	0.04	0.41	0.32	7	60.99	774
13	0.04	0.18	0.11	3	15.91	244.9
14	0.12	0.32	0.19	8.2	36.72	530.5
15	0.24	0.29	0.11	5.8	43.54	428.9
16	0.04	0.18	0.32	7	23.46	375.1
17	0.13	0.21	0.4	7.3	51.67	759.7
18	0.26	0.18	0.34	8.1	59.36	779.4
19	0.04	0.18	0.32	8	24.39	348.5
20	0.04	0.18	0.53	6.3	47.42	768.9

models of the process were developed. Table IV shows the ANOVA results of the established model for responses. For each model equation, the F values implied that the models were significant, and there was only a 0.01% chance that a model F value of this large value could occur because of noise. Values of $p > F$ less than 0.05 implied that the model was significant at the 95% confidence level, whereas values greater than 0.1 are usually considered as insignificant. $p > F$ values of less than 0.0001 denote that all of the employed models are significant.

The lack-of-fit F test describes the variation of the data around the fitted model. If the model does not fit the data well, this will be significant. The lack-of-fit F statistic was not statistically significant as the probability of lack of fit (PLOF) values were greater than 0.05, as presented in Table IV.

The R^2 coefficients give the proportion of the total variation in three responses predicted by the model. In designed experiments, R^2 is a measure of the amount of reduction in the variability of the response obtained with the independent factor variables in the model. High R^2 values for the tenacity and initial modulus responses advocated a satisfactory adjustment of the proposed models to the experimental results. The R^2 for the shrinkage response ($R^2 = 0.899$) showed a fair agreement between the calculated and observed results within the range of the experiment. However, a high value of R^2 does not necessarily imply that the regression model is a good one. Although R^2 always increases with the addition of terms to the model, an adjusted R^2 is preferred. There is a good chance that insignificant terms have been included in the model when the R^2 and adjusted R^2 values differ dramatically.[14,15] The proposed models for the three responses fit very well to the experimental data, and there was reasonable agreement between R^2 and adjusted R^2.

Adequate precision compares the range of the predicted values at the design points to the average prediction error, in other words, a signal-to-noise ratio. Its desired value is 4 or greater.[16] Adequate precision values greater than 4 for all of the responses confirmed that all of the predicted models could be used to explore the experimental domain.

Table IV. ANOVA Results for the Response Parameters

Response	F	$p > F$	PLOF	R^2	Adjusted R^2	AP	SD	CV
Shrinkage	19.46	<0.0001	0.1316	0.899	0.853	13.94	0.59	9.09
Tenacity	259.63	<0.0001	0.3156	0.995	0.991	44.33	1.53	3.39
Initial modulus	181.80	<0.0001	0.2324	0.971	0.966	36.55	1.77×10^{-3}	4.10

AP, adequate precision; SD, standard deviation.

Table V. Model Equations for Each Observed Response

Response	Fitted model	Transformation	Final equation in terms of coded factors
Shrinkage	RQuadratic	None	$7.39 + 0.17A - 0.46B - 2.43C - 1.83AC - 2.27BC - 3.02C^2$
Tenacity	Quadratic	None	$108.63 + 57.62A + 65.08B + 55.34C + 20.26AB + 18.88AC + 21.32BC + 6.77A^2 + 10.81B^2 + 7.15C^2$
Initial modulus	Linear	Inverse square root	$0.024 - 0.013A - 0.014B - 0.014C$

The *A*, *B*, and *C* terms are described in Table II.

The coefficient of variance (CV), as the ratio of the standard error of estimate to the mean value of the observed response, defines the reproducibility of a model. A model normally can be considered reproducible if its CV is not greater than 10%.[17] According to Table IV, the CV value was found to be desirable for all models, and this indicated a good precision and reliability of the experiments.

A multiple-regression analysis of the experimental data was performed, and the model equations in terms of coded factors are presented in Table V. A coded equation was useful for identifying the relative significance of the factors through a comparison of the factor coefficients.

The shrinkage response did not fit well in a proposed quadratic model. Therefore, further modification was made, some insignificant variables and their interactions were eliminated, and the model was transformed to a reduced quadratic model. In the Design Expert software, the response data were analyzed by default. Some raw data might not have been fitted and transformation, which applies a mathematical function to all the response data, might have been needed to meet the assumption that made the ANOVA valid. Data transformation was needed for the initial modulus response as error (residual) was a function of the magnitude of the response (predicted value). Therefore, an inverse square root function was applied for this response.[18,19]

Diagnostic plots, such as the predicted values versus the actual values, and the normal probability plot helped us judge the model satisfaction. In Figure 1, the residuals show how well the models satisfied the assumption of the ANOVA. As shown in Figure 1(a,b), there was no apparent problem with the normality, and this indicated that there was no need for the transformation of the shrinkage and tenacity responses. The predicted versus actual values plots of the responses are presented in Figure 2. The plots for the tenacity and initial modulus responses indicated adequate agreement between the real data and those obtained from the models. The fair correlation between the actual and predicted values for the shrinkage response [Figure 2(a)] was most likely due to the three different variables selected in wide ranges with a limited number of experiments and the nonlinear influence of the investigated parameters on the process responses.[14]

A detailed analysis of the models is presented in the following sections. To save space, only the most interesting and informative 3D and two-dimensional plots are presented later.

Effects of the Parameters

The perturbation plot [Figure 3(a–c)] shows the comparative effects of the three draw ratios on the responses. These plots were obtained at DR1 = 1.1, DR2 = 1.5, and DR3 = 1.3. In Figure 3(a), a steep curvature in the DR3 shows that the response of shrinkage was very sensitive to this factor. The relatively flat lines of DR1 and DR2 showed less sensitivity of the shrinkage to changes in these factors. As shown in Figure 3(b,c), the sensitivity of the tenacity and initial modulus responses to the factors was nearly the same. As shown in Figure 3(b), the factor B (DR2) was steeper than the other factors, and this was correlated with the coefficient of this parameter in the final equation for the tenacity response (Table V). As shown in Figure 3(c), the initial modulus showed the same sensitivity to the changes in the three factors. As shown in Table V, no interaction effects appeared to be significant for the initial modulus.

The equations in Table V were used to facilitate the plotting of the response surfaces. Two parameters were plotted at one time, with another parameter set at the minimum point value. Figure 4 illustrates the effects of the draw ratios at the three stages on the tenacity of the drawn yarns. All of the figures show that the tenacity of the drawn yarn increased with increasing DR1, DR2, and DR3. The curvature of the graphs implies that there was a relatively strong interaction between the variables; this was also reflected by the corresponding low p value (<0.0001). As shown in Figure 4(a), an increase in A from 0.04 to 0.045 at $B = 0.18$ gave an increase in the tenacity of the drawn yarn from 15.68 to 57.64 cN/tex. Meanwhile, an increase in B from 0.18 to 0.62 at $A = 0.04$ gave an increase in the tenacity from 15.58 to 63.08 cN/tex. In both conditions, C was fixed at 0.11.

Figure 4(b) shows that an increase in A from 0.04 to 0.45 at a fixed C value of 0.11 led to an increase in the tenacity of the drawn yarns from 15.68 to 55.62 cN/tex, whereas an increase in C from 0.11 to 0.53 at a fixed A value of 0.04 gave an increase in the tenacity of the drawn yarn from 15.8 to 47.42 cN/tex. In Figure 4(b), B was fixed at 0.18.

Figure 4(c) shows the effects of B and C on the tenacity of the drawn yarn. As observed, an increase in B from 0.18 to 0.62 at a fixed C of 0.11 gave rise to an increase in the tenacity of the drawn yarns from 15.68 to 63.08 cN/tex. Meanwhile, an increase in C from 0.11 to 0.53 at a fixed B of 0.18 led to an increase in the tenacity from 15.68 to 47.42 cN/tex. In Figure 4(c), A was kept at a minimum value of 0.04.

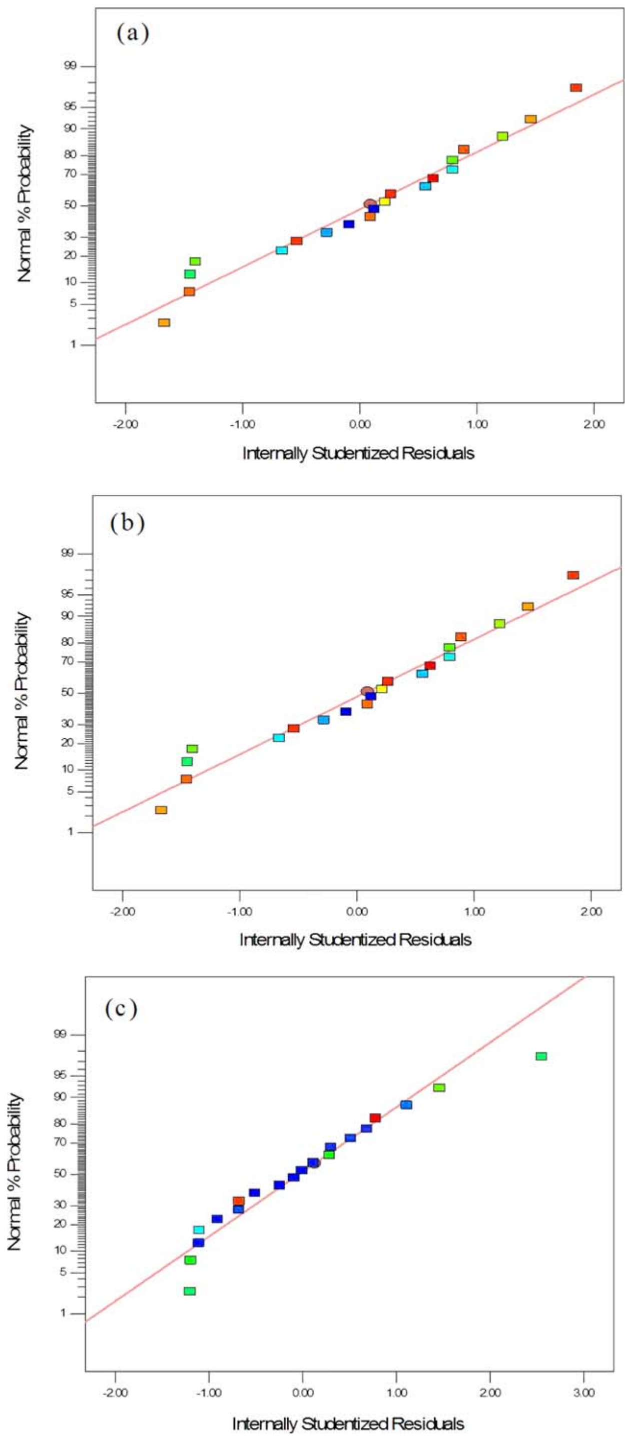

Figure 1. Studentized residuals and normal percentage probability plot for (a) shrinkage, (b) tenacity, and (c) initial modulus. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

By considering the previous results, we found that DR2 was the most important factor affecting the tenacity of the drawn yarns. Moreover, the increase in the tenacity caused by an increase in DR1 at a constant DR3 was greater than the increase in DR3 at a fixed DR1.

Figure 5 represents the effect of A and B on the initial modulus at minimum value of $C = 0.11$. It was found that with simultaneous increases in both variables, the maximum value for initial modulus increased. We found that the variable interaction had a significant effect on the initial modulus. As shown in Figure 5, the increase in the maximum value of the initial modulus caused by an increase in DR2 at a constant DR1 was

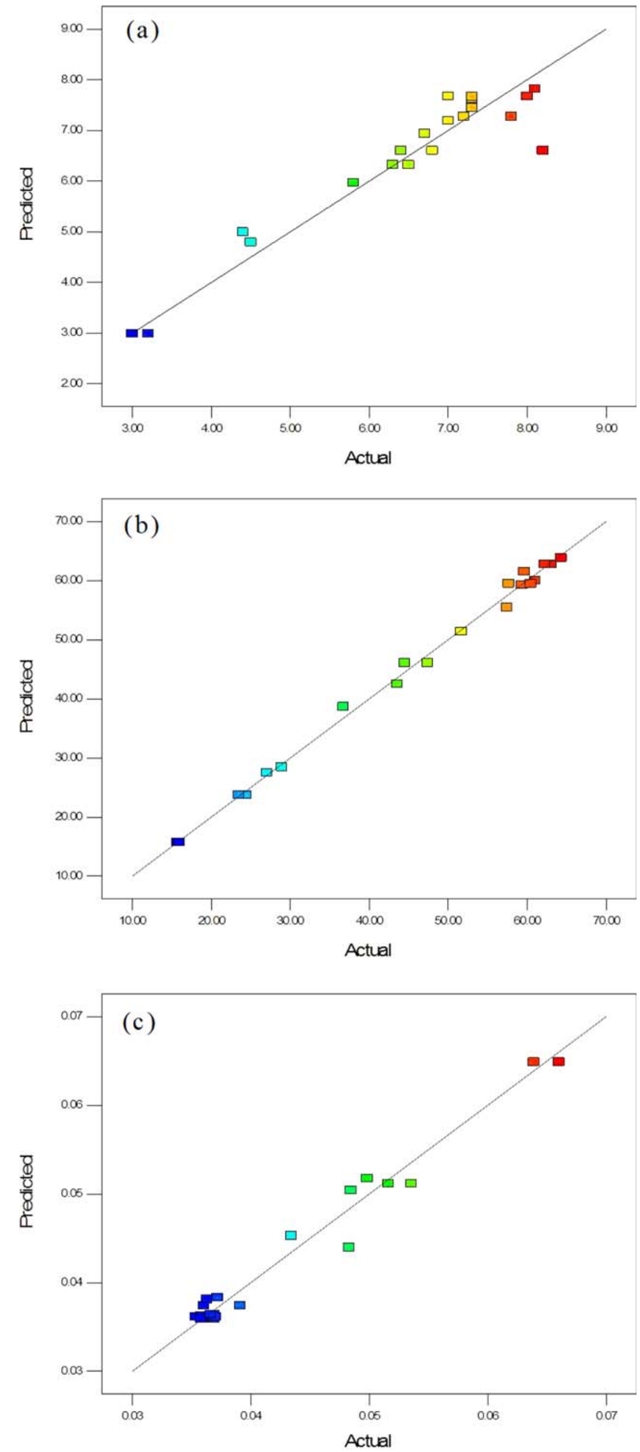

Figure 2. Actual and predicted plots for the (a) shrinkage, (b) tenacity, and (c) initial modulus. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

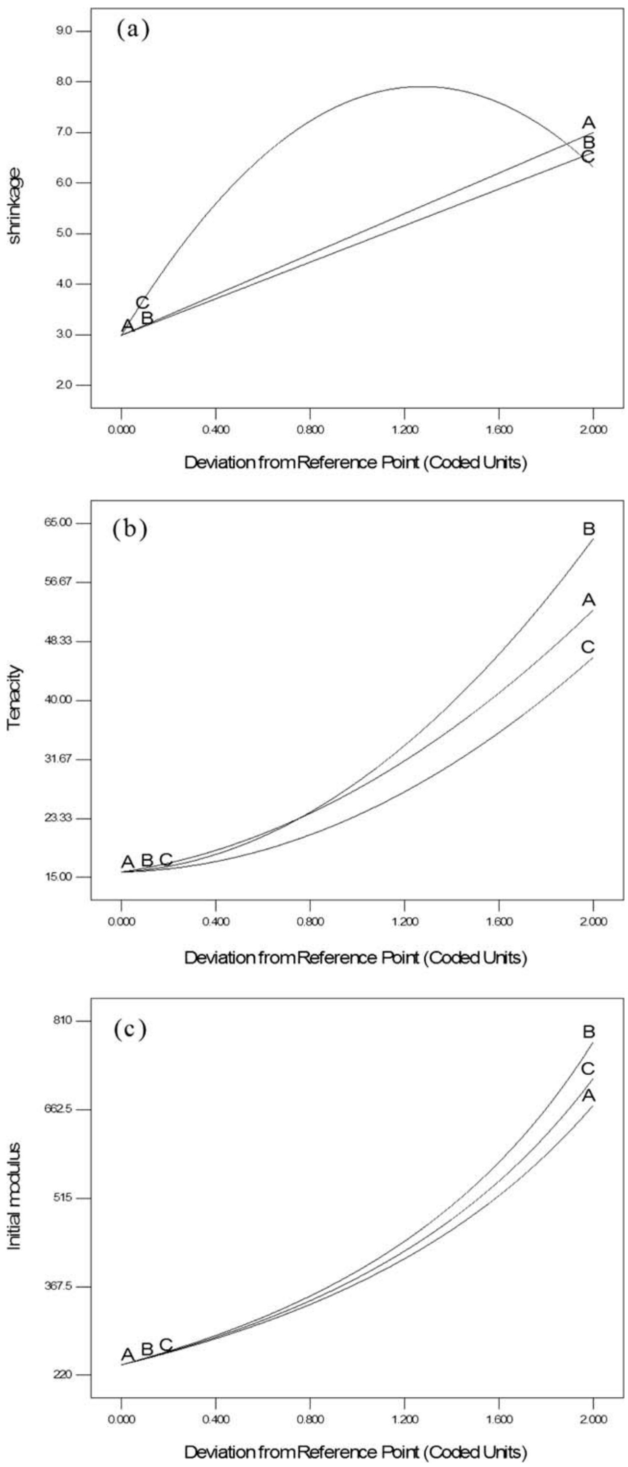

Figure 3. Perturbation plot for the (a) shrinkage, (b) tenacity, and (c) initial modulus.

greater than the increase in the maximum value of the initial modulus resulting from the increase in DR1 at a constant DR2.

According to the model (Table V), the maximum value for the initial modulus was predicted around the middle level of the region, that is, 0.24 and 0.41 for *A* and *B*, respectively.

Figure 6 shows the response surface plots as functions of the *C*–*A* and *B*–*C* interactions on the shrinkage. As shown, there was an increase in the shrinkage value with increasing *A* and *B* at each value of *C*, whereas in the other axis, when *C* exceeded a certain limit value, the shrinkage started to decrease.

The obtained crystallinity and polymer chain orientation in the filaments in the first and second stages of the drawing process strongly affected the final properties of the drawn fibers. So we considered how the conditions in the first and second stages of drawing affected the structure of the drawn fibers before the final stage. According to Figure 6, when DR1 or DR2 was set at a low level, an increase in DR3 gave rise to an increase in

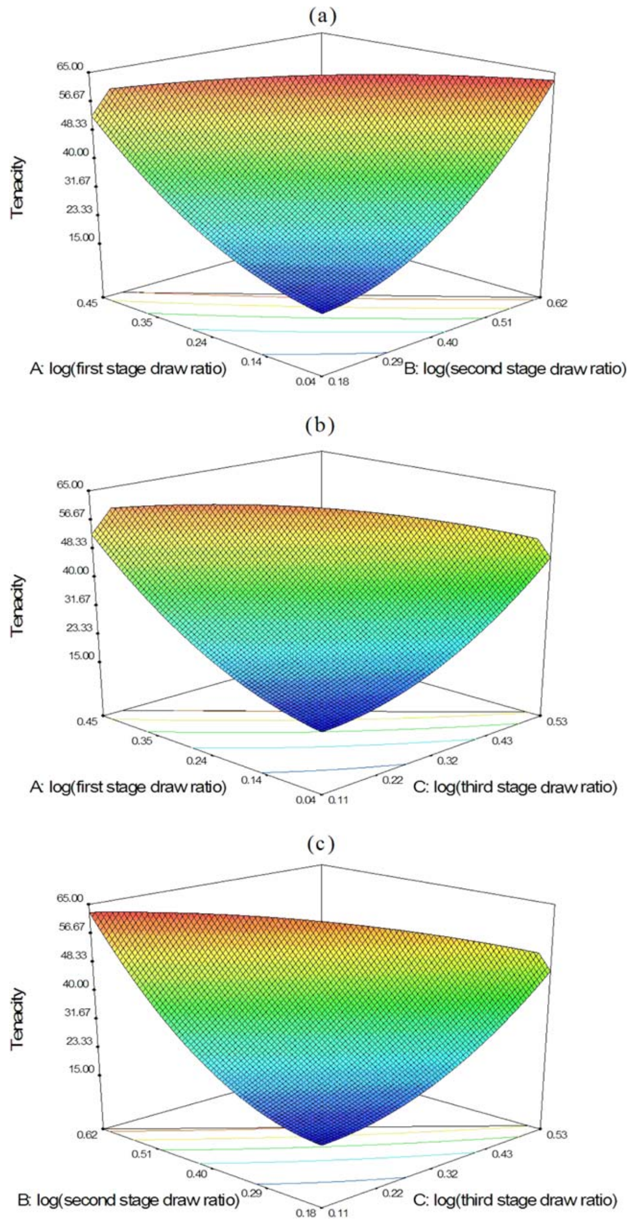

Figure 4. Response surface plot for the tenacity; values of $C = 0.11$, $B = 0.18$, and $A = 0.04$ were constant for parts a, b, and c, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

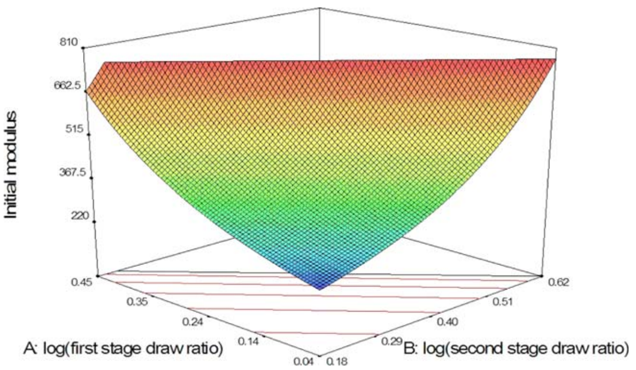

Figure 5. Response surface plot for the initial modulus; $C = 0.11$ was constant. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

shrinkage. This may have been related to the extension and alignment of the polymeric chains in the noncrystalline regions without a significant improvement in crystallinity. However, after a DR3 of 2.7 was applied, the crystallinity started to increase, along with a promotion in the orientation of the noncrystalline regions, and therefore, the shrinkage value of the drawn yarn decreased.

The initial modulus is a relatively structure-insensitive property, whereas the tenacity and shrinkage are structure-sensitive properties. In fact, the modulus of the fiber is determined by the crystalline and amorphous orientations, chemical structure of the polymer, crystal size, and the fraction of taut–tie molecules.[11,20] These parameters can affect the tenacity, but the presence of defects may control the ultimate stress. Shrinkage is caused mainly by the randomization of strained, oriented, noncrystalline chains. The amorphous orientation more likely determines the shrinkage.[21]

Tenacity is affected by every structural parameter in exactly the same manner as that of shrinkage. It should be noted that large crystal orientations seem to have a greater effect on tenacity than on shrinkage.[22] It seems that with the appropriate control of the fiber morphology during melt spinning and drawing, fibers with a high tenacity and low shrinkage can be obtained. In the applied drawing process in this research, heating the drawn fiber on the third godet would be a solution for obtaining drawn fibers with a high tenacity and modulus and a low shrinkage.

Process Optimization

The optimal conditions for obtaining the drawn yarn with the desired mechanical properties were predicted with the optimization function of the Design Expert software. In this study, the desired goals in terms of the tenacity and initial modulus were defined as *maximized,* and shrinkage was selected to be in the range. These are presented in Table VI along with their predicted and actual values.

As shown in Table VI, there was close agreement between the experimentally achieved mechanical properties and those predicted by the empirical model. According to these results, it was possible to save considerable time and effort in the estimation of desired mechanical properties for the hot multistage drawn nylon 6 fibers. Usually, obtaining a drawn yarn with a high tenacity and initial modulus and a low shrinkage is desirable for end use. So, in another optimization process, the desired goals in terms of the tenacity and initial modulus were defined as maximized, whereas the shrinkage response was selected to be minimized. Under the optimized working conditions (DR = 1.1, DR2 = 4.2, and DR3 = 1.3), the model predicted values of 6.6%, 62.79 cN/tex, and 774.77 cN/tex for shrinkage, tenacity, and initial modulus, respectively. However, the desirability factor of 0.663 for this optimization process was low. It seems that under the hot multistage drawing process, all of the final properties could not simultaneously meet the desired goals. This could have been related to reasons discussed in the last paragraph of the previous section.

CONCLUSIONS

From the results obtained in this research, we concluded that the given predictive models described the studied hot multistage drawing very well. These models could then be used to predict the tenacity, initial modulus, and shrinkage of the drawn yarn under given conditions because they were based on experimentally tested parameters. The results obtained from RSM showed that the tenacity and initial modulus were affected in the same manner by DR2. In addition, they revealed that at a low level of DR2, a critical DR3 was involved; up to that, shrinkage

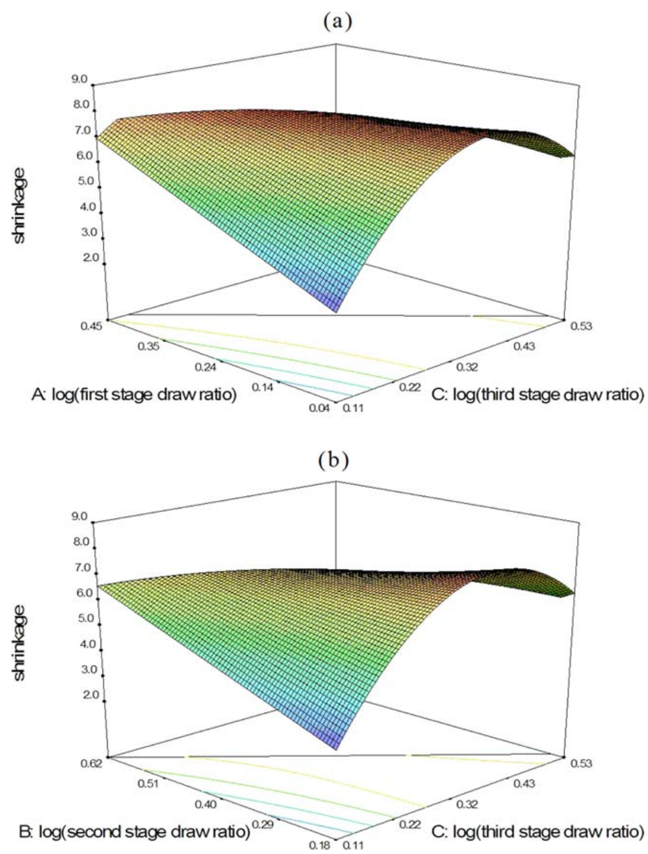

Figure 6. Response surface plot for shrinkage; values of $B = 0.18$ and $A = 0.04$ were constant for parts a and b, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table VI. Optimum Conditions Established for the Mechanical Properties of the Drawn Yarns

Experiment	Draw ratio			Desirability	Predicted		Actual	
	First stage	Second stage	Third stage		Tenacity (cN/tex)	Modulus (cN/tex)	Tenacity (cN/tex)	Modulus (cN/tex)
1	1.48	3.10	1.30	0.98	64.09	767.9	63.41	765.8
2	1.44	3.10	1.30	0.98	64.08	768.1	64.26	766.3
3	1.44	3.16	1.30	0.98	64.07	768.3	62.71	765.4
4	1.44	3.16	1.30	0.98	64.06	768.5	64.29	763.2
5	1.41	3.16	1.30	0.98	64.05	768.6	63.93	761.3

increased, and it decreased thereafter. Moreover, it was demonstrated that the optimum drawing conditions could be successfully predicted by RSM.

REFERENCES

1. Richards, A. F. In Synthetic Fibres: Nylon, Polyester, Acrylic, Polyolefin; McIntyre, J. E. Ed.; Woodhead: Cambridge, United Kingdom, **2005**.
2. Vasanthan, N. In Handbook of Textile Fiber Structure. Volume 1: Fundamentals and Manufactured Polymer Fibers; Eichhorn, S. J., Hearle, J. W. S.; Jaffe, M.; Kikutani, T. Eds.; Woodhead: Cambridge, United Kingdom, **2009**.
3. Allan, G.; Yang, R.; Fotheringham, A.; Mather, R. *J. Mater. Sci.* **2011**, *36*, 3113.
4. Yang, R. D.; Mather, R. R.; Fotheringham, A. F. *J. Mater. Sci.* **2001**, *36*, 3097.
5. Yang, R.; Mather, R. R.; Fotheringham, A. F. *J. Appl. Polym. Sci.* **2004**, *93*, 568.
6. Yang, R.; Mather, R. R.; Fotheringham, A. F. *J. Appl. Polym. Sci.* **2005**, *96*, 144.
7. Yang, R.; Mather, R. R.; Fotheringham, A. F. *J. Appl. Polym. Sci.* **2012**, *124*, 3606.
8. Prevorsek, D. C.; Harget, P. J.; Sharma, R. K.; Reimschuessel, A. C. *J Macromol Sci Phys* **1973**, *8*, 127.
9. Heuvel, H. M.; Huisman, R. *J. Appl. Polym. Sci.* **1981**, *26*, 713.
10. Gianchandani, J.; Spruiell, J. E.; Clark, E. S. *J. Appl. Polym. Sci.* **1982**, *27*, 3527.
11. Murthy, N. S.; Bray, R. G.; Correale, S. T.; Moore, R. A. F. *Polymer* **1995**, *36*, 3863.
12. Haji, A.; Semnani Rahbar, R. *Chem. Ind. Chem. Eng. Q* **2012**, *18*, 233.
13. Semnani Rahbar, R.; Mojtahedi, M. R. M. *J. Eng. Fibers Fabrics* **2011**, *6*, 7.
14. Myers, R. H.; Montgomery, D. C.; Anderson-Cook, C. M. Response Surface Methodology: Process and Product Optimization Using Designed Experiments; Wiley: Hoboken, NJ, **2009**.
15. Montgomery, D. C. Design and Analysis of Experiments; Wiley: Hoboken, NJ, **2009**.
16. Mason, R. L.; Gunst, R. F.; Hess, J. L. Statistical Design and Analysis of Experiments: with Applications to Engineering and Science; Wiley: Hoboken, NJ, **2003**.
17. Khuri, A. I.; Cornell, J. A. Response Surfaces: Designs and Analyses; Marcel Dekker: New York, **1996**.
18. Draper, N. R.; Smith, H. Applied Regression Analysis; Wiley: New York, **1998**.
19. Chapra, S.; Chapra, S. C.; Canale, R.; Canale, R. P. Numerical Methods for Engineers; McGraw-Hill: New York, **2009**.
20. Salem, D. R. Structure Formation in Polymeric Fibers; Hanser: Munich, **2000**.
21. Murthy, N. S. *Text. Res. J.* **1997**, *67*, 511.
22. Murthy, N. S.; Grubb, D. T. *J. Polym. Sci. Part B: Polym. Phys.* **2003**, *41*, 1538.