STUDY CONCERNING THE INFLUENCE OF PLASMA TREATMENTS ON POLYPROPYLENE FIBERS TENACITY

Ș. l. dr. ing. **Catalin VÎLCU,** Ş. l. dr. ing. **Liliana HRISTIAN,**
Prof. dr. ing. **Demetra Lăcrămioara BORDEIANU**

"Gheorghe Asachi" Technical University of Iaşi,
Faculty of Textiles & Leather Engineering and Industrial Management

REZUMAT. În lucrare s-a realizat un studiu privind tratarea în mediu de plasmă a fibrelor de polipropilenă, în variante diferenţiate prin parametrii de lucru ai instalaţiei, rezultând producerea unor modificări apreciabile ale tenacității fibrelor. Dintre parametrii instalaţiei Corona, care au influenţat cel mai mult procesul, s-au evidenţiat: puterea activă a instalaţiei şi numărul de cicluri parcurse de probă în zona de tratament. Astfel, studiul efectuat s-a axat pe evidenţierea modului de influenţă a celor doua variabile independente asupra proprietăţilor fibrelor de polipropilenă, precum şi determinarea unor condiţii de optimizare a acestora. În acest scop s-a utilizat programarea factorială, prin adoptarea unui program central compus rotabil, cu două variabile independente (x_1–numărul de cicluri, Nc; x_2 –puterea activă, Pa, [W] și o variabilă dependentă (y_1 – tenacitatea fibrelor, τ [cN/dtex]).

Cuvinte cheie: plasma, program factorial, fibre de polipropilenă, tenacitate, coeficient de corelație.

ABSTRACT. The work refers to a study concerning plasma treatment of polypropylene fibers in variants differing by the working parameters of the Corona installation, which result in significant modification of fiber tenacity. Among the parameters of Corona installation that influenced the process the most, one can mention the installation active power and the number of cycles traveled by sample within the treatment zone. The performed study was focused on showing off the manner in which the two independent variables influence the polypropylene fibers' properties, as well as on the determination of the conditions necessary to optimize them. With this aim in view, the factorial programming was used, by adopting a central compound rotable program with two independent variables (x_1 – number of cycles, Nc; x_2 – active power, Pa, [W] and a dependent variable (y_1- fibers tenacity, τ[cN/dtex]).

Keywords: plasma, factorial programming, polypropylene fibers, tenacity, coefficient of correlation.

1. INTRODUCTION

Plasma-surface modification is an effective and economical surface treatment technique for many materials and of growing interest in biomedical engineering corrosion (Dai, X., J., et al., 2001).

The last decade witnessed an impressing increase of the interest for diverse techniques of material surfaces preparation and modification. The basic principle of these technologies consists in the possibility to modify the surface properties without changing the characteristics of the basic material. In essence, this fact creates "new" materials with new destinations, opening inedited perspectives in solving the production and design problems (Nejneru, C., et al., 2013, Earar K., et al., 2015).

The production-related difficulties appear frequently when an already existing material is replaced by a novel one, which seams to be incompatible with processing (Manea, L.R., et al., 2013, Hristian, L., et al., 2015).

Recent methods for surface modification can deal with such problems and, at the same time, are a source of inspiration for unexpected design solutions (Popescu, I.I., et al., 1981).

Devices with plasmas generated by low-pressure electric gas discharges are widely used in industry, in applications like nitriding, ionic implantations, thin films deposition, surface corrosion. RF discharge plasmas in low-pressure gases are widely used in electronic industry to produce microchips (Chu, P.K., et al., 2002).

The treatment of polypropylene fibers in plasma medium was performed in order to establish if and how it affect their surface properties, physico-mechanical properties, chemical properties, tinctorial affinity, and also if one can improve in this way the processing and finishing of polypropylene fibers.

As these fibers belong to non-polar materials, the adhesion of inks, dyestuffs or adhesives is very low, which makes difficult operations like printing, dyeing, stratification, flocking, etc.

2. MATERIALS AND METHODS

The utilized materials are wool-type polypropylene fibers of 6 dtex/60 mm, which were previously degreased to remove the preparation applied on fibers during fabrication process. Plasma treatment of polypropylene fibers was carried out on a CORONA CG-20, ARCOTEC GmbH Rotweg installation.

The factorial programming was used by adopting a central compound rotable program with two independent variables (x_1 – number of cycles, Nc; x_2 – active power, Pa and a dependent variable (y_1 – fibers tenacity, τ [cN/dtex]).

Table 1 presents the levels with coded and real values of the independent variables.

Table 1. **Coded and real values of independent parameters**

Independent parameter \ **Code**	-1.414	-1	0	+1	+1.414
x_1 – number of cycles (Nc)	7	8	10	12	13
x_2 – active power (Pa)	240	340	620	900	1000

Experiments were carried out by maintaining constant the following parameters:

- distance between electrodes – 6 mm;
- electrode type- ceramic;
- backing electrode- isolated support;
- backing electrode speed – 10 m/min.

Table 2 presents the experimental matrix based on the variation levels of the two independent variables, with 13 experimental variants (Taloi, D., et al., 1983).

In order to avoid appearance of some systematic errors, the experimental variants were executed in a random order. Treatment effect was investigated through laboratory tests, the determined variables being dependent variables, namely: y_1 – yarns tenacity/denier strength, τ [cN/dtex]. One obtains mathematic models of interdependence between the independent variables x_1 and dependent/resulting variables y_1 by processing experimental data with CAV 1 software (Vilcu, C., 2007).

3. RESULTS AND DISCUSSIONS

Tenacity is a specific index for appreciating fiber behavior to traction. This stress is most frequently present during fiber processing and while using the products. Therefore, it is useful to establish the effect of plasma treatment on this index.

Table 3 presents the mean measured and computed values of yarns tenacity, as well as their deviations [%].

Table 2. **Experimental matrix**

Experimental variants	**Independent variables**			
	x_1 - Nc		**x_2 - Pa**	
	Code	**Real**	**Code**	**Real**
1.	-1	8	-1	340
2.	1	12	-1	340
3.	-1	8	1	900
4.	1	12	1	900
5.	-1.414	7	0	620
6.	1.414	13	0	620
7.	0	10	-1.414	240
8.	0	10	1.414	1000
9.	0	10	0	620
10.	0	10	0	620
11.	0	10	0	620
12.	0	10	0	620
13.	0	10	0	620

Table 3 The values of polypropylene fiber tenacity

Experimental variant	y_{m1} **[cN/dtex]**	y_{c1} **[cN/dtex]**	**A [%]**
1.	2.75	2.75935	0.340115
2.	2.21	2.33986	5.87616
3.	2.60	2.55935	1.56334
4.	2.46	2.53986	3.24647
5.	2.74	2.77989	1.45573
6.	2.6o	2.46953	5.01815
7.	2.58	2.47480	4.07759
8.	2.46	2.47480	0.601549
9.	2.98	3.02863	1.63178
10.	3.02	3.02863	0.285661
11.	3.04	3.02863	0.374113
12.	2.97	3.02863	1.97397
13.	3.13	3.02863	3.23876

The significance of the coefficients of the regression equation was established by means of Student test (Table 4), which imposes to satisfy the condition:

$$t_c \geq t_{tab}(\alpha, \nu) \qquad (1)$$

From Table 4 it follows that, except for b2, all the coefficients are significant, the response being given by the explicit equation of a 2nd order algebraic surface:

$$y_1 = 3,02863 - 0,109745 \cdot x_1 - 0,202021 \cdot x_1^2 - \\ -0,276998 \cdot x_2^2 + 0,1 \cdot x_1 \cdot x_2 \qquad (2)$$

Table 4. **Coefficients of regression equation and their significance**

Coefficient		Statistic t_c	Statistic $t_{tab}(\alpha,\nu)$	Significance
b0	3.028630	106.1530	$t_{tab}(0.05;4) = 2.132$	significant
b1	-0.109745	4.865560		significant
b2	-0.008710	0.386159		significant
b12	0.100000	3.134970		significant
b11	-0.202021	8.350630		significant
b22	-0.276998	11.44990		significant

The mathematic model is adequate, because it complies with the three adequacy conditions:

− adequacy condition 1:

$$F_c = 4.49504;\ F_{tab}(0.05;4;4) = 6.39;\ F_c < F_{tab} \quad (3)$$

− adequacy condition 2:

$$F_c' = 13.112;\ F_{tab}(0.05;12;12) = 2.69;\ F_c' > F_{tab} \quad (4)$$

− adequacy condition 3: all the percentage deviations are lower than 10%.

The statistic interdependence between the independent variables and the dependent/resulting variable is given by the coefficient of multiple correlation, i.e. the relation:

$$R_{y,x_1,x_2} = \sqrt{1 - \frac{\sum_{i=1}^{N}(y_{mi} - y_{ci})^2}{\sum_{i=1}^{N}(y_{mi} - \bar{y})^2}} \quad (5)$$

where: y_{mi} are the measured values of the independent variables, yci are the computed values of the dependent variable.

The significance of the coefficient of correlation is verified with Fisher test, imposing the condition:

$$F_c \geq F_{tab(\alpha,\upsilon_1,\upsilon_2)} \quad (6)$$

The computed statistics Fc is determined with the relation:

$$F_c = \frac{N-k-1}{k} \cdot \frac{R^2_{y \cdot x_1 x_2}}{1 - R^2_{y \cdot x_1 x_2}} \quad (7)$$

The statistics from the table correspond to a threshold of significance α= 0.05 and to the degrees of freedom:

$$\upsilon_1 = k, \quad \upsilon_2 = N - k - 1 \quad (8)$$

The coefficient of determination $R^2_{y \cdot x_1 x_2}$ indicates the contributions of the independent variables to the variation of dependent/resulting variable.

The strong statistic connection between the tenacity and independent variables is revealed by the coefficient of multiple correlation $R_{y,x_1,x_2} = 0.961$, significant value certified by the F test:

$$F_c = 60.53;\ F_{tab(0,05,2,10)} = 4,1;\ F_c > F_{tab} \quad (9)$$

The coefficient of multiple determination $R^2_{y \cdot x_1 x_2} = 0.9237$ shows the significant contribution of 92.37% of the two independent factors to tenacity, the influence of other factors being of only 7.63%.

In order to determine the optimum in the multifactor space, i.e. the extreme value, maximum or minimum, one has to transform the regression equation in a canonical form. Determine at first the coordinates of the new center (O'):

$$x_{1s} = -0.2843200$$

$$x_{2s} = -0.0513217,$$

where the response value is $Y_s = 3.04423$ cN/dtex.

After the rotation of α= 26.5692°, one obtains the new coordinates:

$$X_1 = 0.894395.(x_1 + 0.28432) + 0.447278.(x_2 + 0.0513217) \quad (10)$$

$$X_2 = 0.447278.(x_1 + 0.28432) - 0.894395.(x_2 + 0.0513217) \quad (11)$$

where the regression equation for the analyzed parameter takes the standard form:

$$Y - 3.04423 = -0.177016\ X12 - 0.302003\ X22 \quad (12)$$

The response surface represents an elliptic paraboloid, figure 1.

The contour responses or the lines of equal significance given in figure 2 represent a family of ellipses, the coefficients B11 and B22 having the same negative sign – the ellipses center will mark a point of maximum of the analyzed parameter.

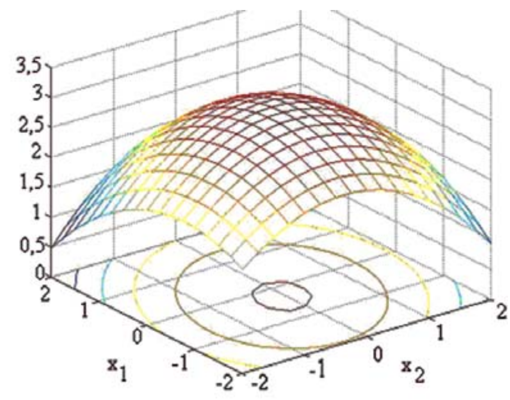

Fig. 1 Response surface expressed by the function $\tau = f(x_1, x_2)$.

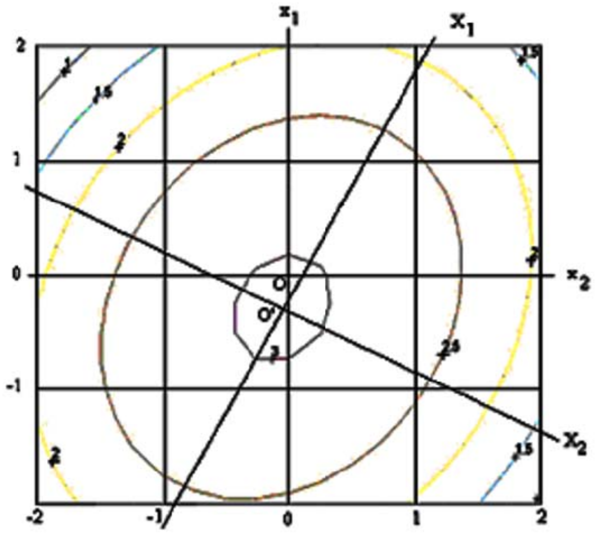

Fig. 2. Level curves for the response surface $\tau = f(x_1, x_2)$.

From the analysis of the regression equation and the equation of standard form, it follows that the tenacity of polypropylene fibers is influenced in different ways by the two independent parameters x_1 – number of the fiber sample cycles in the treatment zone, and x_2 – active power:

− the absence of the linear term b_{2x2} shows that the rated power does not have a significant influence on tenacity, a parameter that can take any value in the experimental space;

− simultaneous variation of the two parameters influences the resulting characteristic, resulting in an increase when the two variables have the same sign, and a decrease when they have different signs.

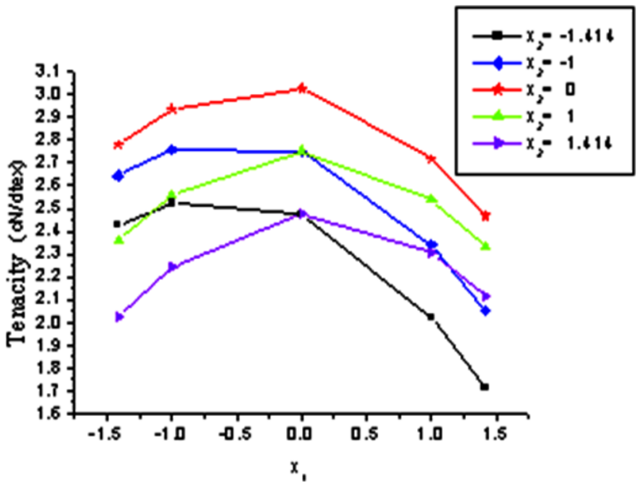

Fig. 3. Variation of fibers tenacity in terms of the number of cycles for different values of the active power.

For optimum values of the variables x_1 = 10 cycles, x_2 = 620W, the cross-linking of polypropylene chains is the dominant process which assures tenacity increase, after which, at higher powers and more cycles, a degrading process starts and the tenacity decreases.

Analyzing the influence of variable x_1 on fibers tenacity for different constant values of the variable x_2, one can notice from figure 2 that:

− for minimum active power, tenacity records an increase of only 2% within the interval [-1.414;0] and a significant decrease of about 30% within the interval [0;1.414] at a value of 1.7158 cycles, while for the maximum value of x_2 [1000 W], the characteristic analyzed for the same intervals records increases of 22% and decreases of 14.6% respectively;

− for the central value of x2, tenacity modifications in terms of x1 are slower, remaining at high values between 2.7798 cN/dtex and 2.4695 cN/dtex, with a maximum of 3.0286 cN/dtex;

− in the plane of coordinates x_1, x_2 one obtains the maximum tenacity value for the code coordinates $x_1 = 0$; $x_2 = 0$, real values x_1 = 10 cycles, x_2 = 620 W, a point that represents also the modification of the sense of analyzed parameter variation.

In figure 3, the level curve $y = 2.5$ circumscribes the area that provides the required STAS conditions for the minimum value of tenacity for wool-type polypropylene fibers. For Y = 2.5 cN/dtex, one obtains $X_1 = \pm 1.7534$ and $X_2 = \pm 1.3424$, with which one can determine the values of the coordinates x_1, namely: for x_1- code values (-1.911; 1.3685) and real values (7-12.89) cycles; for x_2 – the code values ±1.38, and the real values respectively (248-992) W.

4. CONCLUSIONS

1) Tenacity is a specific index for the estimation of fiber tensile behavior. This stress is most frequently present during fiber processes, as well as during fibers utilization, therefore it is useful to establish the plasma treatment effect on this index.

2) The equation of regression contains the both quadratic terms with significant importance of the response, which decreases over the entire experimental surface regardless the sense of independent variables variation.

3) From graphical representations of the level curves, one can delimitate the zones and the limits of independent variables' variations, in order to provide certain response values.

4) The apex of the elliptic paraboloid described by the canonic equation represents a point of maximum, τ_{max} = 3.04423 cN/dtex, of coordinates x_{1s} = = –0.284320, and x_{2s} = -0.0513217 code values, and respectively x_{1s}= 9.44 cycles and x_{2s} = 605.6 W, real values, this being the quested optimum.

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Despre autori

Şef lucr. dr. ing. **Cătălin VÎLCU**
Universitatea Tehnică „Gheorghe Asachi“ din Iaşi

Absolvent a Universităţii Tehnice „Gheorghe Asachi“ din Iaşi, Facultatea de Textile - Pielărie – 1997, doctor inginer din anul 2007, şef lucrări la Facultatea de Textile Pielărie şi Management Industrial – 2009. Domenii de competenţă: optimizarea proceselor tehnologice de filare şi ţesere, proiectare tehnologica asistata de calculator, limbaje de programare – Turbo Pascal, C++.

Şef lucr. dr. ing. **Liliana HRISTIAN**
Universitatea Tehnică „Gheorghe Asachi“ din Iaşi

Absolventă a Universităţii Tehnice „Gheorghe Asachi“ din Iaşi, Facultatea de Textile - Pielărie – 1997, doctor inginer din anul 2008, şef lucrări la Facultatea de Textile Pielărie şi Management Industrial – 2012. Domenii de competenţă: testarea şi controlul calităţii produselor textile, metode de prelucrare a datelor statistice.

Prof.. dr. ing. **Demetra Lăcrămioara BORDEIANU**
Universitatea Tehnică „Gheorghe Asachi“ din Iaşi

Absolventă a Universităţii Tehnice „Gheorghe Asachi“ din Iaşi, Facultatea de Textile – 1976, doctor inginer din anul 1996, profesor la Facultatea de Textile Pielărie şi Management Industrial – 2001. Domenii de competenţă: fibre textile, tehnologii de filare, filarea neconvenţională.