



## Morphology Characterization of Electrospun Polystyrene Membranes

<sup>1,2</sup>Ahmed H. Oleiwi\*, <sup>2</sup>Akram R. Jabur, <sup>3</sup>Qusay F. Alsahy, <sup>4,5</sup>Suriani Abu Bakar

<sup>1</sup>College of Engineering / Wasit University – Iraq

<sup>2</sup>Department of Materials Engineering – University of Technology – Iraq

<sup>3</sup>Department of Chemical Engineering – University of Technology – Iraq

<sup>4</sup>Nanotechnology Research Centre, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris – Malaysia

<sup>5</sup>Department of Physics, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris – Malaysia

### Article information

#### Article history:

Received: June, 28, 2023

Accepted: October, 24, 2023

Available online: March, 10, 2024

#### Keywords:

Electrospinning technique,  
Nano-fibers membranes,  
FE-SEM

#### \*Corresponding Author:

Ahmed H. Oleiwi

[aoleiwi@uowasit.edu.iq](mailto:aoleiwi@uowasit.edu.iq)

### Abstract

The polymeric solution concentration is one of the most critical factors in forming of fibers in electrospinning technology. In this study, various polymeric concentrations of thermoplastic polystyrene are used. At the same time, keeping the other electrospinning process operating parameters (such as flow rate, voltage, capillary to collector distance), solution parameters (such as solution conductivity and molecular weight), and environmental conditions (such as temperature and humidity) constant. Field emission scanning electron microscopy was used to investigate the morphological changes that took place on the surface of the fibres and determine the typical fibre diameter. It was found that as the concentration of polystyrene increased from 15% to 30%, the average pore size increased from (0.5 $\mu$ m and 0.44 $\mu$ m) to (2.7 $\mu$ m and 2.6 $\mu$ m). The FT-IR demonstrated the main chemical bonds in polystyrene membranes and the change in peak intensity caused by increasing the polymeric concentration. According to contact angle measurements, which examine the change in hydrophobic properties, the hydrophobicity of membranes decreases as the water contact angle falls from 135 to 116 with increasing the polymeric concentration from 15% to 30%.

DOI: [10.53293/jasn.2023.7010.1225](https://doi.org/10.53293/jasn.2023.7010.1225), Department of Applied Sciences, University of Technology - Iraq.

© 2024 The Author(s). This is an open access article under the CC BY 4.0 License.

### 1. Introduction

Electrospinning is an efficient, common, and flexible method for producing wide fibers from submicron to nano-scale for various polymeric types [1, 2]. Electrospinning fibers have a wide range applications due to their desirable characteristics, like high aspect ratio, specific surface area, porosity, and good mechanical characteristics [3-5].

The electrospinning method includes applying high potential difference on the capillary needle that contains the polymeric solution. A polymeric droplet is created at the needle tip and produces the “Taylor cone” When

electrostatic forces beat the solution surface tension, the solution ejects from the Taylor cone and moves toward the collector, fibers produced since the solvent evaporated rapidly and then collecting on the collector [6-8]. The fiber morphologies depend on different factors, like applied voltage, flow rate [9], and polymeric solution characteristics that include viscosity, surface tension, viscoelastic properties, and spinning ability [10, 11]. Also, the surrounding conditions like temperature and pressure can affect fiber formation, by rates changing of phase separating and solvent evaporating [12-14]. Polystyrene is a common polymer with widely used applications like producing fibers by electrospinning technique. Polystyrene fiber morphology has a great role in improving the physical and mechanical properties of end products to use in specific applications [15]. This study focused on the effects of polymeric concentration on electrospinning fibers morphology, also the effect of polystyrene concentrations on mechanical behavior and wettability for membranes was investigated. All electrospinning operating parameters were adjusted such as collector to needle distance, polymer flow rate, and electrical voltage.

## 2. Experimental Procedure

### 2.1. Materials

Polystyrene with molecular weight of ( $M_w = 250,000$  g/mol) was purchased from (American polymers services inc. APS, USA). Dimethylformamide was purchased from (Central Drug House (P) Ltd, India).

### 2.2. Membranes Preparation

Polystyrene (PS) in various proportions (e.g., 15, 20, 25, and 30 wt.%) was dissolved in dimethylformamide (DMF) with continuous stirring at room temperature for 120 minutes until the formation of transparent homogeneous solutions, which were termed PS15, PS20, PS25, and PS30, respectively.

In order to create submicron and nanofiber membranes, the produced solutions were electrospun (Bio-electrospinning/Electrospray system ESB-200, South Korea). A needle is connected to a 10 ml syringe for storing solutions, a syringe pump to control feeding, and a collector to gather and store the created material that makes up the apparatus. The electrospinning operating parameters were 20KV voltage, 1ml/h flow rate, and 15cm collector-to-needle distance, while the surrounding environmental variables (temperature and humidity) were similar to those found in a laboratory [16].

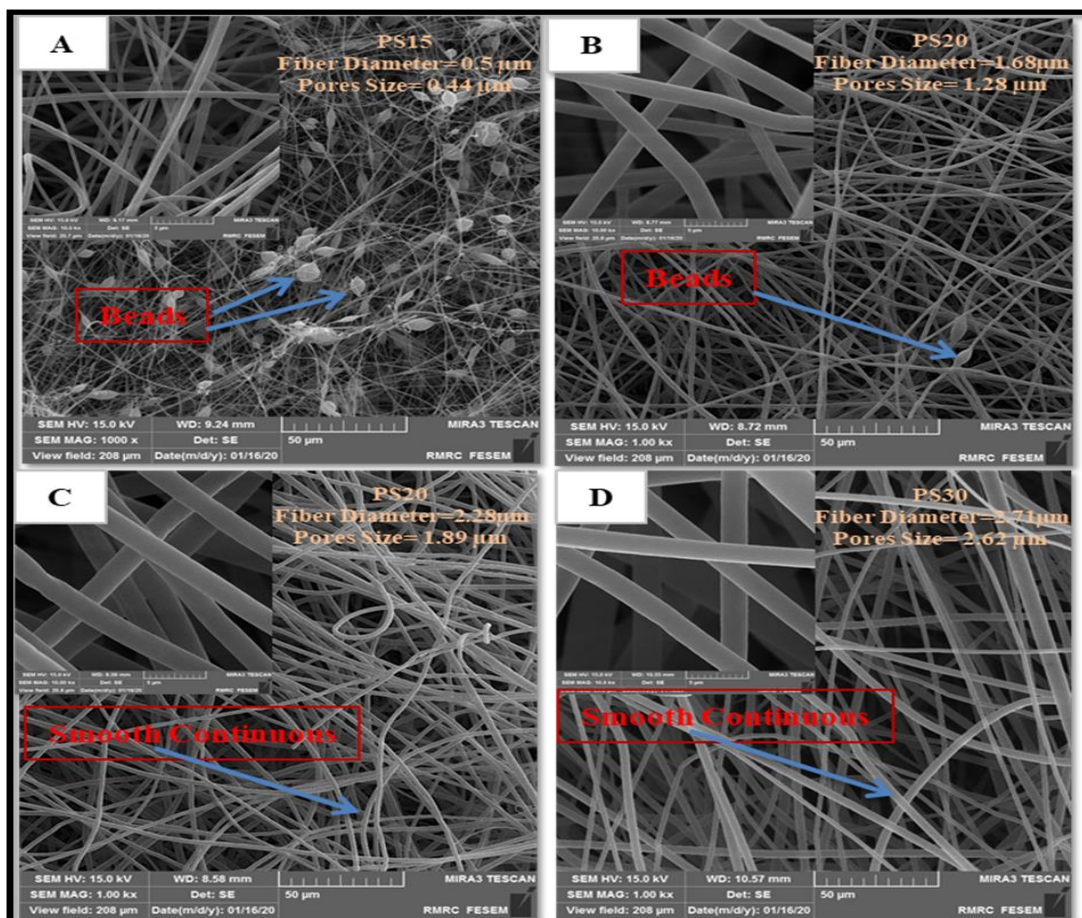
### 2.3. Membranes Characterization

- FE-SEM microscopy (Model: MIRA 3-XMU) was used for morphological characterization of the electrospinning membranes.
- EDX spectroscopy was done with the FE-SEM test to have details about the membrane contents.
- FT-IR Spectroscopy (Model: BRUKER, TENSOR-27), for estimating the bonds in polystyrene electrospinning membranes.
- Wettability Measurement of the membranes can be estimated by measuring the angle between the membrane surface and water droplets (drops onto the membrane surface) using a camera device (type: CAM 110, Germany).
- Mechanical strength measurement by using the tensile mechanical tester (type: Tinus Olsen, H50 KT) used to measure tensile strength and elongation percent at breaking for electrospinning polystyrene membranes.

## 3. Results and Discussion

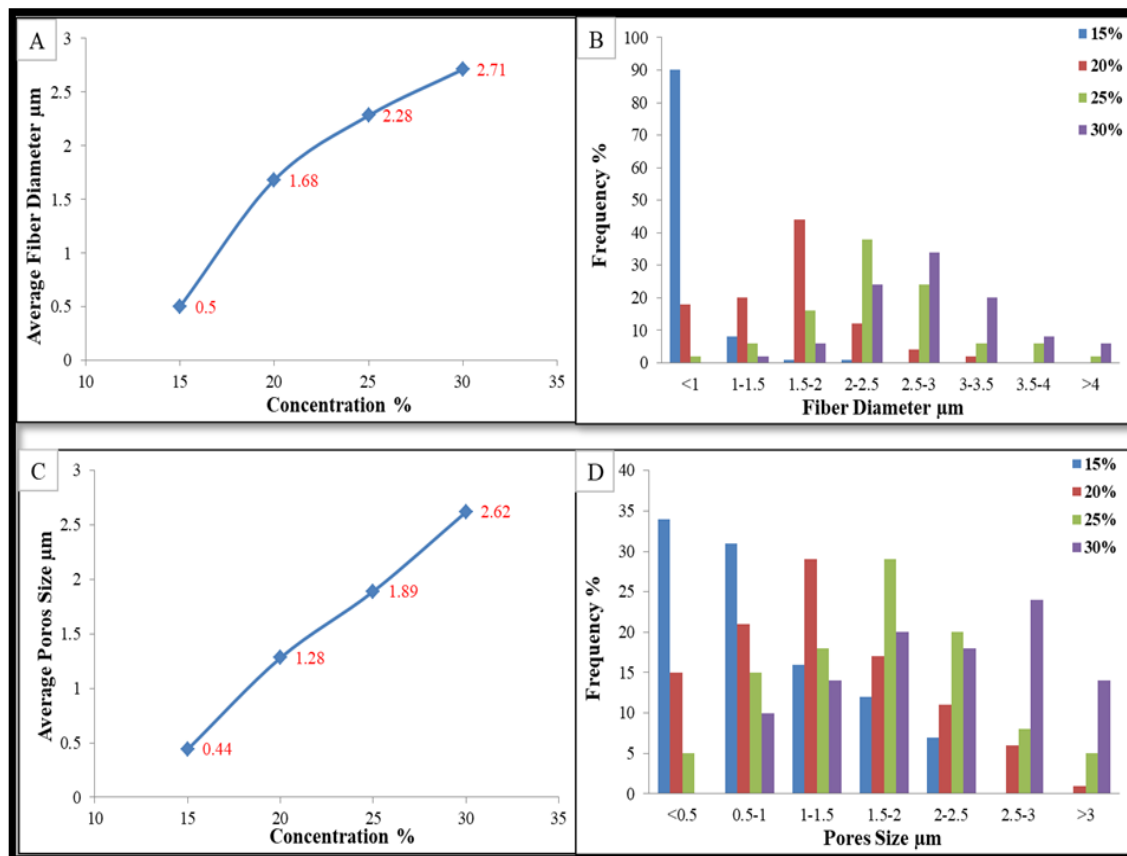
### 3.1. FE-SEM Microscopy

Fig. 1 (A and B) shows the morphologies of PS15 and PS20, respectively, and exhibited structures with bead-on-string because the polymeric concentrations were low, causing incomplete entanglement in chains and causing the solution to break in the form of droplets beads rather than fibers [17]. Fig. 1(C and D) depicts the morphologies of PS25 and PS30, respectively, and displayed structures devoid of beads due to high polymer concentrations causing adequate entanglements in the polymer chains. Electrospinning produces fibers when the solvents in the solutions evaporate [18].



**Figure 1:** FE-SEM images for (15, 20, 25 and 30%) polystyrene concentrations membranes, respectively.

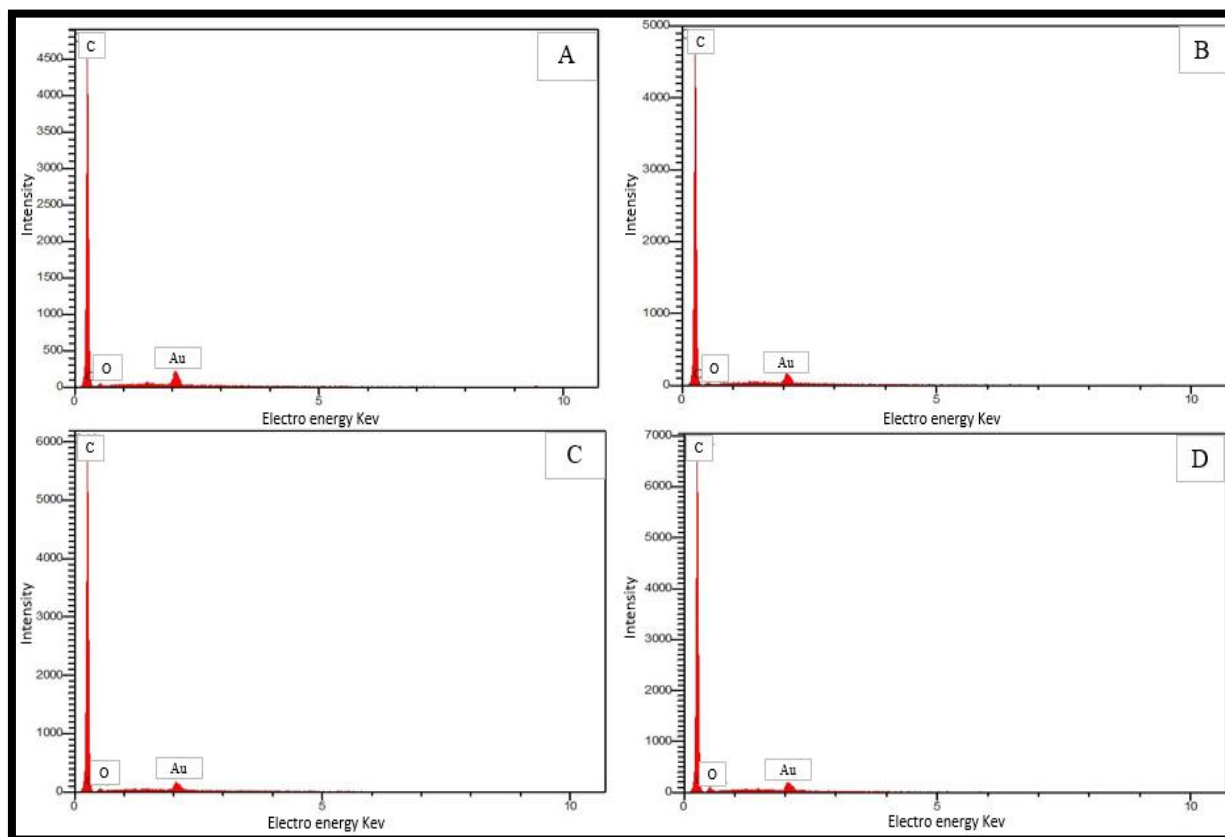
The average fiber diameters of the membranes produced in PS15 and PS30 range from (0.5 - 2.71  $\mu\text{m}$ ), as shown in Fig. (2), which shows the histogram for FE-SEM image analysis. Additionally, it was shown that the diameter of the fibers increased with increasing polymeric concentration. This is because the high viscosity of the ejected jet allowed for sufficient stretching and elongation. Polystyrene membranes made with cotton-structured fibers had lower mechanical properties, though. 15% polystyrene concentration was challenging to get out of the collector. Due to the high humidity in the area, which creates a layer that acts as a skin and covers the surface of the fibers before they reach the collector, it is difficult to maintain its original form. In comparison to highly stable condition, the surface of the fibers is more continuous and smoother due to the increase in solvent evaporation rate as the polymeric solution concentration rises from low to high [19].



**Figure 2:** The fibers diameter, pores size and their distributions for different PS concentrations.

### 3.2. EDX spectroscopy

Fig. 3 shows the chemical composition of the electrospun PS membranes and the analysis of EDX spectroscopy for the membranes. In addition to the gold element detection, which is related to coating the sample with a thin film of gold for a test, it is observed that these membranes are primarily made of carbon and oxygen, establishing that the fiber structure is made of carbon. The EDX spectrum is more sensitive to heavier than lighter elements, but as the atomic number decreases, sensitivity declines as well, making it impossible to detect light atoms like hydrogen and lithium [17, 19].



**Figure 3:** EDX for (15, 20, 25 and 30%) polystyrene concentrations membranes, respectively.

### 3.3. FT-IR Spectroscopy

The main peaks of FTIR spectra for polystyrene electrospinning membranes are shown in Fig. 4 at concentrations of 15% and 30%, respectively. Start by showing the transmittance peaks at wave numbers 3082.44 and 3025.74  $\text{cm}^{-1}$ , which are connected to the stretched vibration of the C-H bond in an aromatic ring. The presence of benzene rings is also suggested by transmittance peaks at wave numbers 1601.23, 1492.73, and 1451.83  $\text{cm}^{-1}$  that are linked to the C=C bond in an aromatic ring (stretching vibration). Finally, at 753.69 and 696.43  $\text{cm}^{-1}$  wave numbers, show a transmittance peak connected to an out-of-plane C-H bond (bending vibration), and provide a hint that the benzene ring only contains one substituent. While the 2921.77 and 2850.39  $\text{cm}^{-1}$  wave transmission peaks indicate the presence of the methylene group. All of the current peaks are agreed with Noor M. Jalal [20].

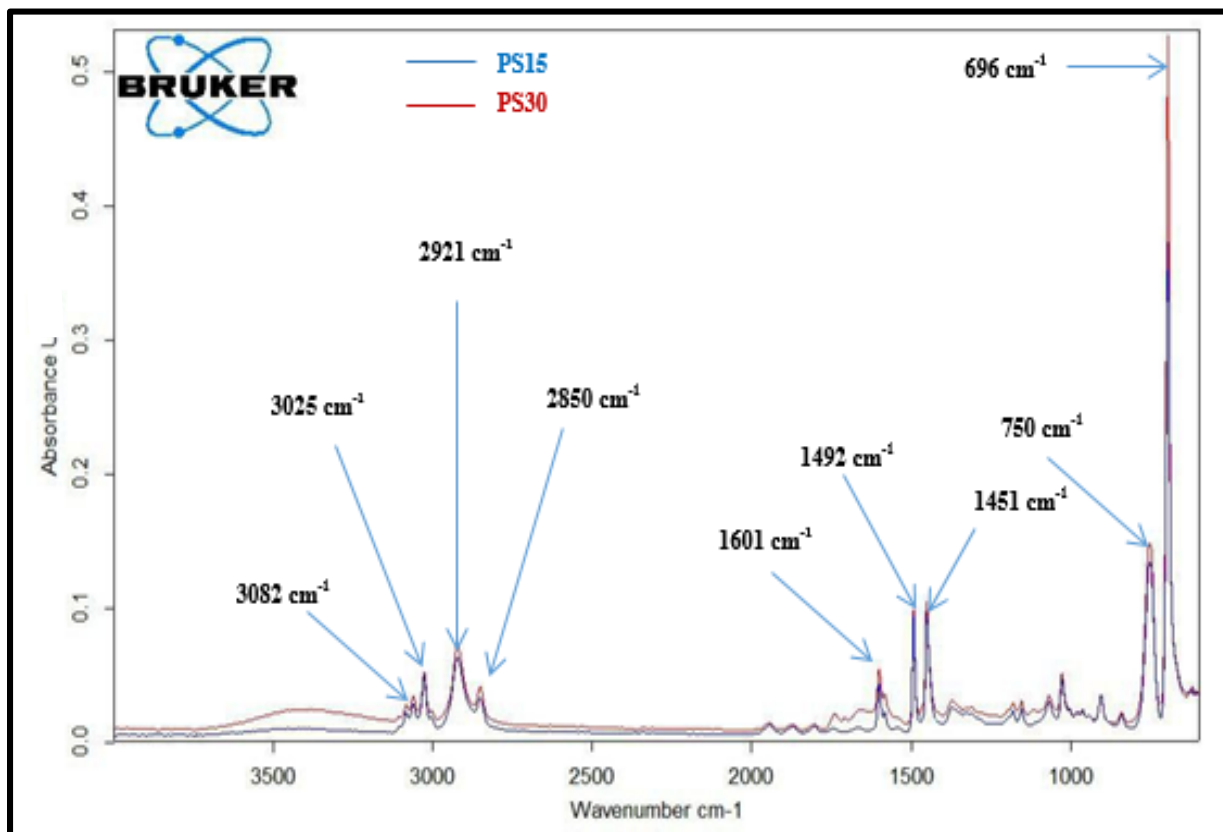


Figure 4: FTIR for (15 and 30%) polystyrene membranes.

### 3.4. Contact Angle Measurement

Fig. 5 depicts the effect of polystyrene concentration on water drop contact angle. It shows a steady decrease in contact angle from  $135^\circ$  to  $116^\circ$  as polymeric content increased from 15% to 30% due to a decrease in beads, which resulted in an increase in pore size [21]. Electrospinning fibers hydrophobic properties are significantly influenced by their shape. In addition to the creation of protuberant structure, beads, and porosity, in manufactured membranes. Due to the fact that these structures changed the fibers surface roughness and increased the amount of air trapped as the polystyrene surface rose. These structures have the advantage of increasing membrane hydrophobicity since the Cassie equation considers their contact angle with air to be  $180^\circ$ . These structures provide a benefit for improving membrane hydrophobicity [22].

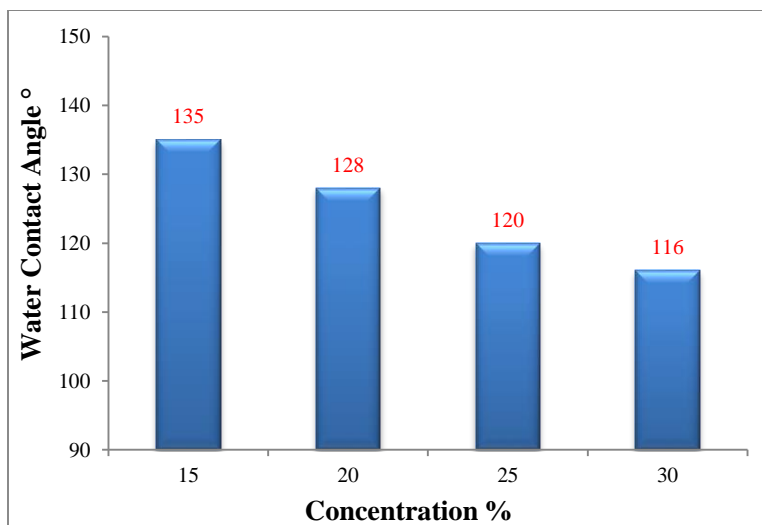
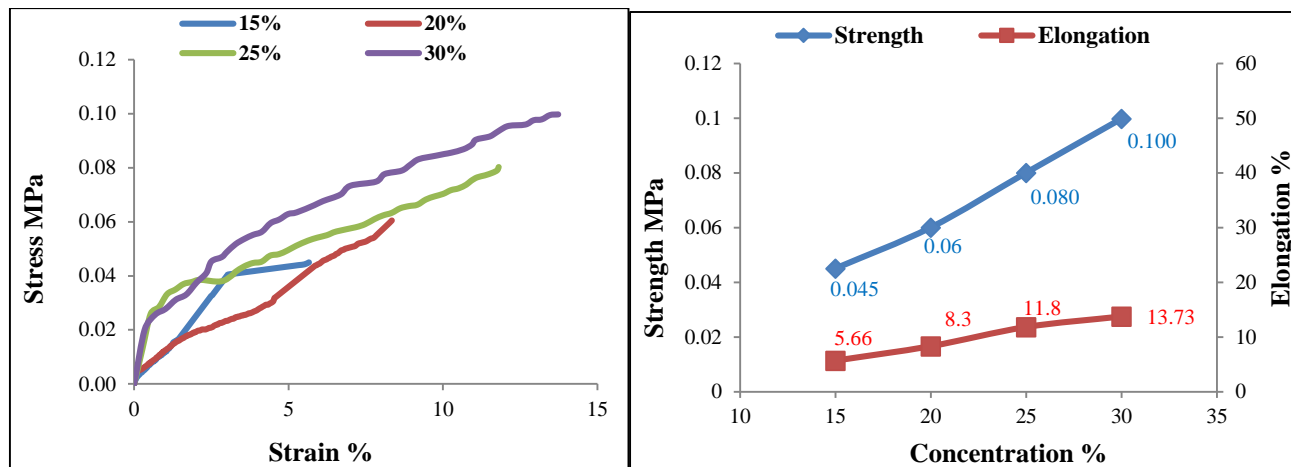


Figure 5: Contact angle for (15, 20, 25 and 30%) polystyrene concentration membranes, respectively.

### 3.5. Tensile Test

Fig. (6) illustrates how mechanical behaviour changes as polystyrene concentrations rise. It was found that the amount of polystyrene increased with the tensile strength of electrospinning membranes; therefore, polystyrene at 15% weight has a lower strength (0.1 MPa), while polystyrene at 30% weight has a higher one (0.1 MPa), and this is related to the higher amount of polymeric material that is present in forming fibers [23,24].



**Figure 6:** (A) Stress-Strain curves for polystyrene electrospinning membranes, (B) Effect of polystyrene concentration on tensile strength and elongation.

### 4. Conclusion

The effect of polymeric concentration on the morphologies of electrospinning membranes is successfully examined in this study. From thinner fibers with bead structures in low polystyrene solution concentrations (PS15) to thick fibers with smooth and continuous structures in the highest concentrations (PS30), different polystyrene solutions produce different fiber morphologies. Polystyrene solution concentrations significantly increased fiber strength and elongation, according to tensile tests. The measurements of wettability showed a decline in hydrophobicity.

### Acknowledgement

The authors thank laboratory staff in department of material engineering in university of technology for helping and supporting in making the manuscript.

### Conflict of Interest

The authors declare that they have no conflict of interest.

### References

- [1] S. Maghsoodlou, B. Noroozi, and A. K. Haghi. "A simple model for solvent evaporation in electrospinning process." *Nano*, vol. 12, no. 03, p.1750028, 2017.
- [2] R. Mohamat, et al. "Methylene blue rejection and antifouling properties of different carbonaceous additives-based polyvinylidene fluoride membrane." *Materials Today Communications*, vol.35, p.105862, 2023.
- [3] N. Sabetzadeh, and A. A. Gharehaghaji. "How porous nanofibers have enhanced the engineering of advanced materials: a review." *Journal of Textiles and Polymers*, vol. 5, no. 2, p. 3-21, 2017.
- [4] X. X. He, et al. "Near-field electrospinning: progress and applications." *The Journal of Physical Chemistry C*, vol. 121, no. 16, p. 8663-8678, 2017.
- [5] N. Meng, Y. Zheng, and B. Xin. "Fabrication and characterization of graphene enriched polysulfon amide nanocomposites by electrospinning system." *Fibers and Polymers*, vol. 19, p. 357-363, 2018.
- [6] M. S. Al-Eissa, R. S. Almkhtar, and B. Y. Sherhan. "Experimental Study of Thermal and Catalytic Decomposition of PVC Wastes." *Journal of Applied Sciences and Nanotechnology*, vol.2, no. 4, p. 56-69, 2022.

- [7] N. Bhardwaj, and S. C. Kundu. "Electrospinning: A fascinating fiber fabrication technique." *Biotechnology advances*, vol. 28, no.3, p. 325-347, 2010.
- [8] K. K. Hameed, I. H. Gatea, and A. A. Hussein. "Biosynthesis and Optimization of PolyHydroxyalkanoate (PHA) Production by *Acinetobacter lwoffii* Isolate." *Journal of Applied Sciences and Nanotechnology*, vol.3, no.1, 2023.
- [9] M. T. Hameed, B. M. Al-Dabbagh, and R. K. Jasim. "Effect of Natural Fibers on Some Thermal and Physical Properties of Denture Base Materials." *Journal of Applied Sciences and Nanotechnology*, vol. 2, no.3, p. 64-77, 2022.
- [10] W. Liu, C. Huang, and X. Jin. "Tailoring the grooved texture of electrospun polystyrene nanofibers by controlling the solvent system and relative humidity." *Nanoscale research letters*. Vol. 9, p. 1-10, 2014.
- [11] P. Lu, and Y. Xia. "Maneuvering the internal porosity and surface morphology of electrospun polystyrene yarns by controlling the solvent and relative humidity." *Langmuir*, vol. 29, no.23, p. 7070-7078, 2013.
- [12] F. A. Asim, E. H. A. Al-Mosawe, and W. A. Hussain. "The Medical Study of Denture Base Resin Poly (Methyl Methacrylate) Reinforced by ZnO and TCP Nanoparticles." *Journal of Applied Sciences and Nanotechnology*, vol.2, no.4, 2022.
- [13] C. Huang, and N. L. Thomas. "Fabricating porous poly (lactic acid) fibres via electrospinning." *European Polymer Journal*, vol. 99, p. 464-476, 2018.
- [14] M. Eleuteri, L. Pastorino, and O. Monticelli. "On the degradation properties of electrospun fibers based on PLLA: the effect of a drug model modification." *Polymer Degradation and Stability*, vol. 153 p. 109-117, 2018.
- [15] T. Nitanan, et al. "Effects of solution parameters on morphology and diameter of electrospun polystyrene nanofibers." *Advanced Materials Research. Trans Tech Publications Ltd.*, vol. 194, p. 629–632, 2011.
- [16] A. R. Jabur, L. K. Abbas, and S. A. Moosa. "Fabrication and Characterization Membrane Prepared from Oxidized Multiwall Carbon Nanotube/Nylon 6 Composite." *journal of Karbala University*, vol. 11, no.3, p. 200-212, 2015.
- [17] A. H. Oleiwi, A. R. Jabur, and Q. F. Alsally. "Morphology of polystyrene nano-fiber membranes reinforced with copper oxide and zirconium oxide nanoparticles as a sulfur absorbent materials." *AIP Conference Proceedings. AIP Publishing*. vol. 2769, no. 1, 2023.
- [18] R. M. Nezarati, M. B. Eifert, and E. Cosgriff-Hernandez. "Effects of humidity and solution viscosity on electrospun fiber morphology." *Tissue Engineering Part C: Methods*, vol. 19, no. 10 p. 810-819, 2013.
- [19] A. H. Oleiwi, A. R. Jabur, and Q. F. Alsally. "Polystyrene/Mwcnts nanofiber membranes characterizations in reducing sulfur content in crude oil." *Key engineering materials*, vol. 886, p. 86-96, 2022.
- [20] N. M. Jalal, et al. "Preparation, Microstructure and Morphology of Electrospun Sulfonated Polystyrene Films for Proton Exchange Membrane Hydrogen Fuel Cell." *Energy Procedia*, vol. 157, p. 1494-1505, 2019.
- [21] A. H. Oleiwi, A. R. Jabur, and Q. F. Alsally. "Preparation of Polystyrene/Polyacrylonitrile Blends by Electrospinning Technique." *Journal of Physics: Conference Series. IOP Publishing*, vol. 1879, no. 2, 2021.
- [22] A. B. D. Cassie, and S. Baxter. "Wettability of porous surfaces." *Transactions of the Faraday society*, vol. 40, p. 546-551, 1944.
- [23] A. H. Oleiwi, "Study some properties for UPS composites reinforced by sunflower husk ash." *AIP Conference Proceedings. AIP Publishing*, vol. 2437, no. 1, 2022.
- [24] G. Y. Dakhil, R. M. Salih, and A. M. Hameed. "Influence of Infill Pattern, Infill Ratio on Compressive Strength and Hardness of 3D Printed Polylactic Acid (PLA) Based Polymer." *Journal of Applied Sciences and Nanotechnology*, vol. 3, no.1, 2023.