

RESEARCH ON THE BEHAVIOR OF SPINNABLE POLYMER SOLUTIONS IN A HIGH VOLTAGE ELECTROSTATIC FIELD

CIOBOTARU Victor¹, AVRAM Dorin¹, PANTILIMONESCU Florin²

¹ “Gheorghe Asachi” Technical University of Iasi, Romania, Faculty of Textiles & Leather Engineering and Industrial Management, Str. Prof. Dr. Doc. Dimitrie Mangeron, Nr. 29, 700050 Iasi, Romania,
E-mail: decanat@tex.tuiasi.ro

² “Gheorghe Asachi” Technical University of Iasi, Romania, Faculty of Automatic Control and Computer Engineering, Str. Prof. Dr. Doc. Dimitrie Mangeron, Nr. 27, 700050 Iasi, Romania,
E-mail: decanat@ac.tuiasi.ro

Corresponding author: Ciobotaru, Victor, E-mail: victor_clk@yahoo.com

Abstract: Working parameters are very important to understand the nature of electrospinning. Each of these parameters can affect the fibers morphologies and by proper control of these parameters one can fabricate electrospun fibers with desired morphologies and diameters. Jet charge density is known as one of the important parameters affecting the fiber diameter. To investigate the effect of the applied voltage on jet charge density, one must investigate its effects on the jet electric current and flow rate. There are some difficulties in measuring the jet flow rate. In this work, one method is used to calculate the electrospinning jet flow rate. For exemplification, by using a polyethylene oxide (PEO) solution in water, effects of applied voltage and feed rate on jet charge density is investigated. The jet flow rate is calculated applying different voltages, at different constant feed rates. The jet flow rate is independent of the feed rate and dependent on the applied voltage. By defining an equation for dependence of jet flow rate on current, a relation between jet volume charge density and jet current can be defined. By measuring the electrospun fiber diameter, surface charge density of the jet can also be estimated. On increasing the voltage, the jet volume charge density decreases whereas the jet surface charge density remains unchanged.

Key words: electrospinning, nanofiber, jet flow rate, jet charge density, polyethylene oxide

1. INTRODUCTION

Electrospinning of polymer solutions is a successful method in producing continuous fibers with nano diameters.

Working parameters are very important to understand not only the nature of electrospinning but also the conversion of polymer solutions into nanofibers through electrospinning. These parameters can be broadly divided into three parts such as solution parameters, process parameters, and ambiental parameters. Each of these parameters can affect the fibers morphologies and by proper control of these parameters one can fabricate electrospun fibers with desired morphologies and diameters.

There are many reports on the effects of the process parameters, such as applied voltage, on electrospun fiber diameter, but they are inconsistent and sometimes antithetical [1],[2],[3],[4]. For example, it was demonstrated that there is not much effect of electric field on the diameter of electrospun polyethylene oxide (PEO) nanofibers [5]. Several groups suggested that higher voltages facilitated the formation of large diameter fiber. For example, there were investigated the effects of voltage on morphologies and fiber diameters distribution with poly (vinyl alcohol) (PVA)/water solutions as model [6]. Several groups suggested that higher voltages can increase the electrostatic repulsive force on the charged jet, favoring the narrowing of fiber diameter. For example, there were investigated the effects of voltage on morphologies and fiber alignment with polysulfone (PSF)/DMAC/acetone as model [7]. In addition to those phenomena, some groups also demonstrated that higher voltage offers the greater probability of beads formation [8][9][10]. Thus, one can say that

voltage does influence fiber diameter, but the level of significances varies with the polymer solution concentration and with the distance between the needle tip and the collector [11].

On the other hand, the jet charge density is known as one of the important parameters affecting the fiber diameter [12][13]. It is shown that the fiber diameter decreases when the charge density increases [2].

According to the conservation of mass and conservation of electric charge, the following equation can be written for an electrospinning jet [12][14]:

$$\frac{I}{Q_j} = \frac{2\sigma}{r} \quad (1)$$

The ratio of I to Q_j is known as the jet volume charge density (ρ). It is possible to measure the jet electric current during the electrospinning process through putting an electrical resistance in series between the collector and the ground [14][15].

In the work reported here, by measuring the jet current and jet flow rate, the jet charge density is calculated. By using PEO solution in water, effects of applied voltage and feed rate on jet charge density is investigated.

2. EXPERIMENTAL

PEO powder with a molecular weight of 4×10^5 g/mol was dissolved in distilled water at ambient temperature for about 12 h. The concentrations were 5, 7, and 10 wt %.

The electrospinning device was assembled in our laboratory. A syringe with a stainless steel needle was used. The needle dimensions were internal diameter of 0.2 mm and length of 3 cm. The needle was connected to the positive electrode of a high voltage power supply. The high voltage power supply consisted of a flyback transformer salvaged from a cathode ray tube monitor and modified for this purpose. The modified transformer could generate output voltages between 10 and 30 kV. The collector was a metal plate connected to ground. The solution was fed at a constant volumetric flow rate using a syringe pump. The syringe pump was designed in our laboratory and consisted of a 24 V Mabuchi C2162-60006 DC motor coupled to a 1:3025 gear, an optical quadrature encoder feedback and an Infineon Hexagon application kit for control. The distance between the needle tip and collector was 15 cm. The jet current was measured as a voltage drop across a 10 M Ω resistor placed in series between the needle and the ground. The voltage drop was sampled with a digital data acquisition with a sampling time of 10 ms. Solutions of 5 wt %, 7 wt % and 10 wt % PEO in water were used at room temperature, with feed rates of 0.5, 1, 1.5, 2, 2.5, 3 mL/h while applying different voltages between 10 and 30 kV. The morphology of the electrospun fibres can be observed using SEM. For each sample, the average diameter of the individual fibers can be measured from multiple magnified SEM images. The result for each sample can be reported as an average value and confidence interval, from several hundred measurements.

3. RESULTS AND DISCUSSIONS

To understand how to measure the jet flow rate, first one must be familiar with the jet regimes and their electrical current curves. In previous works [1], it has been shown that in a horizontally lain electrospinning set up, at a constant feed rate and different applied voltages, two jet regimes can be observed; they are stable jet and fluctuating jet regimes.

A solution of 10 wt % PEO in water was used at a constant feed rate of 0.5 ml/h, while applying different voltages between 10 and 30 kV, and the jet current was measured. Fig. 1 shows a model for jet current vs. time at different voltages. At the voltage of 15.5 kV and above, the process is in the fluctuating jet regime.

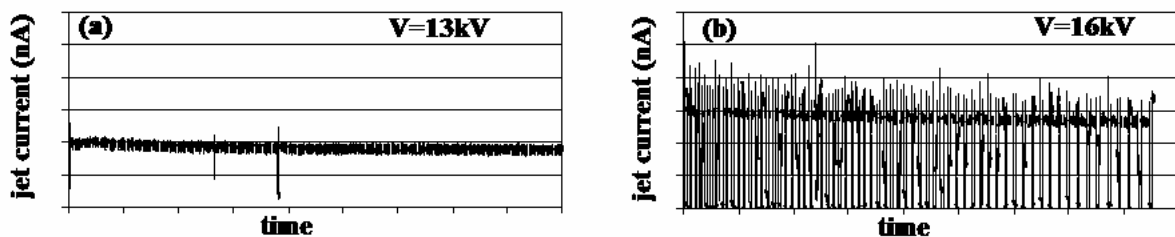


Fig. 1: Model for jet current vs. time at different applied voltages.

(a) Model for jet current vs. time for stable jet regime (b) Model for jet current vs. time for fluctuating jet regime

In the stable jet regime the jet flow rate is less than the syringe pump feed rate, so not all the solution transferred by the pump to the needle tip is carried away by the jet to the collector. In this regime the resulted jet is continuous.

In the fluctuating jet regime the jet flow rate is more than the syringe pump feed rate and the whole solution transferred by the pump to the needle tip is carried away by the jet to the collector. In this regime the resulted jet is not continuous.

Between this two regimes there is the so called quasi stable point, in which the jet flow rate is equal to the syringe pump feed rate.

A solution of 10 wt % PEO in water was used at constant feed rates of 0.5, 1, 1.5, 2, 2.5 and 3 ml/h, by applying different voltages between 10 – 30 kV. In each experiment, by increasing the voltage and investigating the current graphs, one can identify the quasi stable points [16].

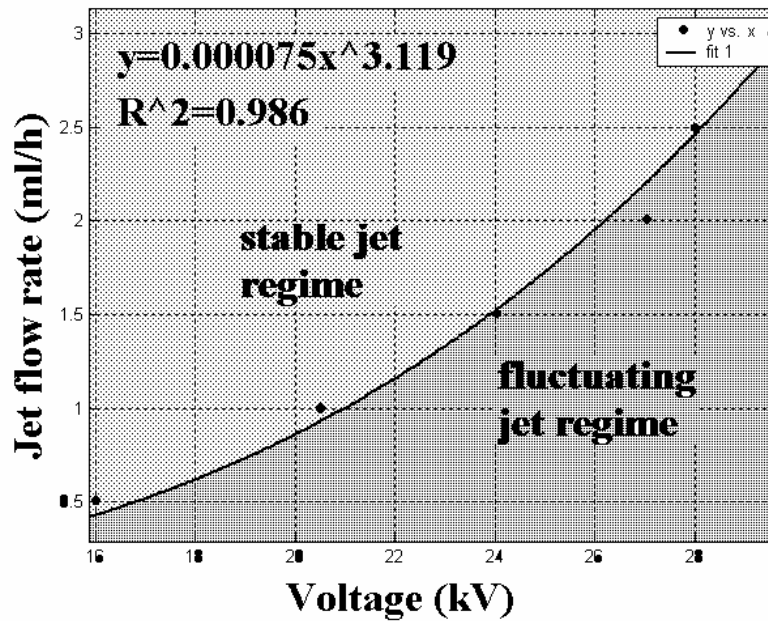


Fig. 2 : Jet flow rate as a function of the applied voltage at different feed rates. The points on the graph represent the quasi stable points. The curve shows the power-law fitting to the data based on the least squares method. The fitted equation is shown.

Fig. 2 shows power-law dependence between the jet feed rate and the applied voltage as:

$$Q_j = 0.000075V^{3.119} \quad (2)$$

If one cannot measure the voltage precisely, then a power-law dependence between the jet feed rate and the current must be taken into account, from the calculations above, as in equation (3):

$$Q_j = f(I) \quad (3)$$

Volume charge density is usually defined as:

$$\rho = \frac{I}{Q_j} \quad (4)$$

By calculating the volume charge density and measuring the jet diameter, the surface charge density can also be calculated:

$$\sigma = \frac{\rho r}{2} \quad (5)$$

The jet diameter was estimated by the following equation [15]:

$$r = r_f C^{-0.5} \quad (6)$$

where r_f is the electrospun fiber diameter, and C is the weight fraction of the polymer in the solution.

Fig. 3 shows the applied voltage, jet current, fiber diameter, jet surface and volume charge density, for a solution of 10 wt % PEO in water.

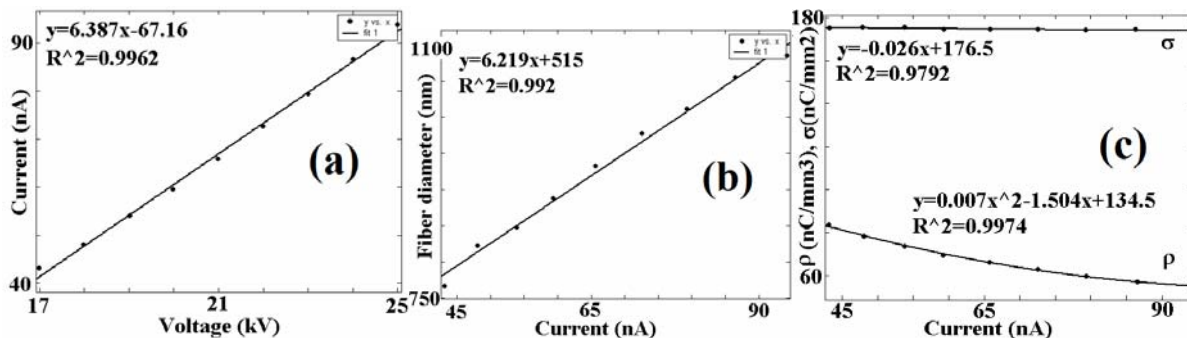


Fig. 3 : The measured and calculated parameters for a 10 wt % solution of PEO in water.
 (a) Jet current as a function of the applied voltage (b) Fiber diameter as a function of jet current
 (c) Volume and surface charge density as a function of jet current

4. CONCLUSIONS

Using polyethylene oxide solutions in water at different constant feed rates, the jet electrical current is investigated in the real time. Using the real time current graphs, the jet flow is calculated and its variations against applied voltage and jet current are investigated. The jet flow rate is independent of the feed rate and the volume charge increased when increasing the voltage. One equation is defined for dependence of the jet flow rate and the volume charge density on the jet current. Volume charge density of the jet decreased when increasing the applied voltage.

REFERENCES

- [1] Fallahi D., M. Rafizadeh, N. Mohammadi and B. Vahidi (2008), *Polym Int* 57, 1363.
- [2] Fridrikh S. V., H.H. Yu, M.P. Brenner and G.C. Rutledge (2003), *Phys Rev Lett* 90, 1445021.
- [3] Li L. and Y.L. Hsieh (2005), *Polymer* 46, 5133.
- [4] Megelski S., J.S. Stephens, D.B. Chase, J.F. Rabolt (2002), *Macromolecules* 35, 8456.
- [5] Reneker DH, Chun I (1996) Nanometre diameter fibres of polymer, produced by electrospinning. *Nanotechnology* 7(3):216–223. doi:10.1088/0957-4484/7/3/009
- [6] Zhang C, Yuan X, Wu L, Han Y, Sheng J (2005) Study on morphology of electrospun poly(vinyl alcohol) mats. *Eur Polym J* 41(3):423–432. doi:10.1016/j.eurpolymj.2004.10.027
- [7] Yuan X, Zhang Y, Dong C, Sheng J (2004) Morphology of ultrafine polysulfone fibers prepared by electrospinning. *Polym Int* 53(11):1704–1710. doi:10.1002/pi.1538
- [8] Buchko CJ, Chen LC, Shen Y, Martin DC (1999) Processing and microstructural characterization of porous biocompatible protein polymer thin films. *Polymer* 40(26):7397–7407. doi:10.1016/S0032-3861(98)00866-0
- [9] Deitzel JM, Kleinmeyer J, Harris D, Beck Tan NC (2001) The effect of processing variables on the morphology of electrospun nanofibers and textiles. *Polymer* 42(1):261–272. doi:10.1016/S0032-3861(00)00250-0
- [10] Demir MM, Yilgor I, Yilgor E, Erman B (2002) Electrospinning of polyurethane fibers. *Polymer* 43(11):3303–3309. doi:10.1016/S0032-3861(02)00136-2
- [11] Yördem OS, Papila M, Mencilog˘lu YZ (2008) Effects of electrospinning parameters on polyacrylonitrile nanofiber diameter: An investigation by response surface methodology. *Mater Des* 29(1):34–44. doi:10.1016/j.matdes.2006.12.013
- [12] Shin Y. M., M.M. Hohman, M.P. Brenner and G.C. Rutledge (2001), *Polymer* 42, 9955.
- [13] Thompson C. J., G.G. Chase, A.L. Yarin and D.H. Reneker (2007), *Polymer* 48, 6913 .
- [15] Halgeson M. E. and N.J. Wagner (2007), *AIChE* 53, 51.
- [14] He J. H., L. Xu, Y. Wu and Y. Liu, (2007) *Polym Int* 56, 1323.
- [16] Rafizadeh et al, 2008