FROM SPYDER WEB TO ARTIFICIAL NANOFIBER WEB

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REZUMAT. Scopul acestei cercetări a fost de a crea un câmp electrostatic necesar pentru procesul de electrofilare. Pentru aceasta, o sursă în comutație, de înalta tensiune, reglabilă între 10 kV si 30 kV precum și un sistem electronic pentru controlul distanței dintre vârful acului și colector între 5 cm și 25 cm, cu pas de 1 mm, au fost realizate. De asemenea, a fost proiectată o pompă pentru soluția polimerică, cu debite între 0.6 ml/h și 3 ml/h. Pentru testarea câmpului a fost utilizată o configurație de electrofilare și soluții de PVA cu apă. În urma măsuratorilor asupra câmpului s-a dezvoltat un model matematic, ce pune în evidență legătura dintre intensitatea câmpului și cele două variabile independente utilizate, tensiunea aplicată și distanța dintre vârful acului și colector.

Cuvinte cheie: electrofilare, câmp electrostatic, tensiune înaltă, modul de alimentare cu energie în comutație, nanofibre.

ABSTRACT. The aim of this research is to create an electrostatic field for the electrospinning process. For this, a high voltage switch mode power supply (SMPS) with an adjustable output between 10 kV and 30 kV and a system for controlling the distance between the nozzle tip and the collector with a range from 5 cm to 25 cm were developed. Also, previously, a fluid pump with an adjustable flow rate between 0.6 ml/h and 3 ml/h was made. For testing the field, it was used an electrospinning setup with PVA/water solutions. Based on measurements performed on the created electrostatic field, it was designed a central composite design quadratic model. The model highlights the dependence of the electrical field intensity on the two selected independent variables, applied voltage and the distance between the nozzle tip and the collector.

Keywords: electrospinning, electrostatic field, high voltage, switch mode power supply, nanofibers.

1. INTRODUCTION

The high voltage electrostatic field is a very important part of the electrospinning setup. Two of the process parameters that characterise it are the applied voltage and the distance between the nozzle tip and the collector.

Applied voltage is a critical factor for the electrospinning process because only applied voltages higher than a threshold voltage can produce the phenomenon. However, the connection between the applied voltages and the diameter of the obtained nanofibers is controversial. Reneker and Chun have shown that in the case of PEO, the effect of the electric field on the diameter of the fibers is minimal [1]. Zhang et al, using PVA/water solution, suggested that higher voltages mean higher fiber diameter [2]. Yuan et al found that higher voltages, favour the narrowing of fiber diameter, in the case of PSF/DMAC/acetone solutions [3]. It has also been demonstrated that the probability of beads formation is greater for higher voltages [4], [5], [6]. Applied voltage influences fiber diameter, but the level of significance depends on the polymer solution concentration and the distance between the nozzle tip and the collector [7].

It has been demonstrated that the distance between the nozzle tip and the collector affects the fiber diameter and morphology [8]. If the distance is too short, the fiber will not solidify before reaching the collector. If the distance is too long, bead fiber will appear. Yuan et al demonstrated a little longer distance results in thinner fiber diameter.

2. MATERIALS AND METHODS

The experimental setup (Figure 1) consists of a fluid pump, a high voltage SMPS, a grounded collector plate, a field isolation chamber and a system for controlling the distance between the nozzle tip and the collector [9], [10].

Syringes of 2.5 ml polypropylene Luer Slip with an internal diameter of 8.66 mm were used. The needles were type 23G (0.337 mm internal diameter) and 25.4 mm length, all metal (stainless steel) hypodermic type.

The polymer used was PVA with a molecular weight of 38000 Da. By stirring PVA in water for two hours at 70˚C, 10% wt PVA/water solutions were obtained. Figure 2 shows the PVA fibers deposited on the collector.

In order to make the central composite design quadratic model, 13 experiments were conducted. Each experiment lasted for 10 minutes. The fibers were deposited on a blue foil applied on the grounded collector plate. The procedure for each experiment was:

- set the corresponding distance between the nozzle tip and collector;
- set the fluid pump feed rate to 0.6 ml/h;
- set the corresponding high voltage by reading it on a multimeter using a 1000:1 voltage divider probe (also developed by the team – Figure 3).

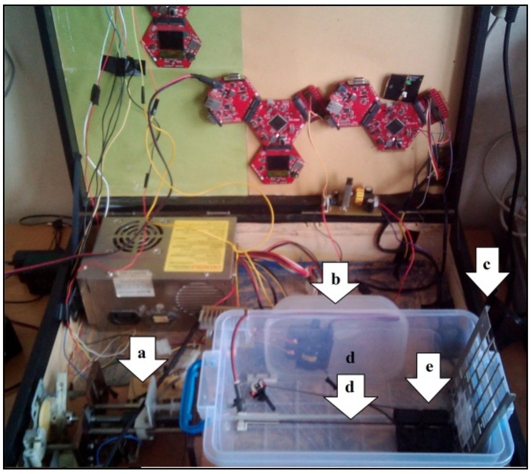

Fig. 1. Experimental equipment
a – fluid pump; *b* – high voltage SMPS; *c* – grounded collector; *d* – field isolation chamber; *e* – system for controlling the distance between the nozzle tip and collector.

Fig. 2. PVA nanofibers deposited on the collector (U = 17.7 kV, d = 10 cm).

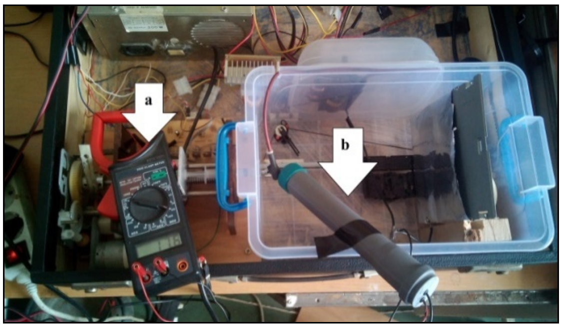

Fig. 3 – Reading the high voltage:
a – multimeter; *b* – 1000:1 voltage divider probe.

3. RESULTS AND DISCUSSIONS

The electrical field intensity Y according to the relation described by the proposed mathematical model is presented in table 1. It highlights the dependence of the electrical field intensity on the two selected independent variables, applied voltage (X_1) and the distance between the nozzle tip and collector (X_2).

Table 1. **Experimental matrix for the two independent variables**

No	X_1			X_2		$Y = X_1/X_2$ [kV/cm]
	X_{1C}	X_{1R}[kV]	X_{1D}[kV]	X_{2C}	X_{2R} [cm]	
1	-1	12.3	12.4	-1	12.4	0.99
2	1	22.9	22.6	-1	12.4	1.85
3	-1	12.3	12.4	1	22.6	0.54
4	1	22.8	22.6	1	22.6	1.01
5	-1.414	10.1	10	0	17.5	0.58
6	1.414	24.9	25	0	17.5	1.42
7	0	17.7	17.5	-1.414	10	1.77
8	0	17.5	17.5	1.414	25	0.7
9	0	17.6	17.5	0	17.5	1.01
10	0	17.5	17.5	0	17.5	1
11	0	17.5	17.5	0	17.5	1
12	0	17.5	17.5	0	17.5	1
13	0	17.5	17.5	0	17.5	1

*X_1 – applied voltage (X_{1C}-coded, X_{1R} – real, X_{1D} –designed) and X_2 – distance between the nozzle tip and collector (X_{2C}-coded, X_{2R}-real).

From the analysis of the mathematical model, the regression equation (1) resulted:

$$Y = 1.002 + 0.315 \cdot X_1 - 0.35 \cdot X_2 - 0.006 \cdot X_1^2 + 0.111 \cdot X_2^2 - 0.098 \cdot X_1 \cdot X_2 \quad (1)$$

Significance of the regression equation coefficients for *Y* – electrical field intensity from processing the experimental data was established using Student test (table value: 2.132 $\alpha = 0.95$ $\nu = 4$), for the two independent variables X_1 (applied voltage) and X_2 (distance between the nozzle tip and collector) are presented in table 2.

From Table 1, all the coefficients of the mathematical model are significant and are found in the regression equation. The correlation coefficient is: 0.9975

Figure 4 illustrates the three-dimensional graphical representation of the electrical field intensity variation.

Variation of *Y* as a function of the independent variables X_1 and X_2 is presented in figures 5 and 6.

The response surface, resulted from the obtained mathematical model processing, is an elliptical paraboloid whose projection in the X_1OX_2 plane represents a family of ellipses, figure 7.

In order to see the nanofibers looking like spyder web, was conducted an experiment in which a multi window collector plate was used (figure 8).

Table 2 **Coefficients of the regression equation and their significance**

Coefficient	Value	Student test value	Coefficient significance
b0	1.002	501.135	Significant
b1	0.315	199.046	Significant
b2	-0.35	-221.595	Significant
b11	-0.006	-3.657	Significant
b22	0.111	65.608	Significant
b12	-0.098	-46.603	Significant

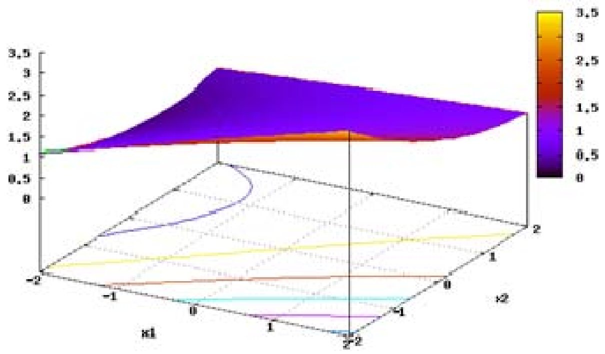

Fig. 4. Variation of the electrical field intensity $Y = 1.002 + 0.315X_1 - 0.35X_2 - 0.06\,X_1^2 + 0.111\,X_2^2 - 0.098\,X_1X_2$

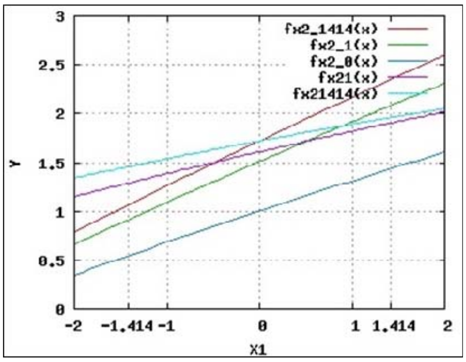

Fig. 5. Variation of electrical field intensity (*Y*) as a function of applied voltage (X_1) keeping the distance between the nozzle tip and collector (X_2) constant.

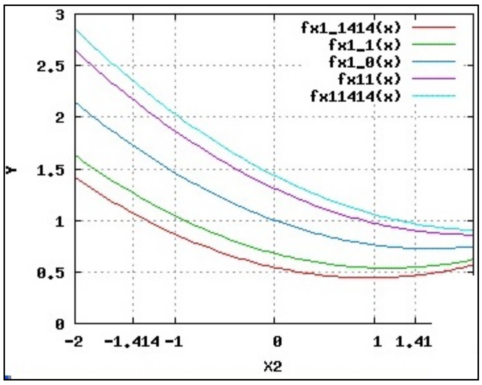

Fig. 6. Variation of electrical field intensity (*Y*) as a function of distance between the nozzle tip and collector (X2) keeping applied voltage (X1) constant

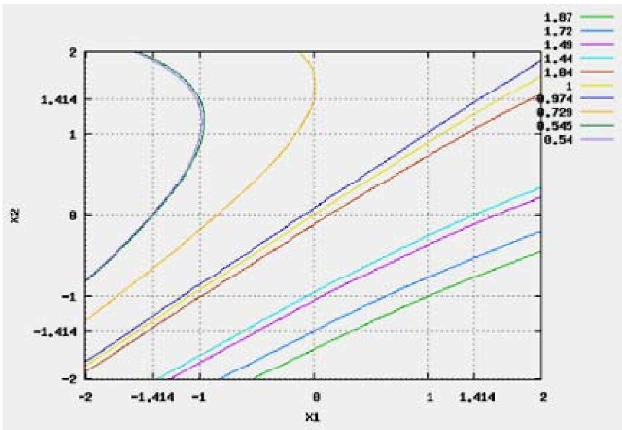

Fig. 7. Graphical representation of the level curves for the response surface $Y = f(X_1, X_2)$.

Fig. 8. PVA nanofibers looking like spider web ($U = 20$ kV, $d = 10$ cm).

4. CONCLUSIONS

A high voltage switch mode power supply (SMPS) with an adjustable output between 10 kV DC and 30 kV DC, for producing the high voltage electrostatic field necessary for the electrospinning process, and an electronic system for controlling the distance between the nozzle tip and collector (5–25cm, 1mm step), were developed.

The two systems were tested in an electrospinning setup using PVA/water solutions. Based on the measurements, a mathematical model was also developed.

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