Electrospinning of polyethylene terephthalate (PET) nanofibers: optimization study using taguchi design of experiment

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Abstract.: Electrospinning polymer has been acknowledged as an effective technique for the fabrication of polymer nanofiber. In recent years many polymers have been successfully electrospun into thin smooth uniform fibers. In this study, the effect of various process parameters on the morphology and diameter of fibers obtained from a solution of PET in Trifluoracetic acid and Dichloromethane in (1:3) ratio were illustrated. The selected factors that were to be controlled for this experiment are the concentration of PET in the solution, flow rate, applied voltage, and needle-tip to the collector distance. L9 Orthogonal arrays of Taguchi design, S/N ratio and analyses of variance ANOVA are used to arrange the number of trials, analyses and observe the optimum conditions for the fabrication of PET. For this, the best level of the factors was determined as PET concentration (5%), flow rate (1ml/h), voltage (15 Kv) and distance (15 cm) in order to obtain small fiber in diameter.

Keywords: Nanotechnology, Electrospinning, Nanofibers, Applications of nanofibers, PET, waste plastic, Taguchi method

1. Introduction

Nanotechnology is the study and using the small structures in the size between 0.1 nanometers to 100 nanometers [1, 2]. Nano is the Greek word for prefix “dwarf”. Materials and devices with special properties can be prepared by nanotechnology by treating of individual atoms, molecules, or compounds into structures [3]. Nanotechnology and nanoscience have been applied in several subjects, for example, health and medicine, energy and environment, Filtration, communication, and computer electronics [4]. Nanoscience is a versatile subject which can be gathering researchers from different fields such as physics, chemistry, engineering, and biology [5, 6]. Electrospinning is an extremely versatile and the most promising technique for producing nanofibers [7, 8]. Electrospinning technique was first remarked by Rayleigh in 1897 and Želeny in early 1900 investigated in details, it patented by Formhals in 1934 [9,10]. In the 1990 electrospinning
gained significant attention, which was partly started by the Reneker group. Over the past two decades, preparation of polymer, composite, ceramic, and hierarchically structured fibers with small diameters ranging from two nanometers to several micrometers have been widely prepared by electrospinning technique [11]. Electrospinning is a remarkably easy, efficient, and inexpensive way for producing ultra-fine fibers using a wide variety of polymers. Electrospinning nanofibers create by an electrically charged jet of polymer solution or polymer melt [12, 13]. Most of earlier investigations on polymeric nanofibers have been focused on the viscosity of polymer solution, the field strength, the type of solvent and the working distance of the system[14]. Electrospinning technique is the most common method for generation of nanofibers non-woven fibrous materials [15]. Spun nanofibers also offer several characteristics such as an extremely high surface-to-volume ratio due to their small diameters, nanofiber mats can be highly porous with excellent pore interconnection, the ability to control the nanofiber composition to achieve the desired results from its properties and functionality[7, 16]. Due to their characteristics, nanofibers are an ideal for a variety of high-value applications including protective textiles, filtration, biomedicine (including tissue engineering, implants, membranes, and drug delivery), photovoltaic cells, optical and chemical sensors, wound dressings, nanocatalysis, defense and security, and Sensors [17-19]. Currently, electrospinning and the resulting nanofibers are not the only concern in research laboratories but many companies from all over the world have started industrial-scale productions of electrospun nanofibers for various applications [20]. There are other techniques that polymeric nanofibers can be fabricated such as drawing, template synthesis, self-assembly, and phase separation [21, 22]. Polyethylene terephthalate (PET or PETE) is polyester, which is formed from ester monomers by condensation polymerization reaction. Ester monomers can be formed by the reaction between carboxylic acid and alcohol [23]. PET becomes the most common polyester for food and liquid packaging [24]. Non-woven nanofiber mat (NFM) has been successfully produced from PET polymer with trifluoroacetic acid by electrospinning technique [25]. Before electrospinning, most of the polymers are dissolved in solvents, and after its totally dissolved will form polymer solution [26]. A simple electrospinning component consists of high voltage, syringe pump, spinneret (solution contained syringe attached with a metallic needle), and collector plate [27-29]. Plastic bottles have been widely used for water drinking packaging in all over the world, which is produced from Polyethylene terephthalate (PET). PET has been expanded due to many advantages such as chemical resistance, excellent tensile strength, reasonable thermal stability, and its inexpensive cost [30,31]. Because of an extensively using of PET, management of PET waste become a serious issue as it is non-degradable plastic in the natural environment and causing environmental pollution after using. The importance of PET recycling is going to be more and more, especially in recent years, due to mentioned issues [32]. Different parameters can influence nanofibers in electrospinning such as intrinsic properties of the polymeric solution, operational parameters, and surrounding conditions. Intrinsic properties, for instance, polymer solution concentration, Viscosity, surface tension, and solvent have been influenced by the nanofibrous membrane morphology. Increase the concentration and viscosity will lead to increase nanofibers diameters [33]. Operational conditions, for example, applied voltage, flow rate, needle size, and needle tip-to-collector distance can also affect on the morphology of nanofibers diameter [34]. High voltage reduces nanofiber diameter [35]. Determination of nanofibers and bead diameters also depend on the flow rate because high flow rate ejects more polymeric droplets in jet and makes the initial jet diameters larger [34]. Increasing the distance between needle tip- to-collector decrease the nanofibrous diameters [36]. Decreasing the surrounding atmosphere such as temperature and increasing atmospheric humidity lead to decrease in nanofibers diameters [37]. The significance of this study is to reduce the waste materials in environmental such as PET packaging drinking mineral water bottles by using electrospinning technique, it
will form non-woven nanofibers mats and using these fibers for different applications as filtration of water purification, drug delivery, etc. Also, the study of electrospinning parameters on PET Nanofibers morphology to find the optimized that yield the finest fibers by using Taguchi design. Then, determination of these parameters such as concentration, flow rate, distance, and voltage on nanofibers diameter. This study show that the effect of these parameters has the same effect that has been illustrated in previous study as it is discussed in following sections.

2. Experimental details
2.1. Materials
Polyethylene terephthalate (PET) from water packing local company (SHIREEN water drinking company, Duhok city/Kurdistan region-Iraq) product as waste materials, after removing the non-PET components such as labels and cleaned with detergent and then dried (Intrinsic viscosity=0.51206 dl/g, The viscosity average molecular weight (Mv) of PET = 8359 g /mol). Trifluoroacetic acid (TFA98%), (CDH, CHINA), Dichloromethane (UNI-CHEM, INDIA).

![Figure 1. The electrospinning devices used in this study.](image)

2.2. Preparation of solution
PET solution was prepared by mix Trifluoroacetic acid and dichloromethane in (1:3) ratio, in this research we used three different concentrations (5,10 and 15%) of PET were carried out to obtain intended product. PET was grinned by (Gran household GR-CG6001) then sieved by (Test sieve 250 µm, Germany). The solution was stirred using magnetic stirrer at room temperature for (4 hours) to complete dissolving of PET.

2.3. Taguchi design for optimum conditions
Taguchi method for experiments is a modern robust design developed by Genichi Taguchi used in both scientific and manufacture industries, it is designed to illustrate the significant factorial effects and optimum conditions to improve quality and reduce both cost and time for desired process or experiments. Taguchi method contains a set of tables that enable main variable and interactions to be investigated
through a minimum number of trials. Using Minitab 2017 (Minitab Inc. USA) an L9 orthogonal array was selected for four factors of (polymer concentration, voltage, flow rate and the distance between the collector and needle tip) in three different levels as shown in Table 1. Taguchi methods are more desired compared to traditional full factorial design, because the traditional full factorial demand above eighty runs to complete the experiment of four different of three levels but using Taguchi method L9 OAs only nine runs required as shown in Table 2, this huge reduction in number of tests safe both experimental cost and time. Further, step DOE will transform data from Table 2 of uncontrollable factors to signal-to-noise ratio comparing to controllable fiber diameters factor to investigate the sensitivity of each factor and obtain the optimum conditions for best quality.

2.4. Electrospinning
The polymer solution was converted to nano-fiber by local designed electrospinning device(Fig.1). The process carried out according to Taguchi data given in Table (2). The solutions were placed in a glass syringe (500µL Model 750 RN SWITZERLAND Hamilton- Bonaduz, Scheweiz calibrated SYR, Large Removable NDL, 22ga, 2in, point style 2) which is attached to a stainless-steel needle with the inner diameter(0.41mm). The distance between tip and collector was(10cm) for position one, (13cm) for position two and (15cm) for position three as shown in (Fig. 1). The syringe connected to a handmade injection pump to control the solution feed rate with the speed of (0.5ml/h,1ml/h, 2 ml/h). The high voltage was applied by between the tip of the needle and an aluminum foil covering the collector using a high voltage Dc power supply (LYBOLD High Voltage Power Supply 25kV). Obtained nanofibers were analyzed by SEM (Cam Scan 3200LV with accelerating voltage 25Kv) working distance (10 nm), fiber been coated with (15 nm) of a gold layer with Magnification (3200) images. Further, the images were analyzed by (Image J 1.48v) randomly counting 50 fiber diameters according to the distance given by SEM in the bottom of images.

2.5. IR spectrum
A Fourier transform infrared spectrometer (Nicolet iS10 FT-IR spectrum) the range of wave number from 4,000 to 500 cm⁻¹ is used to determine the functional groups of fabricated electrospun PET fiber mats.

2.6. ANOVA Analysis
ANOVA is one of the most dependable statistical method for hypothesis testing at present [41]. Analysis of variance (ANOVA) was utilized to gain those parameters which are significantly affecting the quality of electrospinning nanofibers and attain the optimum condition[42]. Well- designed experiment (by Taguchi) is the key to a successful use of ANOVA analysis or any other statistical analyses. The total variation (ST), the summation of squares for each of four factors (Si) and the percentage contribution (%) were computed respectively by ANOVA.

2.7. Total variation (ST)
The following equation expresses the total variation (ST) which is the sum of squares of all trial results:

\[ S_T = \left[ \sum_{i=1}^{N} Y_{i} \right]^2 - \left[ \frac{\sum_{i=1}^{N} Y_{i}}{N} \right] \]  \hspace{1cm} (1)

Where Yi express the mean of fiber diameter and N is the number of trials in (Taguchi DoE).
Table 1. Selected factors and their levels used in Taguchi design.

| Factor           | Level |
|------------------|-------|
|                  | 1     | 2     | 3     |
| PET (%)          | 5     | 10    | 15    |
| Flow rate (µl/min) | 0.5   | 1     | 2     |
| Distance (cm)    | 10    | 13    | 15    |
| Voltage (kv)     | 9     | 12    | 15    |

Table 2. L9 orthogonal design for selected factors with their levels.

| Run | PET (%) | Flow rate(ml/h) | Distance(cm) | Voltage (kV) |
|-----|---------|-----------------|--------------|--------------|
| 1   | 5       | 0.5             | 10           | 9            |
| 2   | 5       | 1               | 13           | 12           |
| 3   | 5       | 2               | 15           | 15           |
| 4   | 10      | 0.5             | 13           | 15           |
| 5   | 10      | 1               | 15           | 9            |
| 6   | 10      | 2               | 10           | 12           |
| 7   | 15      | 0.5             | 15           | 12           |
| 8   | 15      | 1               | 10           | 15           |
| 9   | 15      | 2               | 13           | 9            |

Figure 2. IR spectrum of PET nano-fiber.
Four factors of PET such as concentration, feed rate, the distance of needle-to-tip, and the voltage at three different levels were mentioned as the sum squares $S_A$, $S_B$, $S_C$, and $S_D$, respectively. C.F stay constant for all factors and which is the correction factor. All sums of squares are calculated by the correction factor (C.F).

2.8. **Percentage contribution (%)**

The percentage contribution of $P_A$, $P_B$, $P_C$, or $P_D$ factors is the ratio of the total variance of each factor ($S_A$, $S_B$, $S_C$, and $S_D$) to the total variation (ST) as the following equation:

$$P_i = \frac{S_i}{S_T} \times 100$$  \hspace{1cm} (6)

Where $i$ is the number of factors ($i = 4$ for this study).

2.9. **Signal-to-noise(S/N) of electrospun PET nanofiber diameter**

Optimum combination factors identified by using a “smaller the better” characteristic formula to minimize the fiber diameter and its variation in electrospun PET nanofibers as given below:

$$S/N = -\log \left( \frac{1}{n} \sum_{i=1}^{n} y_i^2 \right)$$  \hspace{1cm} (7)

Where $n$ is the number of measurements, S/N is the signal-to-noise ratio, and $y$ is the diameter electrospun PET Nanofibers. Mathematically the greater the value of S/N, the smaller the variance of PET electrospun Nanofibers [43-45].

3. **Results and Discussion**

Obtained SEM micro graphs of electrospun nanofiber morphology in Fig [3] shows different results between runs (designed by Taguchi method) in surface smoothness, the number of beads, uniformity of nanofibers diameter and its average. Correspondingly the results are discussed qualitatively and quantitatively; the surface smoothness, formation of beads and nanofiber uniformity in diameter are counted as qualitative justifications for fiber morphology, then quantitative ones are established on fiber diameter and S/N ratio. Fig. (2) shows the FTIR spectrum of wasted PET been fabricated by electrospinning. Functional groups of electrospun PET nano-fiber mats were determined at specific peaks, as illustrated at 1719cm$^{-1}$ the (–C=O) carbonyl group was stretched, aromatic and aliphatic –C-H bond was determined to be at 2957 cm$^{-1}$ and 2887 cm$^{-1}$ respectively, and 1409 cm$^{-1}$ bending of –C-H bond, while 726 cm$^{-1}$ was the waging of aromatic hydrocarbon. A clear sharp peak of -C=O asymmetry figured at 1261 cm$^{-1}$. Fig (4) shows the frequency contribution diagram for the SEM micro graphs (analyzed by IMAGEJ 1.48v) in the diameter range of 50-1000 nm. The corresponding fiber diameter average incorporated with its standard divisions have been
illustrated in Table (3). On the overall scale, a random distribution is evident in most mat samples due to stand-alone mesh collector with little fiber alignment control. However, a quick look at SEM micrographs, it can be easily seen that first three runs has smaller fibers compared to the following runs.

A particular increase of fiber diameter denoted in Run-4, Run-8, and Run-9 experiments, comprising fiber diameter 380.6 nm, 450 nm, 403 nm and S/N ratio of -51.6, -53.06 and -52.12 respectively. Which means that an S/N ratio lower than -50 has large fibers diameter, this is due to the concentration of PET in the TFA 1:3 DCM solvent (0.51206 dl/g); as its known that the relationship between the polymer concentration and viscosity is proportional. Consequently, increase in polymer concentration will lead to forming larger fiber diameters [42]. Along with polymer concentration, the flow rate has a direct influence on fiber diameter. When more solution droplets were ejected from the needle tip per hour, these droplets with excessive PETs did not have sufficient time to be elongated to form small fibers in diameter, which were then directly received from the mesh collector. The applied voltage is another factor that influences the fiber diameter; generally, as we increase applied voltage more polymer solution will be ejected cause an increase in the fiber size, as illustrated in Run-4, Run-6, Run-7 and Run-8, with the average diameter of 380.6, 349.5, 342.1 and 450 nm respectively. Run-4, Run-6 and Run-8 have non-uniform fiber morphologies in common as illustrated in fig (a) and table (3), which demonstrated standard division of randomly taken fiber diameter of 380.6±232.9, 349.5±253.2 and 450±245 respectively. Which could be due to combination of high polymer solution concentration, low feed rate and instability in local designed syringe pump. It can be seen that by increasing the low feed rate and low concentration the structure of the fibers is much more uniform such as Run-5 of 170.8±45.35 nm. In addition, Run-6 and Run-8 also have high polymer concentration and high flow rate but typically with non-uniform fibers morphology. This most likely is due to small distance between needle tip and the collector. It is acknowledged that increasing distance will give more time to the ejected polymers to be elongated to form thin uniform fibers. Small-bead defects are mostly noticed in Run-1, Run-2 and Run-3 which could be caused by the low concentration of PET in the solvent leading to low viscosity. Decreasing viscosity will lead to the dispersion of the solution. Uprising the concentration in the next experiments show bead-free and relatively larger electrospun nano-fibers because of increasing the viscosity of the polymer solution [46, 47].

Table 3. PET fiber diameter with standard deviation and their corresponding signal-to-noise (S/N) ratios based on ‘smaller the better’.

| Run | Designation | Average fiber diameter(nm) | S/N values |
|-----|-------------|--------------------------|------------|
| 1   | A1B1C1D1    | 159.7±51.42              | -44.0661   |
| 2   | A1B2C2D2    | 105.03±36.79             | -40.4238   |
| 3   | A1B3C3D3    | 102.7±31.99              | -40.2314   |
| 4   | A2B1C2D3    | 380.6±232.9              | -51.6094   |
| 5   | A2B2C3D1    | 170.8±45.35              | -44.6498   |
| 6   | A2B3C1D2    | 349.5±253.2              | -50.8689   |
| 7   | A3B1C3D2    | 342.10±179.6             | -50.6831   |
| 8   | A3B2C1D3    | 450.00±242.5             | -53.0643   |
| 9   | A3B3C2D1    | 403.7±93.31              | -52.1212   |

Analysis of variance (ANOVA)

The Figure (5) above depicts the effect of processing factors on the response factor (nanofiber diameter) analyzed by Minitab7 based on data given to DOE Taguchi design which in turn organize the factors and the levels selected for this experiment and minimize
the number of trials. Percentage contribution of electrospinning factors obtained by equation [1-6]. PET concentration in the mixture solvent, needle-tip to collector distance, flow rate, and voltage were investigated to minimize the fiber diameter [41].

Fig. (6) demonstrate the effect of each factor by percentage contribution. The concentration of PET was the most significant factor in fiber diameter of a 78%, this improves the previous evidence that diameter size of PET has a linear relationship with PET concentration. The distance of Needle tip-to-collector happens to be the second most effective parameter of 15%, relatively needle-to-collector distance has a little effect on fiber diameter compared to PET concentration. Applied voltage and flow rate appeared to be insignificant in effecting fiber diameter with a percentage contribution of 4% and 3% respectively. The last two factors have a minor impact on electrospun nano-fiber, this situation can be utilized to change the levels of these two factors as it requires. Table (3) shows different values of S/N ratio calculated by equation (7) using “smaller the better” for each run organized by Taguchi design. S/N representing the magnitude of the mean fiber diameter compared to its variation factor. As illustrated by Taguchi, large differences between the values of S/N ratio of a specific factor indicate the magnitude of its significance on fiber diameter. Results illustrated in the Table (3) determine that Run-3 has the largest S/N value of (-40.2) with mean fiber diameter and standard deviation (102.7±31.99) to be the best candidate to make small nanofibers.
Figure 3. SEM images of electro spun PET nanofibers produced according to the experimental design.

However, Run-2 of S/N ratio (-40.42), fiber diameter and standard deviation of (105.03±36.79) is another trusted combination to make small fiber which has slightly smaller S/N ratio and larger fiber diameter and standard deviation compared to Run-3. Optimum combination illustrated in Fig. (5) represent that (A1, B2, C3, and D1) are the best combination of this experiment where concentration of PET (A1) is 5%, flow rate (B2) is (1ml/h), needle tip-to-collector distance (C3) is (15 cm), and applied voltage (D1) of (9 Kv). For this result, it was decided to apply (15 Kv) as the optimum voltage because it is insignificant also this factor may differ according on type of polymer solvents system.
Figure 4. The frequency contributions to nanofibers diameter range in DoE study: (a) T1; (b) T2; (c) T3; (d) T4; (e) T5; (f) T6; (g) T7; (h) T8; and (i) T9.
Figure 5. Main effect plots of each parameter on means of nanofiber diameter.

Figure 6. Percentage contribution of each factor.

4. Conclusion

PET fibers were electrospinning based on four factors of an L9 orthogonal arrays with S/N ratios and ANOVA in Taguchi method. Polymer concentration, needle tip-to-collector distance, feed rate and applied voltage at three different levels were studied to investigate the optimal factors levels for a thinner fiber diameter during electrospinning. The concentration of PET solution was found as the most influential factor on the diameter of PET fibers. The smaller diameters were produced by the
lower concentration. Distance is the second most influential factor on the PET fibers diameter. The optimum electrospinning factors were determined to be as follows: 5% PET concentration, 1 ml/h feed rate, 15 cm needle tip-to-collector and 15 kV for the applied voltage.

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