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Minimization of The Needleless Electrospun Nanofiber Morphology Using Multi-Optimization Method

Deogratias Nurwaha[1,*], Xinhou Wang[2, 3]
[1]*Institute on Applied Pedagogy, Department of Physics and Technology, University of Burundi, Bujumbura, CO/IPA, BP 5223.*
[2] *Donghua University, College of Textile, Shanghai 201620, China*.
[3] *Key Laboratory of Science & Technology of Eco-Textile, Ministry of Education, China*

Abstract
The study describes a new electrospinning method to produce nanofibers. Two straight and parallel copper wire electrodes mounted on a rotating metal spindle was explored. Polyethylene oxide (PEO) solution was used. Spinning distance, applied voltage, wire electrodes diameter and rotation speed were considered as processing parameters. Mean Nanofiber diameter (MFD) and nanofiber diameter standard deviation (FSD) have been measured and recorded. Using gene expression programming (GEP) technique nonlinear mathematical functions were derived based on the processing parameters. The multiple optimization procedure was performed to determine the optimum levels of the five processing parameters leading to the desired minimized MFD and FSD and optimal conditions were found.

Keywords: *Nanofiber; rotary wires electrospinning; GEP.*

1. Introduction
Electrospinning is a process by which nanofibers can be produced through spinning under the driving of an electric potential. Due to their extremely high surface to weight ratio, nanofibers exhibit special properties that have opened up a wide range of potential applications. Nanofibers can be used in filtration, medical applications like drug delivery, tissue engineering and wound dressing [1], nano-electronics [2], nano-composites [3], and functional textiles [4]. In recent years, nanofibers are used in solar energy applications like solar cells batteries [5]. Conductive nanofibers are expected to be used in the fabrication of tiny electronic devices or machines such as Schottky junctions, sensors and actuators. Due to the well-known fact that the rate of electrochemical reactions is proportional to the surface area of the electrode, conductive nanofibers membranes are also quite suitable for using as porous electrode in developing high performance battery [6,7].

Fiber diameter is the main parameter for quality control of electrospun fibers. This property depends on many different factors including processing variables, polymer solution properties and ambient parameters. The problem is that there is a high degree of interaction between processing parameters. Therefore, optimal processing conditions cannot be determined easily. Traditional prediction models [8-13] and only few publications dealing with the optimization methods [14, 15] have been used to determine the effects of material and operating parameters on electrospun fiber morphology.

This study describes the application of multi-optimization method in electrospinning engineering as well as to use these approaches for optimizing processing conditions. A multi-objective optimization method based on gene algorithm (GA) has been proposed for the design and control of electrospinning process. The processing parameters were used as design variables and were mathematically related with the electrospun fiber properties (fiber diameter and its distribution) using gene expression programming (GEP) technique.

* Corresponding Author
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Nonlinear mathematical functions were derived based on the processing parameters. Afterward, using a multi-objective optimization technique based on gene algorithm, optimal conditions were found in such a way that, mean fiber diameter and its distribution to be minimized.

2. Experimental

2.1. Materials

Polyethylene oxide (PEO), with an average molecular weight (Mw) of 1,000,000, was used in this work. Different solution concentrations (4, 6, 8, 9, 10 wt %) concentrations were prepared by dissolving the PEO in distilled water and each solution was stirred until homogenization. The high voltage power supply FI 80 was used.

2.2. Electrospinning method

Figure 1 shows the setup of the method. In this process, two straight and parallel copper wire electrodes mounted on a rotating metal spindle which is connected to a belt drive that is powered by a DC motor to allow for variable speed control. As the spindle rotates along with the two copper wires, the wires are drawn through the polymer solution bath. The metal spindle is connected to a high voltage power supply (positive electrode) and the collector plate is connected to ground. As the wires move through the polymer solution bath, solution is entrained on the wires, resulting in a thin film of solution coating the wires. The forces such as gravity, surface tension, viscosity and inertia acting on polymer solution determine the amount of the solution entrained on the wire. Due to Plateau-Rayleigh instability, the coating breaks up into individual droplets of charged polymer solution on the wires. At sufficiently high local electric field, the individual drops deform and jets are produced from the droplets, giving rise to a form of free surface electrospinning. As the wires rotate, electrospinning continues to occur until the supply of polymer solution is depleted.

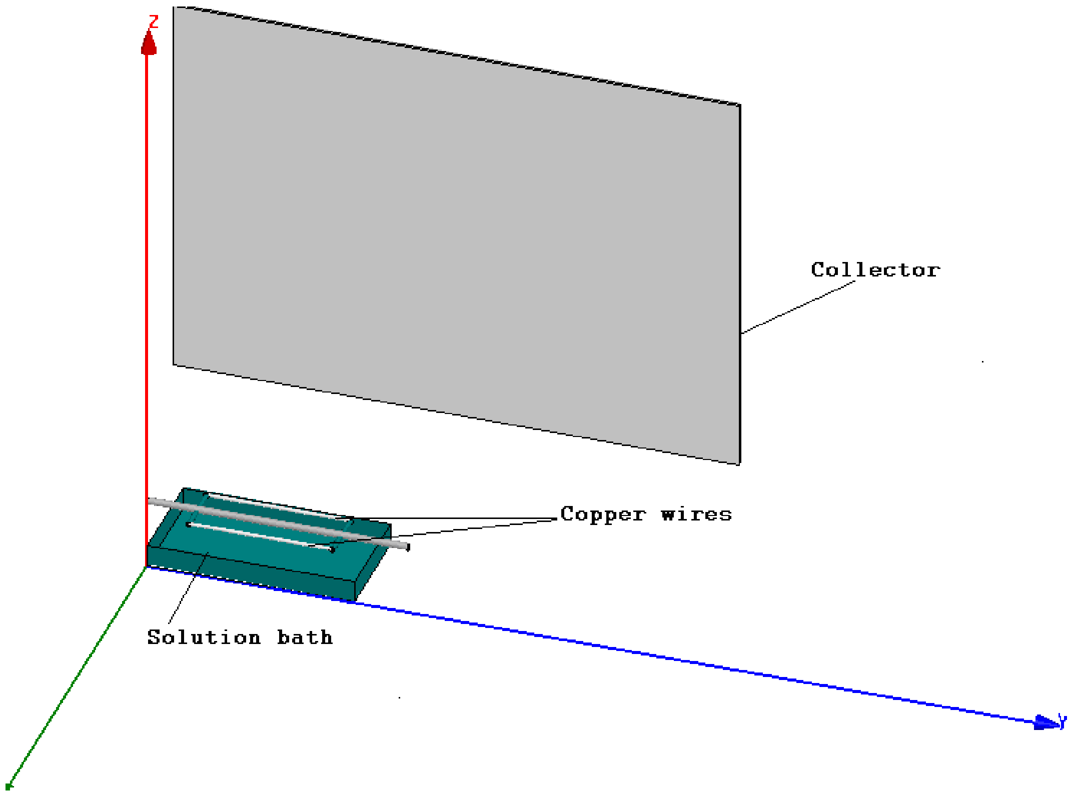

Figure 1. Two straight wires electrospinning setup.

2.3. Experimental design

Spinning distance (D= 40, 50, 60 cm, applied voltage (V=50, 60, 70 kV), wire electrodes diameter (WD= 0.37, 0.43, 0.55 mm), rotation speed (RS=6, 9, 12 rpm) were considered. Fractional factorial design, six factors at three levels (3^6-3=27 runs), was used. All experiments have been carried out at temperature of T=27.5±1.5 °C and under normal atmospheric pressure. Relative humidity varied between 20 % - 55% during experiment. Each experiment process was run for twenty-thirty minutes.

2.4. Characterization of nanofibers

Fiber formation and morphology of the electrospun PEO fibers were determined using a scanning electron microscope (SEM). Samples were cut to obtain the SEM images Fiber diameters were measured with digital image processing software Image J [16] (National Institutes of Health, USA). This program measures the number of pixels and scales the length according to the calibration provided by the user. First the scale was set. Then, pixels between two edges of a fiber perpendicular to the fiber axis were counted. Each fiber diameter was measured at the location where the fiber was identified as a single fiber. There may be up to half a pixel error in both directions which should turn out up to 1-pixel error in measuring fiber diameter. The number of the pixels was converted to nanometers (nm) using the scale and the resulting diameters were recorded. Diameters of fibers of each SEM image were measured and average of fiber diameters (MFD) along with standard deviation of fiber diameters (FSD) values were then calculated.

2.5. Decision variables

Table 1 shows the decision variables. Five processing parameters including wire diameter(x_1) applied voltage(x_2), spinning distance(x_3), solution concentration(x_4) and rotating speed(x_5) and were considered as design variables in the optimization process. The experimental results were expressed in terms of mean nanofiber diameter (MFD) ranged between 267 nm to 704 nm and standard deviation of fiber diameter (FSD) ranged between 44 nm to 218 nm.

Table 1. Design variables description.

Design variables	Description of design variables	Units	Ranges
x_1	Wire diameter	mm	0.37-0.55
x_2	Voltage	kV	40-60
x_3	Spinning distance	cm	50-70
x_4	Polymer solution concentration	wt%	6-10
x_5	Rotation rate	rpm	6-12

3. Objective functions

The mathematical functions representing the relationships between processing parameters and both mean of fiber diameter (MFD) and standard deviation of fiber diameters (FSD) were derived by gene expression programming (GEP) model and are expressed by equations (1) and (2) respectively. These functions were generated to visualize the interactive relationship between the involved parameters and were taken as objective functions.

$$MFD(nm)=(((93.176954/WD)+(1.7077874\times(D+V))+((-421.64372)/D)-10.387579) +(-5.0039359\times C)+(((V/WD)+WD)/(WD\times sqrt(RS)))) \quad (1)$$

$$FSD(nm)=(3.0249211\times(C(WD\times V)))+(C(WD\times(D+(5461.1092))))+(2.462077\times V)+(28964.329/RS))-D+2561.2366+((-7387.4802)\times Sqrt(WD))+((D+68246.209+V+(-97154.841))/RS) \quad (2)$$

4. Optimization procedure

The population type was defined as double vector; initial population was <75x5 double>. Two crossover points and a mutation rate of 0.01 were chosen what has been identified as a reasonable default setting in other multi-optimization problems. The multi-objective optimization model has been defined using the expressions in equations 1 and 2 generated by GEP. Thus, the following multi-component objective function, which is the fitness function for the GA, was defined using Matlab M-file where

$$f(1)=(((93.176954/x_1)+(1.7077874\times(x_3+x_2))+(-421.64372)/x_3)-10.387579)$$
$$+(((-5.0039359)\times x_4)+(((x_2/x_1)+x_1)/(x_1\times \mathrm{sqrt}(x_5)))) \quad \text{(for MFD)} \quad (3)$$

$$f(2)=(3.0249211\times(x_4-(x_1\times x_2)))+(x_4-(x_1\times(x_3+(-5461.1092))))+((2.462077\times x_2)$$
$$+(28964.329/x_5))-x_3+2561.2366+((-7387.4802)\times sqrt(x_1))+((x_3+68246.209+x_2$$
$$+(-97154.841))/x_5) \quad \text{(for FSD)} \quad (4)$$

where *f(1)* and *f(2)* are objective functions.

The goal is to minimize *f(1)* and *f(2)*for

$0.33 \le x_1 \le 0.55$ $40 \le x_2 \le 60$ $50 \le x_3 \le 70$ $6 \le x_4 \le 10$ $6 \le x_5 \le 12$

where x_1, x_2, x_3, x_4 and x_5 are processing parameters and act as decision variables for the fitness function.

5. Results and Discussion

Gamultiobj implements the genetic algorithm (GA) at the command line to minimize a multi-component objective function. The multiple optimization procedure was performed to determine the optimum levels of the five processing parameters (design variables) leading to the desired minimized MFD and FSD. Table 2 shows the comparison of optimization results with the experimental ones. From Table 2, one can see that the optimization method has provided a smaller minimum range than the experimental one. The set of optimal designs, called Pareto front (Figure 2(c)) was found. Lower values of average fiber (MFD) and standard deviation of fiber diameter (FSD) are the indication of good quality and better performance. The optimum processing conditions leading to the desired fiber diameter were determined and are presented in Table 3. The maximum MFD value as well as the minimum FSD value were achieved by the combined level of 6.00356 (wt %) concentration, 69.99866 cm electrospinning distance, 40.05473 kV applied voltage 11.9669 rpm rotation speed and 0.49292629 mm wire diameter. Under these optimum conditions, the corresponding MFD and FSD values were found to be 378.4664 nm and 38.91183, respectively. The minimum MFD value as well as the maximum FSD value were obtained by the combined level of 9.998792 (wt %) concentration, 50.07165 cm electrospinning distance, 40.02671 kV applied voltage, 0.54997975 mm wire diameter and 11.89954 rpm rotation speed. Under these conditions, the corresponding MFD and SFD values were found to be 293.097 nm and 92.98008 nm, respectively. Under the above processing conditions, the ranges of MFD and SFD were 293.097 -378.4664 nm and 38.91183-92.98008 nm, respectively. The set of optimal solutions in the Pareto front was found to be 6.00356-9.998792 wt% for concentration, 50.07165-69.99866 cm for spinning distance, 40.02671-40.05473 kV for applied voltage, 11.89954-11.9669 rpm rotation speed and 0.54997975-0.49292629 mm wire diameter. This set of optimal solutions should allow the engineer to choose a good compromise between several goals, by picking a point somewhere along the Pareto front. To date, this advantage has not yet been explored enough in electrospinning engineering-based problems.

Table 2. Comparison of the optimization results (Pareto front) and experimental results.

Electrospun fiber properties	Optimization model	Experimental
MFD(nm)	293 - 378	267 - 704
FSD(nm)	38.9 – 92.9.	44.3 - 218.1

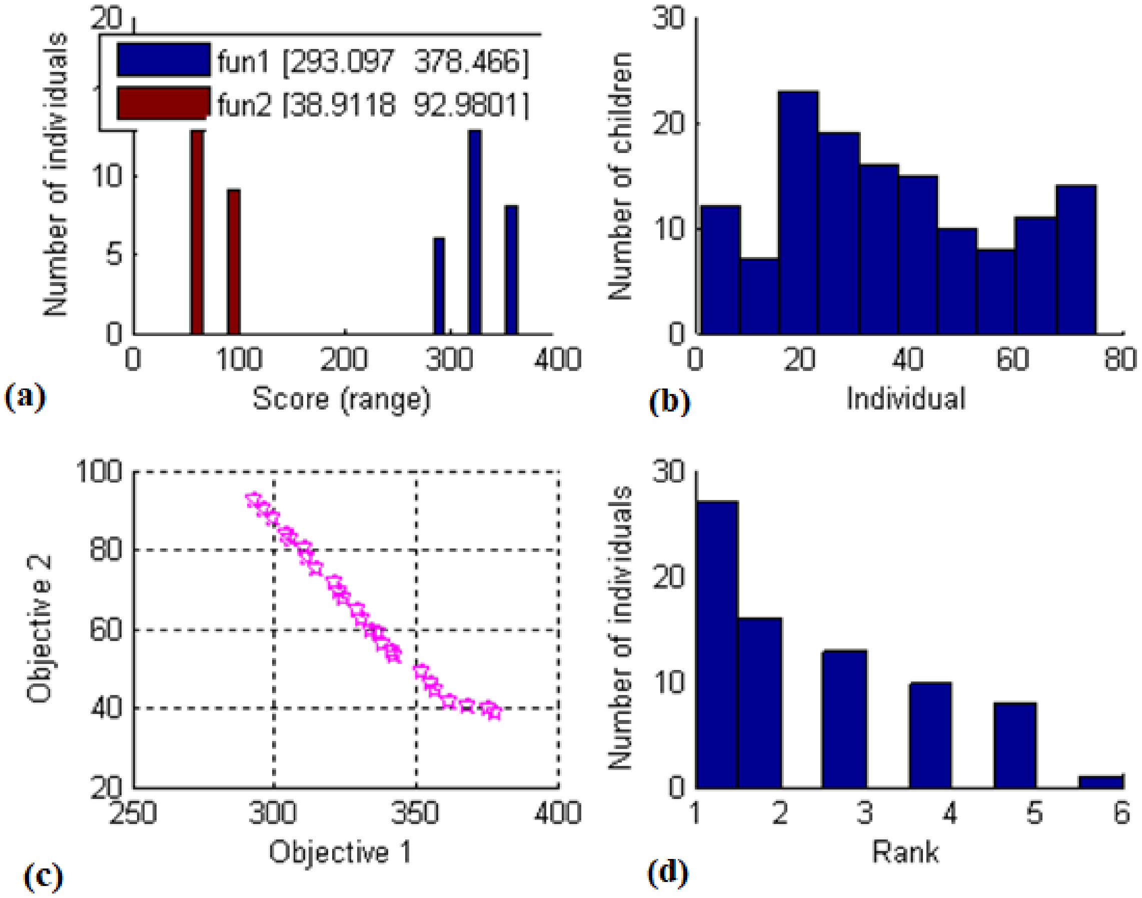

Figure 2. Various aspects of the genetic algorithm: (a) score histogram, (b) selection function, (c) Pareto front, (d) rank histogram.

Table 3. Optimum processing conditions leading to the minimum values of the targets.

Processing parameter	For MFD	For FSD
x_{1min}(mm)	0.549994	0.49379
x_{2min}(kV)	40.17213	40.52263
x_{3min}(cm)	54.22429	69.83992
x_{4min} (wt %)	9.999981	6.091517
x_{5min}(rpm)	11.67689	11.84783

6. Conclusions

In this study, a new multi-objective optimization algorithm based on improved gamultiobj was proposed to determine the optimal operating processing parameters in electrospinning process. Gene expression programming (GEP) model was first developed to predict electrospun fiber morphology using processing parameters processing parameters. These process parameters including PEO solution concentration, applied voltage, spinning distance, wire diameter and rotation speed were then mathematically related with the fiber properties (mean fiber diameter and standard fiber diameter) using a multi-objective optimization technique based on gene algorithm. Afterward, using a multi-objective optimization technique based on gene algorithm (GA), optimal conditions were found in such a way that fiber diameter to be minimized. One of the most important advantages of the proposed multi-objective formulation is that it obtains several non-dominated solutions allowing the system operator (decision maker) to exercise his personal preference in selecting each of those solutions based on the operating conditions of the system. This advantage has not yet been explored enough in the past in electrospinning engineering. The results of this study need to be repeated and compared against others from similar analysis.

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