The Effect of Electrospinning Parameters on Morphological and Mechanical Properties of PAN-based Nanofibers Membrane

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Abstract:

The electrospun nanofibers membranes (ENMs) have gained great attention due to their superior performance. However, the low mechanical strength of ENMs, such as the rigidity and low strength, limits their applications in many aspects which need adequate strength, such as water filtration. This work investigates the impact of electrospinning parameters on the properties of ENMs fabricated from polyacrylonitrile (PAN) solved in N, N-Dimethylformamide (DMF). The studied electrospinning parameters were polymer concentration, solution flow rate, collector rotating speed, and the distance between the needle and collector. The fabricated ENMs were characterized using scanning electron microscopy (SEM) to understand the surface morphology and estimate the average fiber sizes. The membrane porosity percentage was measured using the dry-wet weight method. Also, a dynamic mechanical analyzer was used to determine the mechanical strength properties (tensile strength and Young's modulus) (DMA). The obtained results revealed that the polymer concentration and flow rate mainly affect the porosity and fiber size in ENMs. Increasing the polymer concentration improves the strength and flexibility, while the flow rate did not show a clear effect on the mechanical strength of ENMs. Both fibers collecting speed and spinning distance did not clearly impact the membrane morphology. ENMs flexibility significantly increased with increasing the collector speed and decreasing the spinning distance. Strong and flexible ENMs with small fibers can be fabricated using 10% PAN/DMF at a flow rate of 1 mL/h, collector speed of 140 rpm, and spinning distance of 13 cm.

Keywords: Electrospinning, Flexibility, Nanofibers, Polyacrylonitrile, Strength.

Introduction:

Electrospinning is a cost-effective technique to form continuous fibers within a nanosize scale from natural and synthetic polymers under electrostatic forces[1]. The electrospun nanofibers have outstanding characterizations such as a vast surface area-to-volume ratio, flexibility in surface functionalities, intrinsically high porosity, fully interconnected pore structures, low hydraulic resistance, and ease of scalable synthesis [2]. Thus, electrospun nanofibers membranes (ENMs) have been used in various applications due to their unique characterizations and promising potentials such as scaffolds in tissue engineering [3], electrical storage [4], drug delivery [5], and oil removal[6]. In addition, the ENMs are increasingly considered good candidates for applications that need high permeability and low energy cost.

However, despite the potential properties mentioned above, ENMs have poor mechanical properties such as tensile strength at breaking point, rigidity, and elongation, restricting the application of the nanofibers in many aspects [7]. The low mechanical strength of nanofibers membrane is attributed to their highly porous structure and the weak bonding between the nanofibers [8]. The intrinsic fiber modulus and small-fiber size also contribute to the low strength of these membranes [9]. As a result, using ENMs in applications that require sufficient mechanical strength is limited because of their incapability to withstand operational conditions.

Previous studies have described various aspects to improve the mechanical strength of ENMs, including embedding particles within the raw polymer solution such as graphite Nano platelets [10],

carbon nanotubes [11], and silica nanoparticles [12]; however porous nature of ENMs changed. The mat strength also can be improved by enhancing the binding strength at the junction points of the fibers throughout the fiber mat [13,14]. Hydrophilic electrospun nanofibers adopted this method to improve their integrity in water [9]. The other approach is changing the electrospinning process parameters to increase polymer chain orientation and fiber crystal [7]. For example, the strength of fibers can be enhanced by reducing the fiber diameter due to improving the polymer chain orientations in ultrafine fibers [15,16]. However, a decrease in fiber diameter may decrease pore diameter, which may or may not be desired.

In the electrospinning technique, the main key process parameters influencing fiber generation and nanostructure are viscosity, surface tension, the distance between the needle to collector, voltage difference, and solution flow rate. The precursor solution's viscosity and surface tension can be varied by changing the polymer concentration [17]. In other words, the properties of ENMs can be controlled by manipulating electrospinning parameters either in solution (e.g., concentration, viscosity) and/or process (e.g., applied voltage, solution feed rate) to meet the requirements of a specific application [18]. In electrospinning, increasing the orientation and crystallinity of the molecular chain decreases the defects and improves the mechanical property [7]. The effect of these parameters on the structure and mechanical properties of ENMs were not yet well defined in the previous reports [19].

The main objective of this work is fabricating PAN-based ENMs with enhanced mechanical strength by improving the strength of junction areas between the fibers via adjusting the electrospinning parameters. The effect of polymer concentrations, polymer feed rate, collector speed, and the distance between needle tip and collector on producing homogenous, uniform, and strong fiber mats were studied in this research. The morphological and mechanical properties of the prepared PAN-based ENMs were evaluated using the surface morphology, fiber size, porosity, stress at the breaking point, and flexibility of the fabricated membranes.

Materials and Methods:
Electrospinning of Nanofibers Membranes

The polymer Polyacrylonitrile (PAN) (Mwt. of 150,000 g/mole) was purchased from (Sigma Aldrich, Germany). The solvent N, N-Dimethylformamide (DMF) (density of 0.948 g/cm³) was supplied by (Alfa Aesar, Germany). To prepare PAN/DMF solution, a specific amount of PAN was dissolved in DMF and stirred continuously for 4 hr at 50 ºC. All the nanofibers membranes in this work were fabricated using the electrospinning technique, a pulling motion of polymer droplets under a high-voltage electrostatic field. The basic electrospinning setup comprises four main parts: a syringe containing a polymer solution, a metallic needle, a voltage power supply, and a collector [20].

To investigate the effect of the electrospinning parameters on the fabricated PAN-based nanofibers, various membranes were prepared under different electrospinning parameters, including PAN/DMF precursor solution concentration (10, 13, and 16 wt.%), precursor solution injection flow rates (1, 1.5, and 2 mL/hr), collector rotating speed (70, 140, and 210 rpm), the distance between the needle and collector (13, 15, and 17 cm). Depending on our previous work [21], the base electrospinning parameters were 10 wt.% PAN/DMF solution concentration, 1 mL/hr solution flow rate, 70 rpm collector rotating speed, and 15 cm distance between the needle and collector. All the produced ENMs were fabricated at room temperature.

Membrane Characterizations
Membrane surface morphology

Scanning electron microscopy (SEM) is a highly effective technique for characterizing the surface properties of nanofibers membranes, such as nanofibers structures and fiber diameter. It delivers high-resolution pictures using a high-energy electron beam. The average fiber diameters were estimated from the obtained images by assessing the fiber sizes of thirty fibers of each ENMs sample using Image J software (National Institutes of Health, USA).

Porosity

The porosity of a membrane is a very important characterization in many membrane applications, especially the separation performance of membranes. To evaluate the porosity of ENMs membranes, a sample of each membrane was weighed, and then it was immersed in distilled water for 1 hr. The weight of the sample before and after immersing in water was recorded as dry and wet weights, respectively. Then porosity percentage of the nanofibers membranes was calculated using Eq. 1:

$$Porosity\ (\%) = (W_1 - W_2)/A * t * \rho \qquad 1$$

Where: W_1 and W_2 are wet and dry membrane mass (g), A is membrane area (cm²), t is membrane thickness (cm), and ρ: water density at room temperature g/cm³.

Mechanical Properties

The mechanical property of the membrane is an important aspect of the membrane's practical applications like reusability, handling, and anti-deformation capacity [22]. Mechanical properties of the

membranes were evaluated using the breaking strength and Young's modulus of the membrane samples using a Dynamic Mechanical Analyzer (DMA) (AG-A10T, Shimadzu, Japan). The specimen size of 10 cm length and 1 cm width was used for the tests, all performed at 25 °C and ambient humidity. Young's modulus measures the rigidity of an elastic material; it can be defined as the ratio of stress to strain [23]. The flexible materials have low Young's modulus.

Results and Discussion:
The Effect of Solution Concentration

The showed results in Fig. 1 indicated that the fabricated nanofibers at the various PAN/DMF concentrations are uniform beadless, smooth, and long fibers. Fig. 1 showed that the polymer concentration has a significant effect on the average diameter of electrospun PAN-based nanofiber; the average fiber sizes significantly increased (260 to 485 nm) with increasing the PAN/DMF solution concentration (10 to 16 wt.%), which can be explained by increasing the viscosity of the precursor solution [24]. Increasing the concentration and viscosity of the polymeric solution significantly reduces the stretching of the charged jet. Thus, increasing the chain entanglement among the polymer chains that overcome the surface tension and ultimately form thicker fibers [22,25]. The prepared ENMs also showed high porosities mainly due to the entangled randomly-oriented fibers in the electrospun nonwoven membrane structure [22]. Fig. 2 clarifies increasing the average fiber sizes and porosity with increasing the polymer concentration, which can be attributed to the large size of the fibers and settling the heavier fibers into a tighter mat during electrospinning [26].
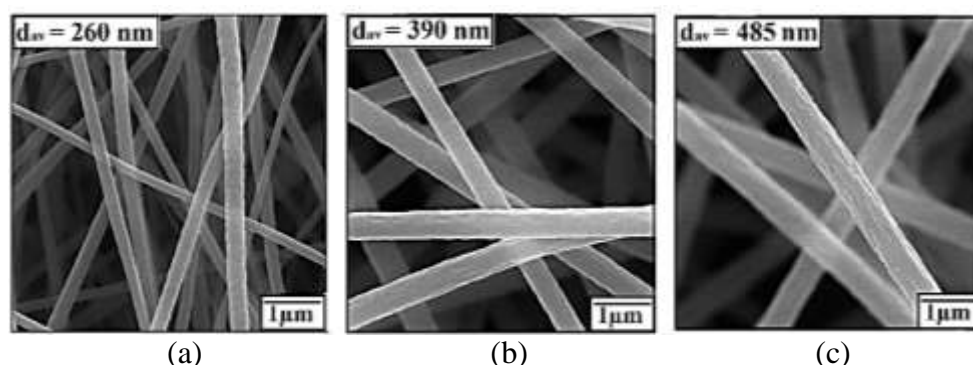

(a)   (b)   (c)

Figure 1. The SEM images of PAN-based ENMs using various concentrations of PAN (a) 10 % PAN/DMF, (b) 13% PAN/DMF, and (c) 16% PAN/DMF
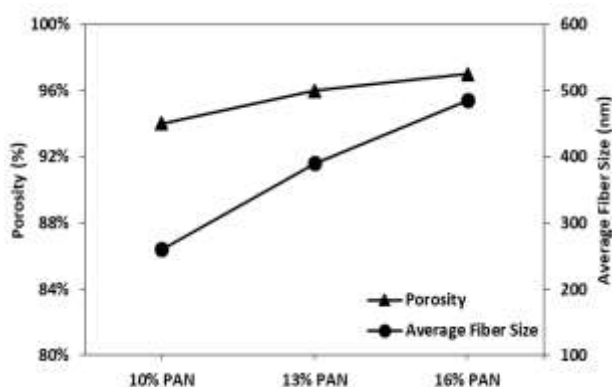

Figure 2. The porosity and average fibers size of fabricated PAN-based ENMs at various concentrations of PAN solutions

Fig. 3 shows the tensile strength and Young's modulus of the fabricated ENMs as a function of polymer concentration. These results showed increasing the tensile strength and decreasing Young's modulus of ENMs with increasing polymer concentration. This is attributed to increased fiber size and decreased defects with increased polymer concentration, improving the breaking strength and flexibility [7]. Polymer chains per fiber increase chain interaction and entanglement at higher polymer concentrations. Also, the larger fibers contain a more residual solvent in their core than smaller fibers; thus, the evaporation of the solvent facilitates the bonding of fibers, which improves the strength of ENMs [24].
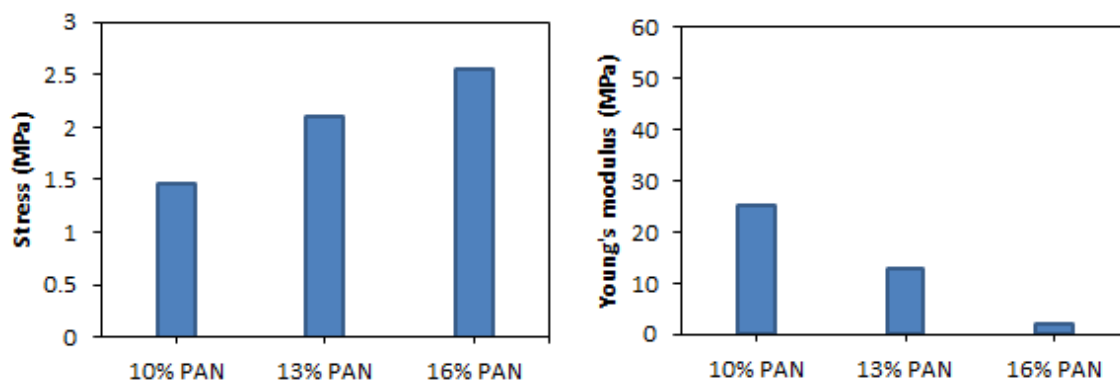
Figure 3. Mechanical properties (breaking strength and Young's modulus) of PAN-based ENMs using various PAN concentrations

The Effect of Solution flow rate

The flow rate of the precursor solution through the needle controls the morphology of the electrospun nanofibers. Fig. 4 shows that raising the injection rate (1 to 2 mL/hr) increased the fiber size (260 – 410 nm) due to increasing the volume and initial radius of the electrospinning jet. Uniform beadless electrospun nanofibers could be fabricated using a critical flow rate of the precursor solution. When the solution feed rate is too high, periodic dripping occurs due to insufficient drawing of the solution away from the nozzle tip and causing the formation of the beads [27]. Exceeding the critical value of the polymer flow rate results in the non-evaporation of the solvent and low stretching of the solution between the needle and the collector [28]. Also, increasing the polymer flow rate decreases the bending stability, subsequently increasing the porosity, as shown in Fig. 5.
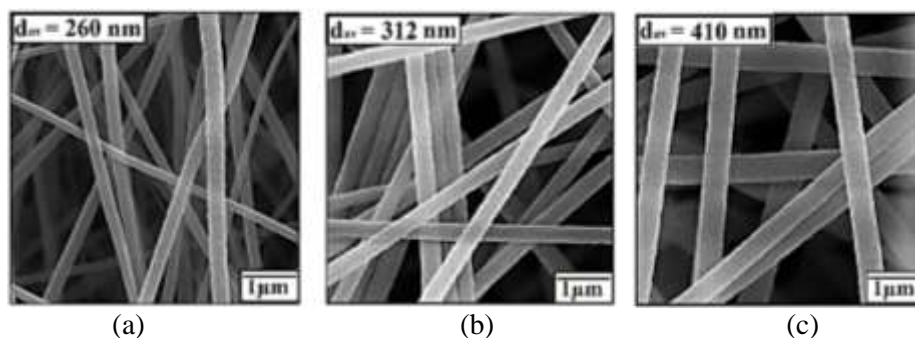

(a)   (b)   (c)

Figure 4. The SEM images of 10% PAN/DMF ENMs at various precursor solution injection rates (a) 1 mL/hr, (b) 1.5 mL/hr, and (c) 2 mL/hr
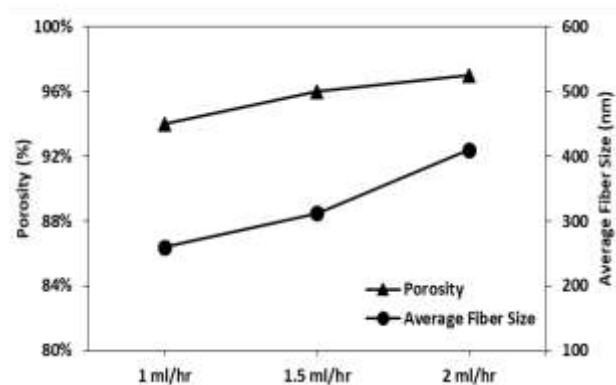

Fig. 6 showed that increasing the flow rate does not significantly impact the strength of the ENMs fabricated. Decreasing Young's modulus can be attributed to the non-evaporation of the solvent and low stretching of the solution between the needle and the collector at the high polymer flow rate [7].
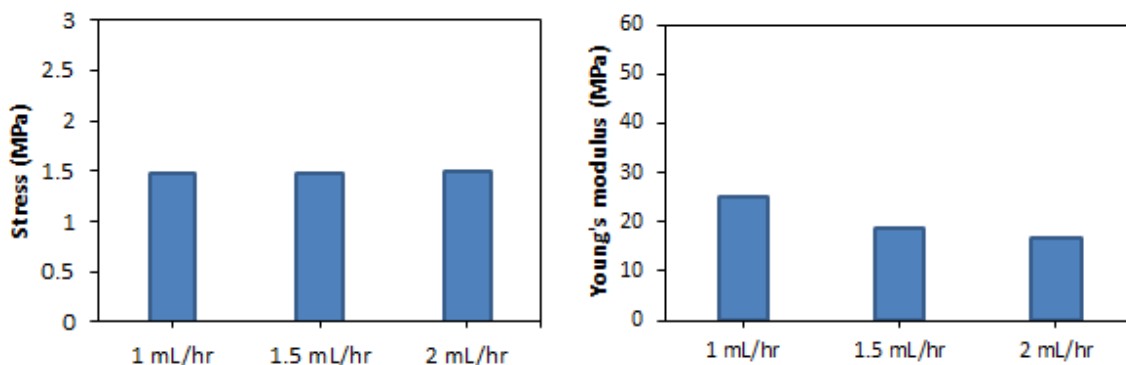
Figure 5. The porosity and average fibers size of fabricated 10% PAN/DMF ENMs using various injection rates

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Figure 6. Mechanical properties (breaking strength and Young's modulus) of 10 % PAN/DMF ENMs using various solution injection rates

The Effect of Fiber Collecting Speed

The collector speed significantly affects the productivity and arrangement of the collected electrospun nanofibers due to its influence on the ability of the charges on deposited fibers to be conducted to the ground, which in turn affects the amount of collected fibers on the substrate. Fig. 7 showed that increasing the rotation speed (70 to 210 rpm) reduced the size of the fibers (260 to 175 nm).

This can be endorsed to the mechanical drawing of the fiber as it is deposited on the collector. However, beads formation can be noticed at the high collector speed (210 rpm) because of the breakage of some of the collected fibers. Also, increasing the collector speed decreases the porosity of the fabricated ENMs, as shown in Fig. 8, due to the smaller average fiber size.
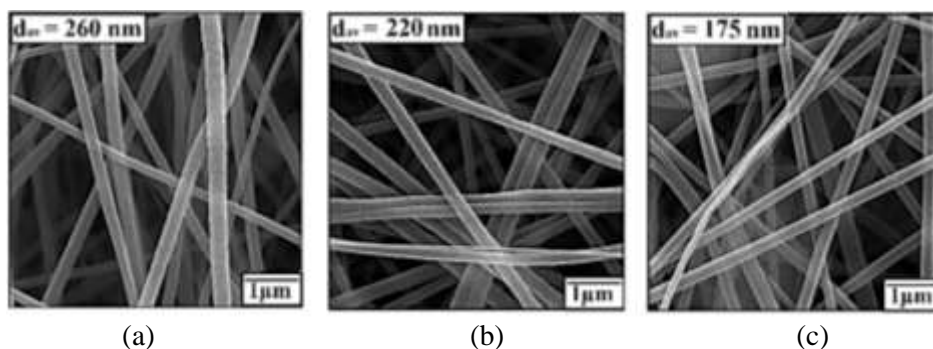

(a)   (b)   (c)

Figure 7. The SEM images of 10% PAN/DMF ENMs at various collecting speeds (a) 70 rpm, (b) 140 rpm, and (c) 210 rpm
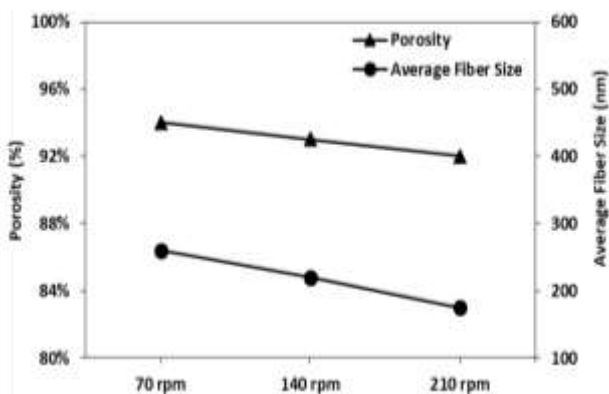

Figure 8. The porosity and average fibers size of fabricated 10% PAN/DMF ENMs at various fiber collecting speeds

Fig. 9 indicates that increasing the collector speed resulted in a slight increase in the breaking strength. This can be attributed to the fact that most of the fibers are not stretched simultaneously in the same direction, resulting in differences in the arrangement and orientation of the fibers [7]. Young's modulus showed a noticeable decrease with increasing the collector speed from 70 to 140 rpm, while the higher speed (210 rpm) raised the modulus value. Although the extremely high collecting speed can produce aligned and oriented nanofibers along the axis of rotation, it can result in necking, fiber breakage, and the beads resulting in poor mechanical strength of the ENMs [28,29].
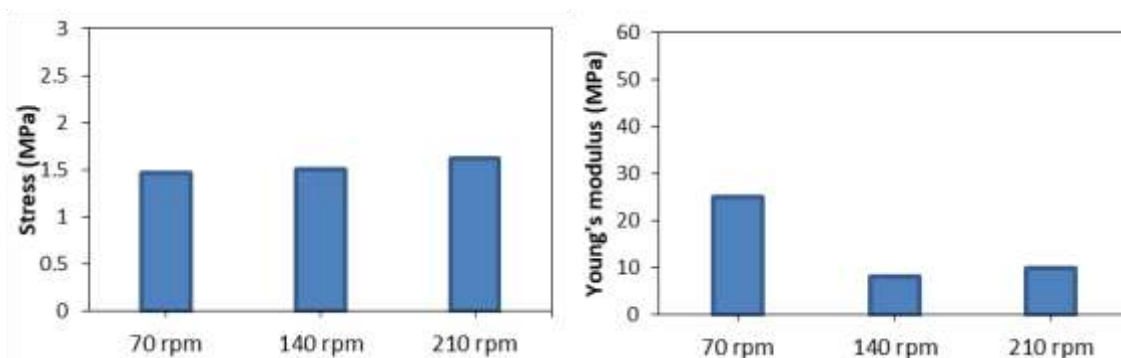
Figure 9. Mechanical properties (breaking strength and Young's modulus) of 10 % PAN/DMF ENMs at various collecting speeds

The Effect of Distance between the Needle and Collector

The distance between the needle tip and collector could easily affect the nanofiber morphology because it depends on the deposition time, evaporation rate, and whipping or instability interval [30]. The distance between the tip and collector affects the jet path and the traveling time before resting on the collector, allowing a sufficient flight time for the solvent to vaporize. Fig. 10 illustrates the effect of the distance between the needle tip and the collector; increasing the distance (13 to 17 cm) led to decreased fiber size (360 to 245 nm). This result can be attributed to the greater stretching distance at the fiber diameter's more considerable distance [31]. The porosity decreases slightly with increasing the distance (as shown in Fig. 11), possibly because heavier fibers settle into a tighter mat during electrospinning. Increasing the spinning distance resulted in less porosity due to reducing the fiber size
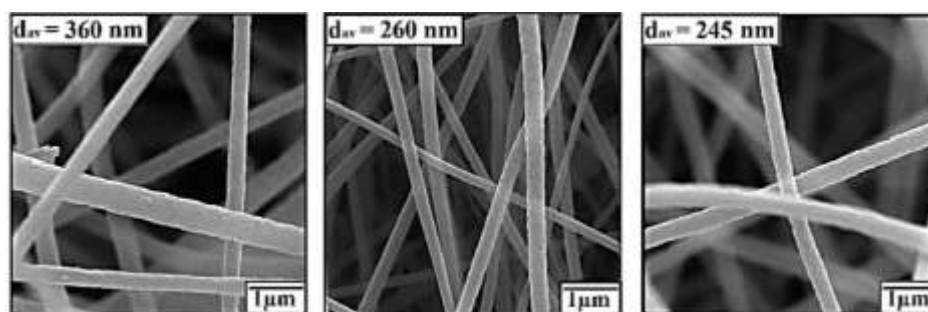

Figure 10. The SEM images of 10% PAN/DMF ENMs at various distances between the needle tip and the fiber collector (a) 13, (b) 15, and (c) 17 cm
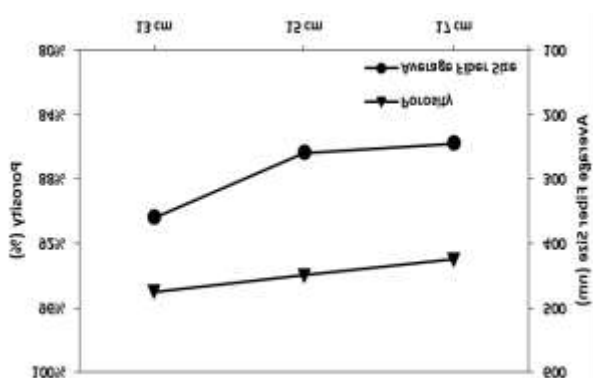

Figure 11. The porosity and average fibers size of fabricated 10% PAN/DMF ENMs at various distances between the needle tip and the fiber collector

Fig. 12 indicated that increasing the collecting distance during electrospinning, can encourage solvent evaporation, cool down the fiber jet flow, and enhance the crystallinity of the fiber leading to improving the membrane's mechanical properties. The crystallinity of the fiber is improved with increasing the spinning distance due to an increase in the jet stretching time [7].
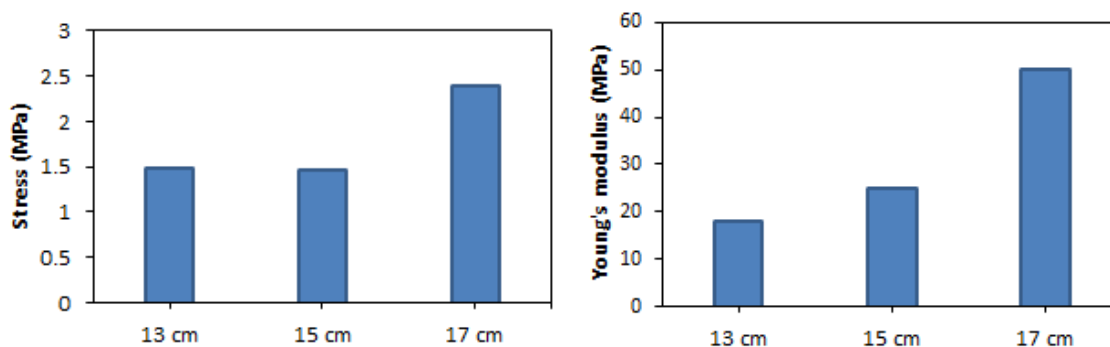
Figure 12. Mechanical properties (breaking strength and Young's modulus) of 10 % PAN/DMF nanofibers membrane at various distances between the needle tip and the fiber collector

The Membrane at the Best Fabrication Parameters

According to the obtained results, the morphological and mechanical properties of PAN-based ENM were adjusted to produce material with the necessary strength for certain applications such as water purification. Although the fabricated ENM using 10% PAN/DMF solution has the lowest breaking strength, it has the highest flexibility. As a result, the 10 PAN/DMF was chosen as one of the best fabrication parameters that give the most flexible membrane and smaller fibers size. Increasing the solution flow rate did not significantly affect the membrane strength; however, the fiber size and porosity were increased significantly. Thus, the best flow rate of the PAN/DMF solution was chosen at 1 mL/hr flow rate. The speed of the fiber collector drum showed a clear impact on the fiber size and the flexibility of the produced ENMs. However, increasing the collector speed to 210 created beads within the fibers and reduced the breaking strength. So, drum speed at 140 rpm was chosen as the best value that produces strong and flexible ENMs. Regarding the distance between the needle and collector, the ENMs fabricated using 17 cm had the highest breaking strength, while its flexibility was the lowest. So, the distance of 13 cm was chosen as the best spinning distance that produces the less rigid nanofibers membrane.

Table 1 shows the effect of the selected best electrospinning parameters on the properties of the fabricated ENMs and compares it with that fabricated at the base parameters. The selected parameters did not clearly impact the fiber size and porosity of the fabricated ENMs, while the mechanical strength (stress at the breakpoint and flexibility) obviously improved.

Table 1. The fabricated ENMs characterizations at the base and best electrospinning parameters

The fabrication parameters	Average fiber size (nm)	Porosity (%)	Breaking stress (MPa)	Young's modulus (MPa)
At the base parameters (10 % PAN/DMF, 1 mL/h, 15 cm, and 70 rpm)	260	94	1.47	25
At the best parameters (10 % PAN/DMF, 1 mL/h, 13 cm, and 140 rpm)	280	94	1.7	16

Conclusion:

In this study, PAN-based electrospun nanofibers were fabricated under various electrospinning parameters. Polymer concentration, polymer solution flow rate, rotating collector speed, and the distance between the needle and collector have been selected to see their effects on the morphology, porosity, and mechanical strength of the ENMs. Increasing the polymer concentration (10 to 16 wt.%) improved the mechanical strength of ENMs, while the average fiber sizes increased significantly. However, increasing the solution flow rate did not significantly impact the mechanical properties of the fabricated membranes. For the rotating collector speed, increasing the speed (70 rpm to 140 rpm) resulted reducing in the size of the fibers (260 to 220 nm) while the strength and flexibility increased.

Further increase in the collector speed resulted in creating beads within the fibers and decreasing the membrane strength. Increasing the distance between the needle and collector (13 to 17 cm) led to decreased fiber sizes (360 nm to 245 nm) with increasing tensile strength; however, the membrane rigidity increased. According to the obtained results, the best electrospinning parameters to fabricate strong and flexible ENMs with small fibers are 10% PAN/DMF, a flow rate of 1 mL/h, a spinning distance of 13 cm, and a collector speed of 140 rpm.

Authors' declaration:
- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

Authors' contributions statement:
H. S. A.: Drafting the MS, performed the xyz experiments, acquisition of data, and analysis. B. I. W.: Supervision, designed the study, revision, and proof reading

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تاثير معاملات البثق الكهربائي على الخصاص السطحية والميكانيكية لاغشية الالياف النانوية المصنوعة من بوليمر البولي اكريلونايتريل

بسمة اسماعيل ويسي

حنين صبيح الكعيدي

قسم الهندسة الكيمياوية، كلية الهندسة، جامعة بغداد، بغداد، العراق.

الخلاصة:

حظيت أغشية الألياف النانوية المنبثقة كهربائيا باهتمام كبير بسبب أدائها المتفوق. ومع ذلك، فإن القوة الميكانيكية الضعيفة للألياف النانوية المنبثقة كهربائيا، مثل الصلابة وقوة الكسر، تحد من تطبيقاتها في العديد من الجوانب التي تحتاج إلى قوة كافية ، مثل تنقية المياه. يبحث هذا العمل في تأثير معاملات الغزل الكهربائي على خصائص الألياف النانوية المنبثقة كهربائيا المصنعة من بوليمر البولي اكريلونايتريل والمذاب في المحلول في ن-ن-ديميثايلفور مامايد. كانت معاملات الغزل الكهربائي المدروسة هي تركيز البوليمر، ومعدلات تدفق المحلول، وسرعة دوران المجمع، والمسافة بين الإبرة والمجمع. تم تحليل الألياف النانوية المنبثقة كهربائيا باستخدام الفحص المجهري الإلكتروني لفهم شكل السطح وحساب متوسط أحجام الألياف. تم قياس نسبة مسامية الغشاء باستخدام طريقة الوزن الجاف والرطب. أيضًا ، تم تقييم خصائص القوة الميكانيكية (قوة الشد ومعامل يونغ) باستخدام محلل ميكانيكي ديناميكي. أوضحت النتائج المتحصل عليها أن تركيز البوليمر ومعدل التدفق يؤثران بشكل رئيسي على حجم الألياف ومساميتها في الألياف النانوية المنبثقة كهربائيا. تعمل زيادة تركيز البوليمر على تحسين القوة والمرونة، بينما لم يُظهر معدل التدفق تأثيرًا واضحًا على القوة الميكانيكية للألياف النانوية المنبثقة كهربائيا. لم تؤثر كل من سرعة تجميع الالياف ومسافة البثق بشكل واضح على شكل الغشاء. زادت مرونة الألياف النانوية المنبثقة كهربائيا بشكل كبير مع زيادة سرعة تجميع الالياف وتقليل مسافة البثق. يمكن تصنيع اغشية الألياف النانوية المنبثقة كهربائيا قوية ومرنة بألياف صغيرة باستخدام تركيز بوليمرالاكريلونايتريل 10٪ بمعدل تدفق 1 مل / ساعة، وسرعة تجميع 140 دورة في الدقيقة، ومسافة دوران 13 سم

الكلمات المفتاحية : البثق الكهربائي، مرونة، الياف نانوية، بولي اكريلونايتريل، صلابة.