



Relationship between Electrospinning Parameters and Physical Properties of Tubular Structure Nanofibers by Opposite Charge Electrospinning.

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ABSTRACT: This study describes the opposite charge electrospun method for preparation of polyacrylonitrile / DMF nanofibrous tubular structure . Constant a processing parameters such as high polymer concentration and high rotational speed mandrel collector with varied the applied voltage and flow rate in wide range to display the effect of each one in determining the diameter , alignment, porosity, mean surface area and mean pores size of nanofibers. Tubular structures were produced from highly aligned continuous and uniform nanofibers through the opposite charge electrospinning methods and effect of each regulating parameter on tubular structure properties were evaluated and characterized using scanning electron microscope (SEM) and Fast Fourier Transform (FFT) and thresholding method. Increasing flow rate of polymer solution considerably increased the diameter of the nanofibers , ranges of the average diameters were larger , the nanofibers diameter distribution were gradually border. Nanofibers diameter on the other hand, decreased as the applied voltage increased, ranges of the average diameters were smaller and the nanofibers diameter distribution were gradually narrower . while increased the porosity % , heights intensity(Y%) and mean surface area (nm²) and decreased the mean pores size (nm) with increased both the applied voltage and flow rate of polymer solution .

KEYWORDS: Opposite charge electrospinning; Tubular structure; Mean Surface Area; Alignment; Flow rate ; Applied voltage, Porosity %.

INTRODUCTION

The production rate of nanofibres from a single-needle setup or traditional electrospinning method, is considerably low and limiting the large-scale application , therefore , for production aligned fibres should be used the other types of collectors such as rotating drums or mandrel collectors [1,2]. Also, the low production rate and non-oriented nanofiber production play a negative role in the development of the electrospinning method [3]. The rotating collector can be used to produce the 3D nanofibers , by using the rotation speed, which plays an important role in determining the degree of anisotropy. Furthermore, the geometry of the collector can be selected according to the application. For example, a cylindrical can be used to form a sheet, while a rotating mandrel can be used to form a tubular structure[4- 6]. One of the important benefits of using mandrel or drum collector is a fast drying process occurrence that can prevent the fusion at the contact sites and maintain the original fiber shape [7]. Also, these fibers collected at a higher drum or mandrel rotation speed have a narrower distribution of fiber orientations with high porosity [8], also narrowing nanofibers diameter distribution[9]. To fabricate fibers that are not only non-woven meshes but also aligned, patterned, twisted yarn,

and three-dimensional structures, developing new methods for controlling the deposition behavior of the fibers either by mandrel collector shaft, rotating drums, disk collectors or parallel electrodes are required [10,11] Xie, et al ,(2010) used the opposite charge nozzle's to align 3-D nanofibers into a uniaxial array. They modified the typical set-up by installing two needles in opposite directions and pumping the polymer solution into needles by two syringe liquid pumps with the same injection rate[12]. Aligned and molecularly oriented Polyacrylonitrile nanofibers were prepared using a non-conventional approach, that is using two needles in opposite positions and a rotating collector perpendicular to needle axis[1-3] . The electrospinning parameters such as polymer concentration, applied voltage and flow rate of solution strongly affect the solidification rate of the resulting nanofibers and the fast stretching and solidification of the polymer chains lead to another important effect which is the decreasing in crystallinity of polymer nanfibers since the stretched chains do not have enough time for crystal formation. However, these non-crystalline polymer chains in the nanofibers are yet strongly oriented [14-16] . Applied electric field is one of the most important parameters in the electrospinning process due to its direct influence on the

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dynamics of the fluid flow. In the electrospinning process, a high voltage is introduced into a polymer solution such that charges are induced within the fluid. However, the changes in the applied voltage will be reflected in the shape of the suspending droplet at the nozzle of the spinneret [17]. Considering the effects of electrospinning parameters on nanofiber diameter reveals that when keeping other parameters constant, the diameter of nanofibers decrease on increasing the applied voltage [18,19]. The increase in the applied voltage (i.e., by increasing the electric field strength), increases the electrostatic repulsive force on the fluid jet which ultimately favors the narrowing of fiber diameter [1, 15,20,21]. Increases the applied voltage due to increase the acting forces which attempt to align the nanofibers, in spite of, increases whipping instability of electrospinning jet [22]. Also, the increase the electric forces lead to increase surface area of the jet, accelerated the evaporation rate of solvent, decreasing the radius loop and slower elongation rate of electrospinning jet at higher polymer concentrations [23]. On the others hand, the increasing flow rate due to increase the production rate and diameter of the fibers [24,25], also at higher flow rates as more materials are drawn from the nozzle tip, and the time for the jet to dry is reduced [14]. The fiber morphology is controlled by the experimental design and is dependent on solution conductivity, solvent polarity, solution concentration, polymer viscosity, molecular weight, and applied voltage [22,26-28]. Therefore, using the opposite charge electrospinning method for spinning the polyacrylonitrile / DMF solution. For producing 3D nanofibers having a good morphological properties, should be controlling on the electrospinning parameters such as applied voltage and flow rate of polymer solution, the rapid evaporation of the solvent while the jet is accelerated toward the collector lead to improve the electrospinning of a polymer

solution [7,29,30] Bonani, et al, (2011), developed a new double –electrospinning system for produced the bio hybrid nanofiber constructs, they found high results in anisotropic properties of tubular structure nanofibers with using this system [31]. Therefore, one of the goals in this study is to create a structure that reproduces anisotropic properties by combination of the collector shape and the collector rotation allows us to produce a tubular structure with tunable anisotropic properties by used the high concentrated polymer solutions in electrospinning, It offers a unique combination of control over tubular structure nanofibrous at relatively high production rates and increased the percentage of porosity, mean pores diameter with standard deviation and mean surface area with standard deviation.

EXPERIMENTAL

Materials

Commercial Polyacrylonitrile (PAN) powder with molecular weight of 100,000 (g/mol) was supplied by Poly Acryl Company, Iran and Dimethyl formaldehyde (DMF) was obtained from Merck (Schuchardt OHG 85662 Hchenbrunn, Germany).

Preparation of the Electrospinning Polymeric Solutions

The appropriate amounts of PAN and DMF were mixed using a heating magnetic stirrer at a temperature of 40 °C for 4hr to obtain a well-dissolved transparent light yellow homogeneous polymer solution, ready Schematic diagram for the experimental setup was shown (Fig. 1) to produce the corresponding to the electrospinning conditions used for production tubular structure nanofibrous by effect of the applied voltage and flow rate of polymer solution present in Table 1.

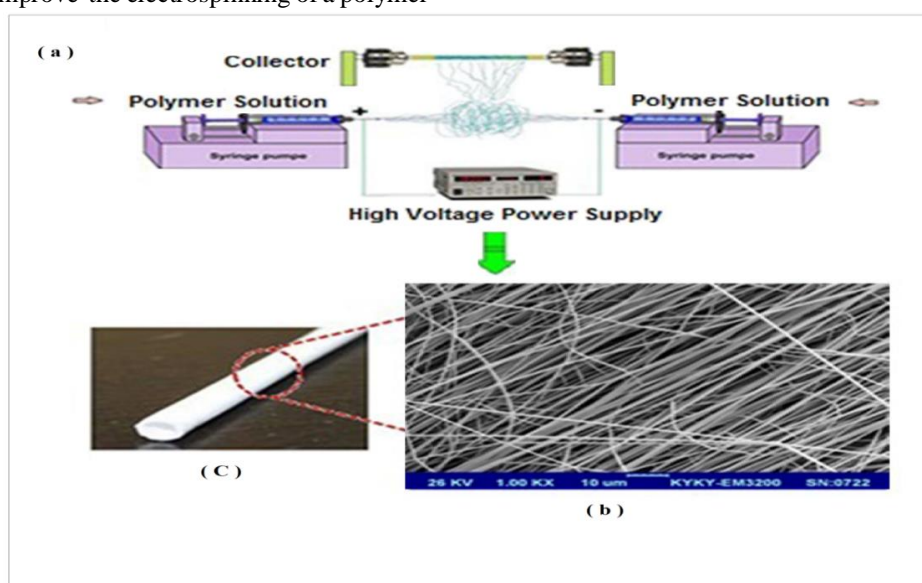


Fig . 1 (a) Schematic diagram of the opposite charge electrospinning set-up for aligned nanofibers, (b) 1.00 KX, 10µm SEM image and (c) tubular structure sample .

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Table1. The electrospinning conditions used for production tubular structure nanofibrous by effect of the applied voltage and flow rate of polymer solution .

Electrospinning Conditions	Values
Applied voltages change (kV)	12,16, 20
Polymer concentration (wt.%)	15
Rotational speed (r.p.m)	4328
Distance between the needles and collector (cm)	20
Distance between two opposite needles(cm)	10
Flow rate of solution (mL / hr.)	0.3,0.4,0.5

Characterizations

Scanning electron microscopy (SEM).

The structural and morphological characterizations of the pure polymeric structures were performed by scanning electron microscopy (SEM), KYKY-EM 3200 model , the surface of samples was coated with a layer of gold with the thickness of 100 Å by using the sputter coater KYKY-SBC12, the average diameter with standard deviation(Std. Dev.) were calculated by using Digimizer image analysis software version 5.3.3 and the Frequency histograms of nanofibers diameter distribution was plotted by XLSTAT Analysis Software , 2016 for Microsoft Excel versions.

Statistical Analysis

All the data were analyzed regarding to Kruskal-Wallis test (XLSTAT Analysis Software , 2016 for Microsoft Excel versions) to determine if the data were normally or non-normally distributed. . The data from which all the samples were extracted follows a Normal distribution . Data of pores diameter distribution according to statistical (one –way ANOVA) significance was accepted at $p < 0.05$.

Alignment of Nanofibrous Analysis.

Fast Fourier Transform (FFT) method were used to identify the functional groups and vibrational modes of the materials as well as to quantify the alignment of nanofibers.

SPIP(TM) version 6.7.4 Image Metrology A/S , Hørsholm , Denmark software analysis (Scanning Probe Image Processor) converted to 8-bit grayscale TIF files and typical images are 256×256 or 512×512 pixels for the FFT/PSD analysis used to calculate the fast Fourier transform and power spectrum density with two –dimension , it calculated as the power value normalized with the area size of each element . $PSD(u, v) = P(u, v) / (x\ Range * y\ Range)$...where $x\ Range$ and $y\ Range$ are physical ranges of the source image and (u, v) are u, v the discrete Fourier indexes $=0,1,2$. For 1D Average Y- PSD, The PSD Line Average Functions will calculate PSD spectra for each Y column and then calculate the average PSD spectrum. Here, each Z value is calculated as the power value normalized with the are size of each element .

$PSD(u) = P(u) / Range$, where $P(u)$ is the power value for the Fourier index u and $Range$ is the physical range of the profiles, if the graph will have a higher accuracy it must be

calculated in high resolution mode and the four highest peaks have been detected (M1,M2,M3 and M4).

Surface Areas and Porosity of Nano fibers Analysis

There are few methods for measuring the porosity such as image analysis [32] by using SPIP(TM) version 6.7.4 Image Metrology A/S , Hørsholm , Denmark software analysis (Scanning Probe Image Processor) to detecte the pores in the SEM image and calculated the porosity (%), mean surface area $(nm)^2 \pm$ standard deviation, mean pores diameter $(nm) \pm$ standard deviation and the heights intensity(Y%) of distribution pores using the threshold method according to the following :

- 1- Limited the inspection box on the surface of image
- 2- Detected the mean pores diameter with standard deviation (Std. Dev.) and surface area with (Std. Dev.) on the surface of image
- 3- Plotted the histogram with limited the heights intensity of pores distribution(Y%) and porosity%

RESULTS AND DISCUSSION

Effect of the Electrospinning Parameters on Nano fibers Diameter .

Applied electric field is one of the most important parameters in the electrospinning process due to its direct influence on the dynamics of the fluid flow. A set of experiments were performed by the opposite charge set-up with the high applied voltage varying from 12 to 16 and to 20 kV. The other parameters were kept constant for the purpose of comparison ,were shown in the (Table 1). Fig. 2 shows the average fiber diameter with standard deviation (Std. Dev) produced from 15 wt.% polymer solutions produced by the opposite charge set-up as a function of the three applied voltages . And Fig. 3 present the SEM macrographs and frequency histogram of the nanofibers diameter distribution of the PAN solution concentration 15 wt.% each at three different applied voltages From the Fig. 2 it can be noted that, on increasing the applied voltage the average diameter of the nanofibers decreased, which can be attributed to the increased pulling effect on nanofibers as the electrical forces between the two needles was high . This was observed by other researchers as well [17,19]. Increasing in the applied voltage lead to accelerating liquid jets from the tip of a capillary and thus, decreasing the time period for producing the nanofibers. This result was

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reported by other researchers as well [20, 33,34] . On the other hand, from **Fig. 3** it was noted that, increasing in applied voltage from 12,16 and 20kV Lead to change in p-values from 0.113, 0.04 and 0.027 , which significate to the nanofibers diameter distribution closed to zero were considered normal distribution and narrowing the nanofibers diameter distribution with constant the solution concentrations and increasing the production rate of alinge nanofibers (increasing the density of nanofibers deposition) [35] , because the electrospinning jets produced by opposite charge method due to combination of mechanical and

electrical forces, which produced higher production rate of nanofibers compared to single-jet electrospinning methods. Therefore, the optimum voltage applied was selected as 20 kV having good quality fibers and high density of nanofibers depositions . Considering the effect of the applied voltage on the average diameter of PAN electro spun nanofibers produced from 15 wt.% polymer solution concentration, as the voltage increased, the fibers became thinner, which agrees with the previous studies performed by Lee, et al(2004) and Wu, et al (2010) [36.37] .

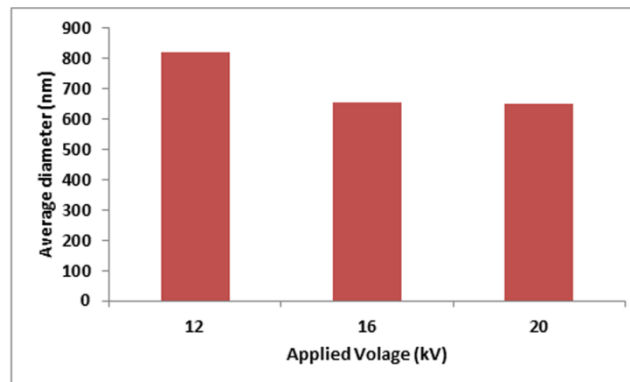


Fig.2 the average fiber diameter produced from 15wt%. polymer solutions by the opposite charge set-up as a function of the three applied voltages.

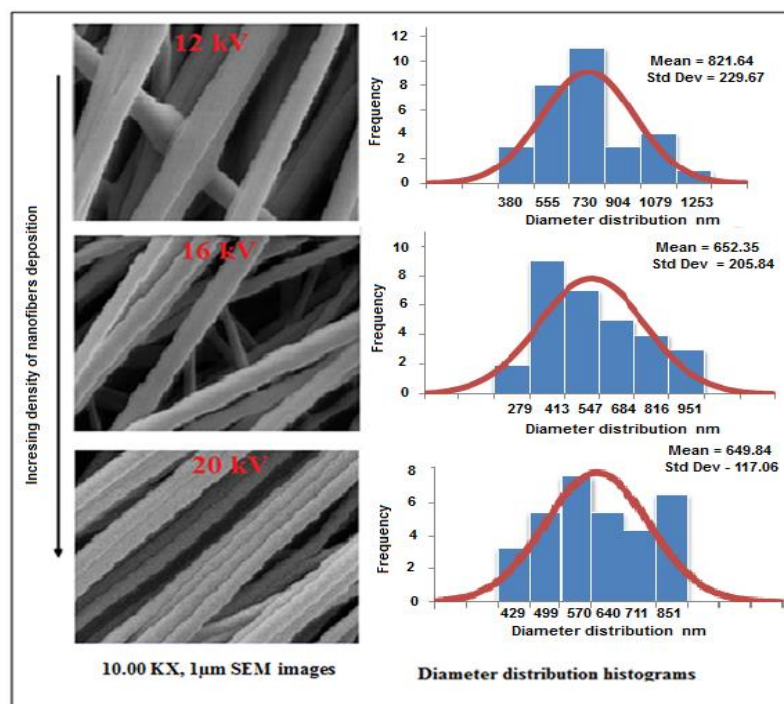


Fig. 3 SEM micrographs and the frequency histogram of the nanofibers diameter distribution of the PAN solution concentration of 15 wt.% at three different applied voltages.

The effect of the polymer flow rate on the resulting nanofibers morphology was studied by increasing it from 0.3 ml h⁻¹ to 0.5 ml h⁻¹ in a set of experiments performed by using the opposite charge method, where the following conditions were kept constant (**Table 1**) . **Figures 4,5** illustrate the effect of

the polymer flow rate on the nanofibers diameter at the 4328 rpm mandrel speeds performed by opposite charge set-up and present the SEM micrographs with frequency histograms of nanofibers diameter distribution with the polymer flow rate of 0.3, 0.4 and 0.5 mL/ hr. respectively.

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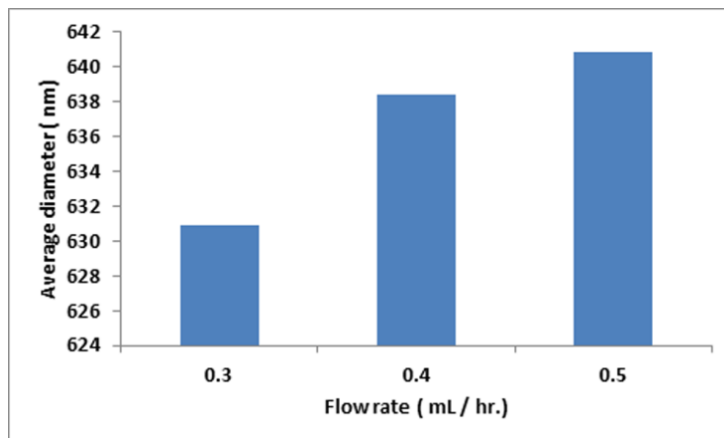


Fig. 4 Effect of the polymer flow rate on the nanofibers diameter at 4328 rpm mandrel speeds .

The obtained data as presented in **Figures 4, 5** well illustrate that, on increasing the solution flow rate, the electro spun nanofibers became thicker and more irregular. At higher flow rates, the suspending droplet at the end of the needle becomes larger, and the solution jet can carry the fluid away with a faster velocity [36]. From micrographs of SEM images (**Fig. 5**) the increasing in the flow rate lead to increase the production rate of nanofibers (increase the density of deposition nanofibers on rotating mandrel collector) which agrees with the reported results from Peterson, et al, (2010) [34]. Few studies have systematically investigated the relationship between solution feed or flow rate on fiber

morphology and size. In general, it was found that lower flow rates yielded fibers with smaller diameters [15, 37]. The flow rate that was too high resulted in beading since fibers did not have a chance to dry prior to reaching the collector [38-40]. The experimental data obtained in this study reveal that, the distribution of nanofibers diameter become gradually broader with increased flow rate of the solution from 0.3 to 0.5 ml hr⁻¹ and the p-values change from 0.34, 0.273 and 0.027 according to Kruskal–Wallis testing method, which agrees with the reported results from Subbiah, et al (2005) [41]. The best result at 0.5 mL / hr. having the good quality of nanofibers and high density of nanofibers depositions.

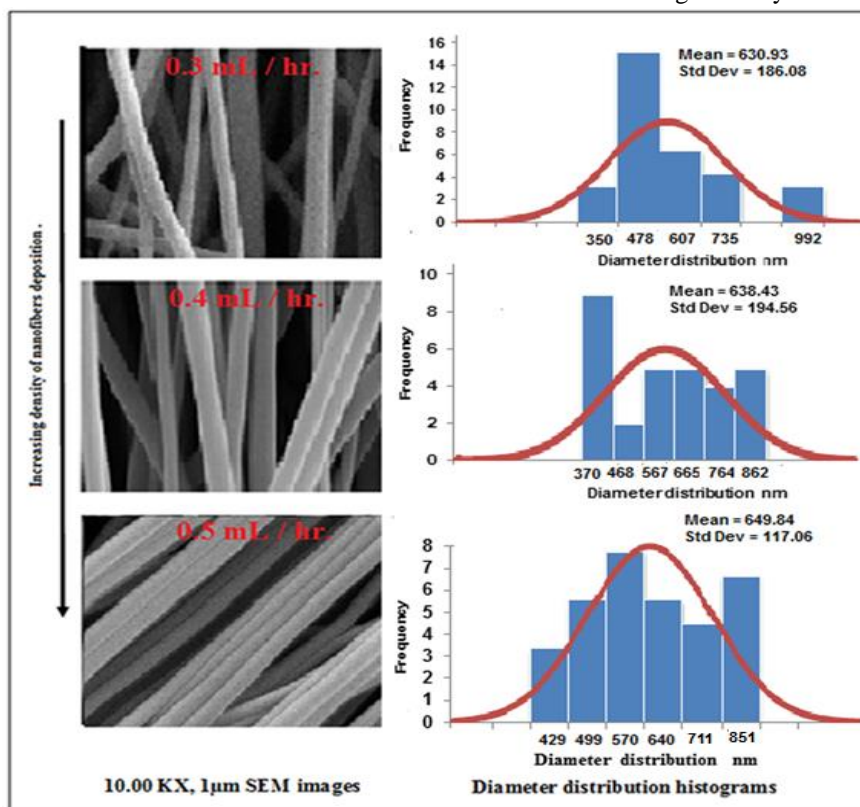


Fig. 5 SEM micrographs and the frequency histograms of nanofibers diameter distribution with the many polymer flow rates .

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Effect of the Electrospinning Parameters on Alignment of Nanofibers .

A set of experiments were performed by the opposite charge set-up with the high applied voltage varying from 12 to 16 and to 20 kV. The other parameters were kept constant for the purpose of comparison. **Fig.6** shows that the SEM micrographs, 1D Average Y-PSD and 2D FFT / PSD Fourier Images Analysis of SEM Images [42, 43] of the

nanofibers produced from the 15 wt.% polymer solutions at three different applied voltages obtained through opposite charge electrospinning method . In 1D Average Y-PSD testing (**Fig. 6**), the first and highest peak reflects the pitch distance which were changed from 115.74, 82.732 and 55.242 pixels and the average power spectrum density(PSD) were changed from 7649, 4243 and 7659 respectively, where the applied voltage varied from 12,16 and 20 kV .

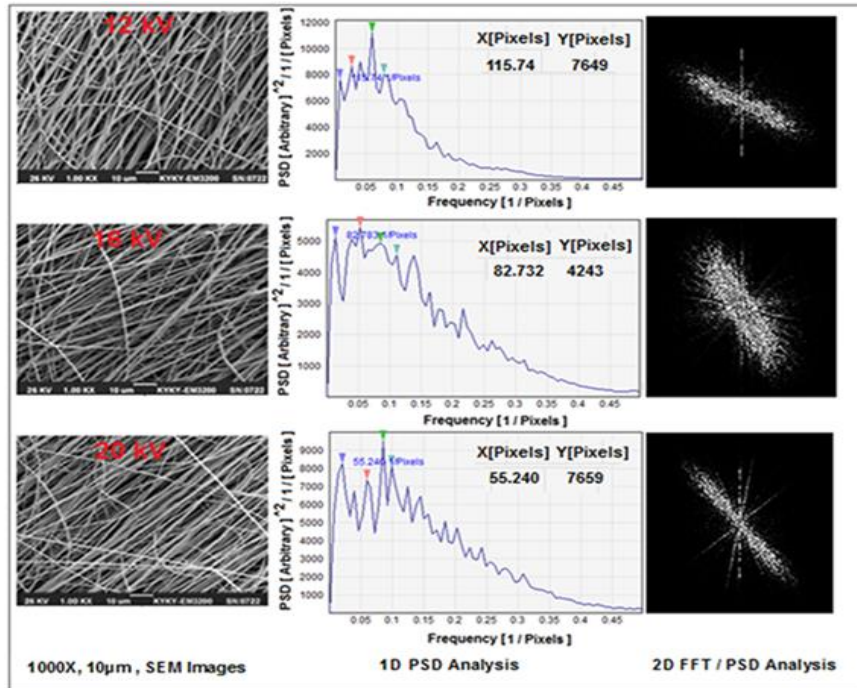


Fig. 6 SEM micrographs, 1D Average Y-PSD and 2D FFT / PSD Analysis of SEM Images of the nanofibers produced from the 15wt.% polymer solutions at many applied voltages .

The FFT analysis of the SEM images of the nanofiber samples was used to characterize the anisotropy of the scaffolds to digitize the alignment level of the nanofibers. Patterned, grayscale pixels are distributed in the output image of the 2D FFT/ PSD analysis to reflect the degree of fiber alignment of the original data image. The FFT data of the image with aligned fibers results in an output image with non-randomly and elliptically distribution pixels at 12,16 and 20 kV . the pixel intensities were plotted between 0 to 360°, and the degree of alignment in the FFT data reflected the shape and height of the peak ,also the increased the intensity and fewer occurrences of peaks indicate that the slightly alternating alignment with voltage, because the directional electric field intensity toward the electrodes lead to align nano fibers and increase the bending instability the optimum results of the nanofibers alignment are presented in the 20 kV. The effect of voltage observed in these experiments may be related to the “whipping” instability .The most important element operating during electrospinning is the rapid growth of a whipping instability that causes bending and allows the electrical forces to elongate the jet [44]. Increasing the

applied voltage, increases whipping instability and consequently increases the as-spun nanofibers entanglement which makes alignment of nanofibers more difficult, but on the other hand increases the acting forces which attempt to align the nanofibers at 12 kV and above. The current results obtained agree with the previous experiments reported by Jalili, et al,(2006) and Rahmani ,et al ,(2014) [22, 44] . On the others hand , the effect of the polymer flow rate on the resulting nanofibers morphology was studied by increasing it from 0.3 ml h⁻¹ to 0.5 ml h⁻¹ in a set of experiments performed by using the opposite charge method .And **Fig. 7** shows that the SEM micrographs, 1D Average Y-PSD and 2D FFT / PSD Analysis of SEM Images of the nanofibers produced from the 15 wt.% polymer solution at (0.3,0.4,0.5) ml/ hr. flow rates of polymer fabricated through the opposite electrospinning set-up. According to **Fig. 7** , we noted that formation fours highest peaks with high resolution mode ,were varied the flow rate ,the first and highest peak reflects the pitch distance which were changed from 48.768, 89.512 and 55.352 pixels and the average power spectrum density(PSD) were changed from 4077, 5824 and 7659 when

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increased the flow rate from 0.3, 0.4 and 0.5 mL / hr. respectively . The FFT analysis of the SEM images of the nanofiber samples was used to characterize the anisotropy of the scaffolds to digitize the alignment level of the nanofibers.

Patterned, grayscale pixels are distributed in the output image of the 2D FFT/ PSD analysis to reflect the degree of fiber alignment of the original data image.

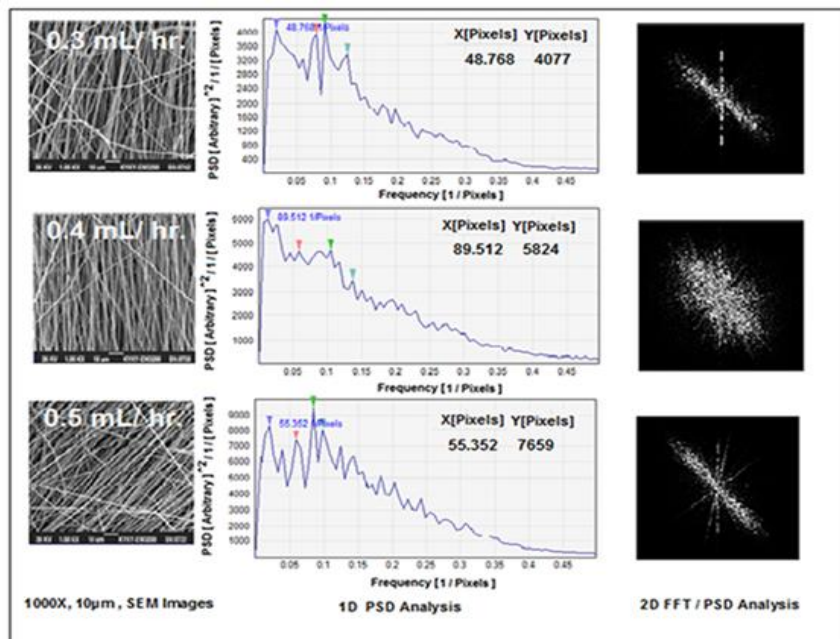


Fig.7 SEM micrographs, 1D Average Y-PSD and 2D FFT / PSD Fourier Images Analysis of SEM Images of the nanofibers produced from the 15 wt% polymer solution at 4328 rpm and at many polymer flow rates .

Effect of the Electrospinning Parameters on Porosity And Surface Area of Nanofibers .

Figures 8, 9 and Table 2 , show the effect of electrospinning parameters such as applied voltage and flow rate of polymer solution on porosity (%) , mean surface area(nm)² with standard deviation (Std. Dev.), mean pores diameter with standard deviation(Std. Dev.) (nm) , height intensity (Y%) of pores distribution by using the threshold method to detect the Pores in the SEM images with SPIP(TM) software analysis [45,46] . When increased the applied voltage from 12, 16 and 20 kV slightly increased the percentage , height intensity of

pores distribution , percentage porosity and the mean surface area, while decreased the mean pores diameter with increased the applied voltage (Fig. 8, Table 2), the highest porosity(%), surface area (nm²) and highest intensity (Y%) of distribution pores on histogram curves and lowest mean pores size at 20 kV . The analyzed using Kruskal-Wallis test (XLSTAT Analysis Software , one-way ANOVA) for the change in the pores size were not normally distributed . The statistical data reported about the changes in mean pore size ± (Std. Dev) was accepted at P < 0.05 with increasing in the applied voltage.

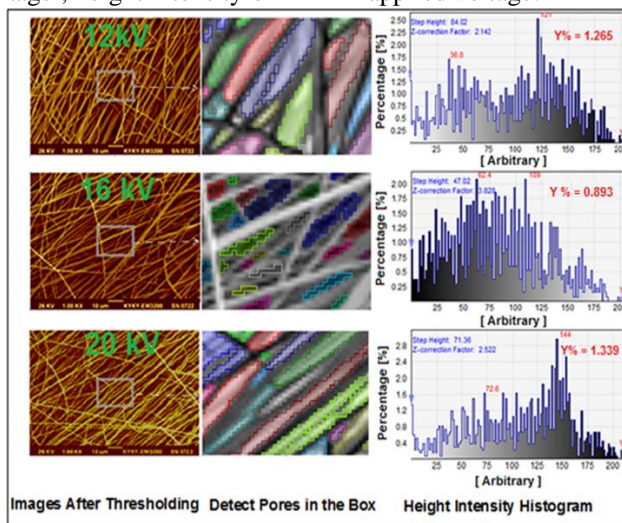


Fig. 8 SEM micrographs after thresholding, Detected pores and Height intensity histogram analysis of the nanofibers produced from the 15 wt% polymer solution at 4328 rpm with many applied voltages, N=6, P< 0.05 .

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On the other hand, when increased the flow rate of polymer solution lead to increasing the porosity(%), the mean surface area (nm)² and height intensity (Y%) of distribution pores on histogram curves with decreased the mean pores diameter (nm) (Fig. 9, Table 2) and the changes in mean pore size ± (Std. Dev) with increased the flow rate was accepted at P < 0.01, the highest Porosity (%), surface area (nm)² and highest intensity (Y%) of distribution pores on histogram curves at 0.5 mL / hr. Therefore, we noted that, polymeric nanofibers web containing the heights surface area with lowest pores

size and pores diameter distribution become more narrowing with increasing the applied voltage and flow rate of polymer solution [41], also, at high polymer concentration and high rotational speed of mandrel collector we found a good relationship between the uniformity, alignment with porosity and surface area of nanofibers. because controlling of the nanofibers deposition with rotating collector lead to linear straight jet by increased the flow rate especially at high concentration compared with conventional electrospinning method [47].

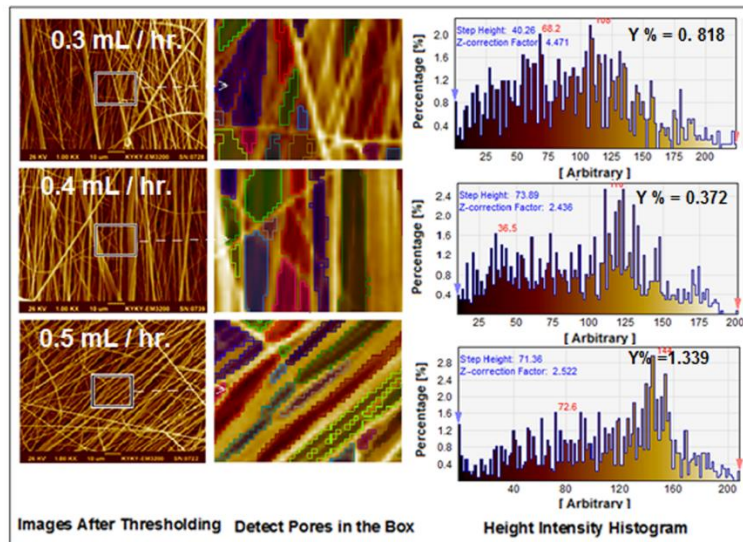


Fig. 9 SEM micrographs after thresholding, Detected pores and Height intensity histogram analysis of the nanofibers produced from the 15 wt.% polymer solution at 4328 rpm with many flow rates, N=6, P<0.01.

Table 2. The results of porosity (%), mean surface area, mean pores diameter and height intensity of pores distribution (Y%) by SPIP image processing software after effect of increased the applied voltage and flow rate of 15 wt.% of polymer concentration

Samples	Porosity (%)	Height Intensity (Y%)	Mean Surface Area (nm) ²	Mean Pores diameter (nm)
12 kV	48.06	1.265	21940.2±81.45	5.11±3.23
16 kV	40.31	0.893	5371.9±71.16	6.15±3.31
20 kV	51.50	1.339	76983.1±98.16	3.72±1.52
0.3 mL / hr.	41.50	0.818	7618.5±8017.5	7.00±3.41
0.4 mL / hr.	44.10	0.372	6257.3±4541.3	5.33±2.76
0.5 mL / hr.	51.50	1.339	76983.1±98.16	3.72±1.52

CONCLUSION

PAN tubular structures were produced from highly aligned continuous and uniform nanofibers through opposite charge electrospinning methods. Increasing flow rate of polymer solution considerably increased the average nanofibers diameter with standard deviation (Std. Dev) and the range of diameter of the nanofibers. The highest alignment, porosity (%), mean surface area (nm)², height intensity (Y%) of pores distribution of nanofibers and lowest mean pores diameter (nm) at 0.5 mL / hr. of polymer solution, among the three flow rates of polymer solutions studied. On the other hand,

the diameter with standard deviation (Std. Dev) and range of nanofibers decreased as the applied voltage increased and the highest alignment, porosity (%) mean surface area (nm)², height intensity (Y%) of pores distribution of nanofibers and lowest mean pores diameter (nm) at 20kV applied voltage, among the three applied voltages. The nanofibers produced from higher concentrations of polymer solutions as well as higher take-up speeds at higher flow rate and applied voltage, as they had higher directional alignment, mean surface area and porosity and lowest mean pores size.

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ABBREVIATIONS

PAN : polycrylonitrile ; DMF: N,N-Dimethylformamide ; SEM: Scan Electron Microscopy; FFT : Fast Fourier Transform ; PSD: Power Spectrum Density

AVAILABILITY OF DATA AND MATERIALS

The datasets produced and analyzed for this work are obtainable from the corresponding author on reasonable request.

COMPETING INTERESTS

The authors proclaim that there are no disagreement of benefit related to the publication of this paper.

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