Effect of Electro-spinning applied Voltage on Electro-spun EPS Membranes Thickness and Fibers Diameters

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Abstract. Expanded Polystyrene (EPS) solutions with a percentage of (30 wt. %) were prepared followed by the fabrication of micro-fibers membranes by electrospinning technique. To study the voltage’s effect of electrospinning device on EPS micro-fibers membranes, different voltages (10, 12, 15, 18, 24, and 30) KV were experimented. The characterization of the electro-spun membranes were made by Fourier transform infrared spectroscopy (FTIR) and the scanning electron microscopy (SEM) for morphological and thickness analysis. The results showed that although electro-spun EPS membranes prepared by 30 KV, they had the minimum membranes thickness and the highest average fiber diameter. As it is cleared by SEM images, increasing the electro-spinning voltage lead to increasing the average fiber diameter membranes. With increasing the electro-spinning voltage, the fibers prefer to deposit on each and result in decreasing the distance between the collector and the needle, hence higher average fiber diameter produced. The thickness of the electro-spun membranes decreases with the electro-spinning voltage as a result of the higher repulsion between the electro-spun fibers which reduce the deposition of these fibers on the collector.

Keywords: EPS, Electro-spinning, average fibre diameter, Thickness test.

1. Introduction

Electrospinning technique is a multi-sided technique has the ability to produce nano and micro polymeric fibers [1]. High aspect ratio as well as large surface area is important characteristics that made the electro-spun fibers attracted a considerable attention due to their special characteristics and potential applications in electrochemical devices [2]. This technique has attracted large attention in recent years due to the easy production of fibers from natural either or synthetic polymers [3]. Every soluble polymer owns enough high molecular weight is suitable to be electro-spun. The electro-spinning device consists of the following parts: (i) a high voltage power supply, (ii) nozzle, and (iii) grounded collector as shown in figure (1) [4].

Due to applying high voltage, the polymeric solution (presents at the tip of the syringe needle) will be charged and this will lead to the formation of a repulsive force. At the same time, the Taylor cone (which is a conical structure) will be formed at the tip of the syringe needle [1]. At a critical voltage, the repulsive forces could be able to dominate the surface tension of the polymeric solution and consequently, a jet blows up from Taylor cone. The jet is accelerated and goes along the distance between the syringe needle tip and the collector, and divided into a large number of filaments. Along
with the distance, the solvent will be evaporated and the fibers form on the surface of the collector [5]. To fabricate fine fibers, adjusting of some special solution-, environment- and operation- parameters is required. The presence of beads is also considered as a common problem. The higher concentration of the solution is the most preferable suggestion to solve this problem because it leads to minimizing the number of beads. At the same time, the higher concentration will lead to a larger diameter of the fiber [6].

Figure 1. Basic set up and phenomenon of electrospinning [4].

The selection of the solvent used in electrospinning technique is one of the critical factors for the smooth and beadles' electro-spun nanofiber formation. The chosen solvent must lead to a complete soluble polymer added to moderate boiling point. The boiling point gives an important impression of the solvent's volatility. Generally, volatile solvents are preferred due to their high evaporation rates [7]. Fast evaporation of the solvents should be avoided because low boiling points causes drying the jet and blocking the needle tip, hence hindering the electro-spinning process. Similar to the fast evaporation solvents, low volatile solvents are avoided also because high boiling points prevent the drying during electro-spinning, whereby electro-spun fibers need to reach the collector [8].

The main electro-spinning technique parameters are the flow rate, the distance between the capillary and collector and applied voltage. At low flow rate, there is enough time for the solution to polarize. While, high flow rate leads to beads formation along the fibers, and it causes thick diameter. This is due to short drying periods whereby electro-spin fibers needed to reach the collector [9]. The distance between the tip of the capillary and the grounded plate directly affected the morphology of electro-spun nanofibers. If this distance increased, more uniform and beadles' electro-spin fibers may be obtained. Higher distance provides sufficient time and results in finer fibers with more uniform diameter distribution [10]. According to the voltage of electrospinning device, there are two different effects notified by the electro-spinning researchers. The first effect of electro-spinning voltage is associated with the vast majority of researchers when the electro-spinning voltage is increased, the volume of the drops at the tip decreased resulting in receding the Taylor cone. The jet originates from the liquid surface within the tip, and more beading is seen. If the applied voltage increased, the diameter of the fibers reduced. In the case of low applied voltages, fibers with no beads are produced (if the electric field is quite enough to overcome the surface tension) [11, 12].
While, some researchers are notified and adopted different and opposite effect depends on the fact that when the applied voltage is high, the coulombic forces were much greater than the viscoelastic forces. This would cause not only the breakage of an over-stretched charged jet during its path to the target but also the charged jet travelled to the grounded target in a shorter time. Therefore, the solvent has less time to evaporate, and this leads to bigger but irregular fiber diameters [13]. Marilena V. et al explained that there is a critical voltage. If the applied voltage during electrospinning is less than a critical voltage, increasing electrospinning voltage leads to decrease the diameters. While, when the applied voltage is higher than the critical value, the fiber diameters are increased, Taylor cone size and the drop’s size at the needle tip are decreased and the jet velocity is decreased [14]. Electro-spun fibers in (nano or micro) scale have versatile spread applications such as solar cells, air filtration membranes, gas and chemical sensors, piezoelectric sensors, fuel cells, energy storage and ion exchange membranes [15], self-cleaning membranes, anti-corrosion, responsive smart materials, anti-icing, micro- and nano-electromechanical systems [16].

The aim of this research depends on utilizing different electrospinning voltages (10, 12, 15, 18, 24, and 30) KV in order to study their effects on the thickness of the electro-spin membranes and the topographical and morphological properties especially on fiber diameters and the general histogram of electro-spin fibers.

2. Experimental part (Preparation of PS electro-spun membranes)

Expanded polystyrene solutions and membranes were prepared by same method mentioned in our previous work [17, 18]. Expanded Polystyrene solutions of 30 wt. % were prepared by dissolving expanded polystyrene in (N, N-dimethylformamide DMF) at 100 °C with a continuous stirring for (20-25) minutes to ensure complete dissolution and homogeneous solution. Fabrication of PS electro-spin membranes was performed by electrospinning technique. The Polymer solution was injected in a (10 ml) syringe pump at fixed electro-spinning parameters. These parameters include the flow rate was 1.0 ml/h, the time of the electro-spinning for each membrane was 1.5 h and the distance between the syringe needle and the collector was 12 cm. Different electrospinning voltages (10, 12, 15, 18, 24, and 30) KV were experimented to study the effect of the voltage on the electro-spin membranes. Then, the obtained electro-spin membranes were removed from the collector, and the membranes were cut into small pieces (2 x 2) cm for subsequent characterizations.

3. Characterization of sulfonated electrospun membranes (SPS):

3.1. Fourier transform infrared spectroscopy (FTIR)

It is a non-destructive technique used for yielding information about the chemical bonds which in turn lead to the chemical composition of present membranes. FT-IR spectrum is recorded for the absorption of electromagnetic radiation of the sample in the range of the wave numbers from (4000 - 400) cm⁻¹ [19]. FTIR was used to characterize the appearance of the chemical bands. The basic peaks related to the chemical structure of PS (C₈H₈)n. FTIR can qualitatively identify the structure of unknown materials and the quantitative measurement of the components in a complex mixture [20].

3.2. Scanning electron microscopy (SEM).

The scanning electron microscope (SEM) is considered as one of the most versatile instruments for the examination and analysis of the microstructure characteristics of solid objects. Scanning electron microscopy (SEM) gives information about topography and morphology of the electro-spin fibers [21].

4. Results and discussion:

4.1. Fourier transforms infrared spectroscopy (FTIR):
FTIR of (30 wt.%) expanded polystyrene membrane appeared clearly in fig. (2). Generally, based on the wavenumber, there are three main regions. Region (1) shows that the bands at (400-900) cm\(^{-1}\) associated with (out-of-plane) C-H bend vibrations of aromatic rings. Region (2) defines the bands at (900-2000) cm\(^{-1}\) associated with the vibration of substituted benzene rings and in-plane C-H deformations. Region (3) represents the bands at (2800-3200) cm\(^{-1}\). These bands are related to aliphatic and aromatic C-H stretching [23].

The principle peaks associated with the chemical bonds of the expanded polystyrene are the peak appeared at a wave number of 695 cm\(^{-1}\) which is related to bending (=C-H) out of the plane, the peaks at (754, 842, 905) cm\(^{-1}\) which related to substitution of the benzene ring, while the peaks at (1451 and 1492) cm\(^{-1}\) are associated with aromatic (C=C) stretching. The peak appeared at 2920 cm\(^{-1}\) belongs to stretching vibrations of (-CH\(_2\)), the peak at 3025 cm\(^{-1}\) is associated with aromatic and aliphatic (C-H) stretching, and the peaks at (1028, 1069, 1154, 1181) cm\(^{-1}\) are related to in-plane C-H bending of the phenyl ring [24,25, 26]. The (in-plane C–H bending) of the phenyl ring is usually weak in most aromatic compounds, and it is observed at (1028, 1069, 1154, 1181) cm\(^{-1}\) [27]. The peaks at 3082, 3060 and 3026 cm\(^{-1}\) correspond to the aromatic C-H stretching vibrations, while the bands at 2923 and 2848 cm\(^{-1}\) come, respectively, from the asymmetric and symmetric stretching vibrations of methylene groups (-CH\(_2\)) [23].

**Figure 2.** FTIR spectra for electrospun expanded polystyrene

4.2. **Scanning electron microscopy (SEM).**

From figs. (3, 4, 5, 6, 7, 8), the higher the voltage during electrospinning, the greater the average fiber diameter. This result is completely different with a very large number of researches specialized in the field of electro-spinning, which confirm the decrease in the fiber’s diameters if the applied voltage increases [28, 29]. To explain the reason for increasing the fiber’s diameter the applied voltage, it is important to notice that DMF is a solvent with high dielectric constant properties. The dielectric constant is a measure of how a material placed in an electric field can concentrate effectively the flux the electrostatic lines. So, with high dielectric properties, the fiber’s diameter decreased, and the density of the surface charge on the jet largely dispersed resulting in the production of fibers with uniform morphologies and less diameter along with lower bead formation [30]. It is important to mention a special case during polystyrene electro-spinning. Only at the first few seconds of the electrospinning process, the fibers are gathered on the collector and form a very thin film. After few
seconds, the formation of the thin film on the collector is stopped, and the fibers vertically gathered towards the needle. During the path of the fiber to the collector (which is usually a conductive material), the fibers were cut and did not continually extended. The fibers stop gathering on the collector and start gathering on each other.

This complex is attributed to the special properties of (PS/DMF) solution where the electro-spun fibers prefer the surface of each other due to the conductive surfaces and shorter distances. This case was somewhat little happening at small voltages, but increases with increasing the applied voltage during electro-spinning. It is important to mention that the fibers were cut into small lengths before reaching the collector. This reduces the distance between the needle and the collector that causes the formation of larger average fiber diameters. The fibers gathered on the collector after few seconds of starting the electro-spinning process, and the fibers have lower diameters due to large distance, then the diameters gradually increased. In other words, the interpretation of increasing the fiber diameter is due to the segmentation of electro-spun fibers when the distance between the collector and the needle decrease in such away making the diameter becoming higher. This explanation was in good agreement with Siqi Huan et al [13].

Table (1) clarifies the relationship between increasing the average fiber’s diameter with increasing the electro-spinning voltage. It is important to mention that there is not any membrane obtained when the electro-spinning voltage was 10 KV since it is very small voltage to form the electro-spun fibers. Also, when the electrospinning voltage was 30 KV, it is difficult to perform the electro-spun due to the high repulsion forces between the fibers themselves.

![SEM image of 30 wt.% EPS prepared at 12 KV](image1)

![Histogram of 30 wt.% EPS prepared at 12 KV](image2)

**Figure 3.** 30 wt.% EPS prepared at 12 KV, A:SEM image B:Histogram.

![SEM image of 30 wt.% EPS prepared at 15 KV](image3)

![Histogram of 30 wt.% EPS prepared at 15 KV](image4)

**Figure 4.** 30 wt.% EPS prepared at 15 KV, A:SEM image B:Histogram.
Figure 5. 30 wt.% EPS prepared at 18KV, A:SEM image B:Histogram.

Figure 6. 30 wt.% EPS prepared at 24 KV, A:SEM image B:Histogram.

Figure 7. 30 wt.% EPS prepared at 30 KV, A:SEM image B:Histogram

Table 1. Electro-spinning voltages with average fiber diameter of EPS membranes

| Electrospinning Voltages (KV) | Average Fiber Diameter (μm) |
|-----------------------------|----------------------------|
| 10                          | There is no electrospun membrane at this voltage |
| 12                          | 1.25 ± 0.25                 |
| 15                          | 1.75 ± 0.25                 |
| 18                          | 2.25 ± 0.25                 |
| 24                          | 4.25 ± 0.25                 |
| 30                          | 4.75 ± 0.25                 |
4.3. Thickness test
Table (2) illustrates decreasing the membranes thickness with increasing the voltage of electrospinning device. With constant conditions of electro-spinning process, the thickness of the electrospun membranes was decreased with increasing the applied voltage during the electro-spinning. Decreasing the membranes thicknesses may be related to increasing the electrical conductivity of the polymeric solution with increasing the applied voltage. Lower deposition rate of electro-spun fibers on the collector was attributed to polymeric fibers repulsion in higher conductive solutions that leads to spread over large areas and finally the produced film will be thinner. Our results agreed with [31, 32, 33]. Indeed, any enhancement for the electrical conductivity for polymeric solutions results in higher charge density on the surface of the charged jet, and this encourages or promotes the solution jet stretching as a consequence of higher level of charges.

It is important to mention that the dielectric constant of the electro-spinning solvents plays a very important role in limiting the amount of available free charges in electro-spinning solution. By using low dielectric constant solvent such as chloroform, lower amount of charge density will result and therefore the available charge on the jet surface reduces and weakens the electrostatic repulsive force (34, 35, 36).

In contrast, our results depend on the use of DMF solvent with high dielectric constant that causes higher charge density on the jet surface and encourages the electrostatic repulsive forces. Therefore, with increasing the electro-spinning voltages along with the effect of high dielectric constant of DMF, higher repulsive forces and lower fibers deposition rate was obtained.

Table 2. Relationship between thickness of electrospun membranes and electrospinning voltage

| Electrospinning Voltages (KV) | Thickness of produced electrospun membranes (Cm) |
|-------------------------------|-----------------------------------------------|
| 10                            | There is no electrospun membrane at this voltage |
| 12                            | 0.1379                                        |
| 15                            | 0.113                                         |
| 18                            | 0.09889                                       |
| 24                            | 0.08167                                       |
| 30                            | 0.0511                                        |

5. Conclusions:
The experimental results of this paper indicate the following conclusions for EPS electro-spun membranes.
1. The average fiber’s diameter of the electro-spun membranes increases with increasing the electro-spinning voltage.
2. There is no electro-spun membrane produced with 10 KV
3. The thickness of the electro-spun membranes decreases with increasing the electro-spinning voltage and becomes difficult to produce electro-spun membranes.

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