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Morphology Characterization of Electrospun Polystyrene Membranes

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A B S T R A C T

The concentration of the polymer solution is one of the most important factors in the formation of fibers in electrospinning technology. Different polymer concentrations of thermoplastic polystyrene are used in this study. At the same time, the other operating parameters of the electrospinning process (such as flow rate, voltage, distance between capillary and collector), the solution parameters (such as conductivity and molecular weight of the solution) and the environmental conditions (such as temperature and humidity) were kept constant. Field emission scanning electron microscopy was used to investigate the morphological changes on the surface of the fibers and to determine the typical fiber diameter. It was found that as the polystyrene concentration was increased from 15% to 30%, the average pore size increased from (0.5µm and 0.44µm) to (2.7µm and 2.6µm). The FT-IR showed the main chemical bonds in the polystyrene membranes and the change in peak intensity by increasing the polymer concentration. The contact angle measurements, which are used to investigate the change in hydrophobic properties, show that the hydrophobicity of the membranes decreases as the water contact angle decreases from 135 to 116 when the polymer concentration is increased from 15% to 30%.

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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 application 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 deference 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 Part

2.1 Materials

Polystyrene with molecular weight of (Mw = 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].

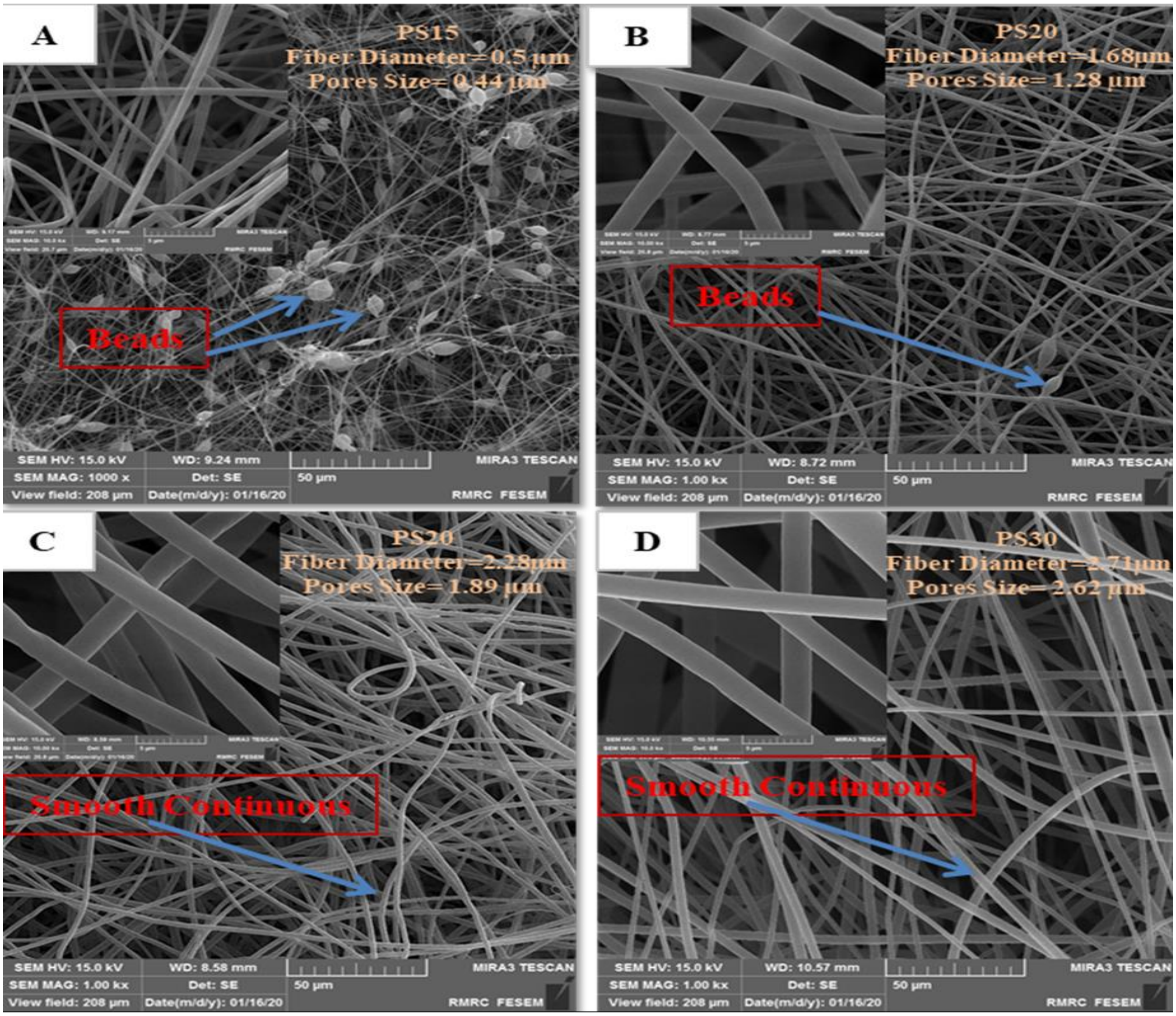

Figure 1: FE-SEM images for (15, 20, 25 and 30%) polystyrene concentrations membranes, respectively.

The average fiber diameters of the membranes produced in PS15 and PS30 range from (0.5 - 2.71 µm), 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].

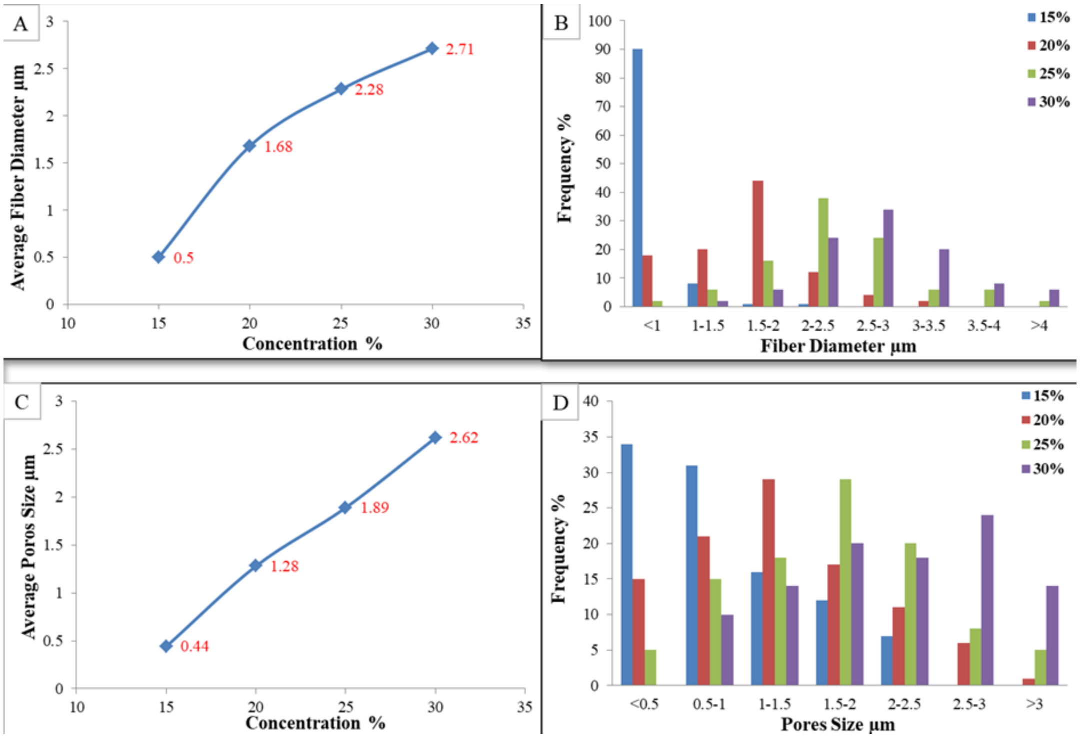

Figure 2: The fibers diameter, pores size and their distributions for different PS concentrations.

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].

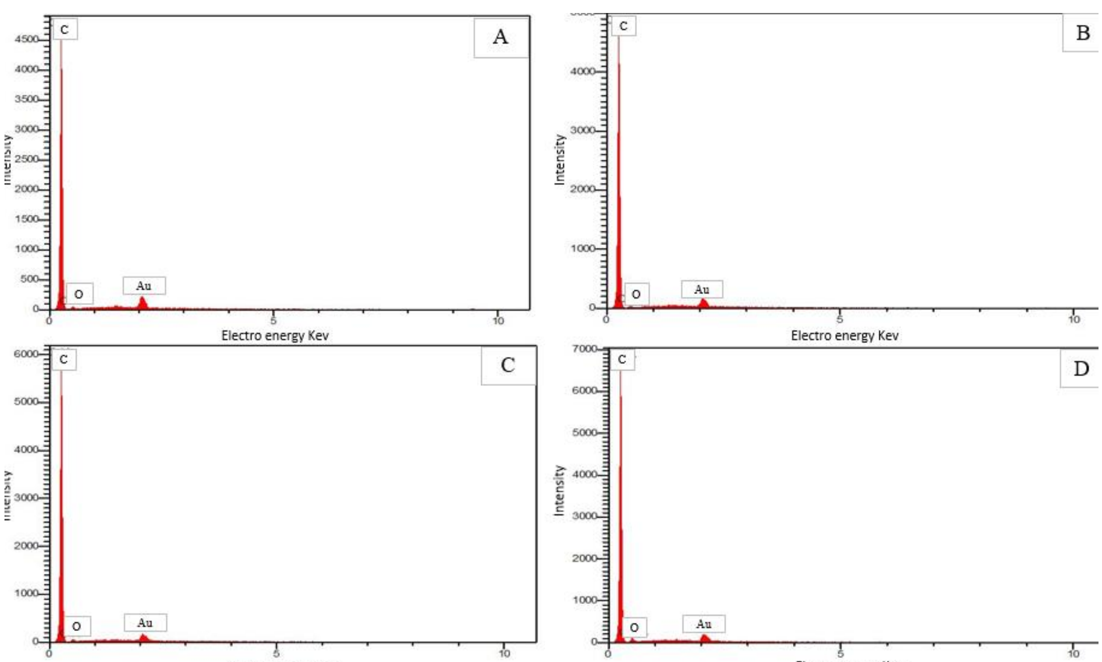

Figure 3: EDX for (15, 20, 25 and 30%) polystyrene concentrations membranes, respectively.

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 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 cm^{-1} that are linked to the C=C bond in an aromatic ring (stretching vibration). Finally, at 753.69 and 696.43 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 cm^{-1} wave transmission peaks indicate the presence of the methylene group. All of the current peaks are agreed with Noor M. Jalal [20].

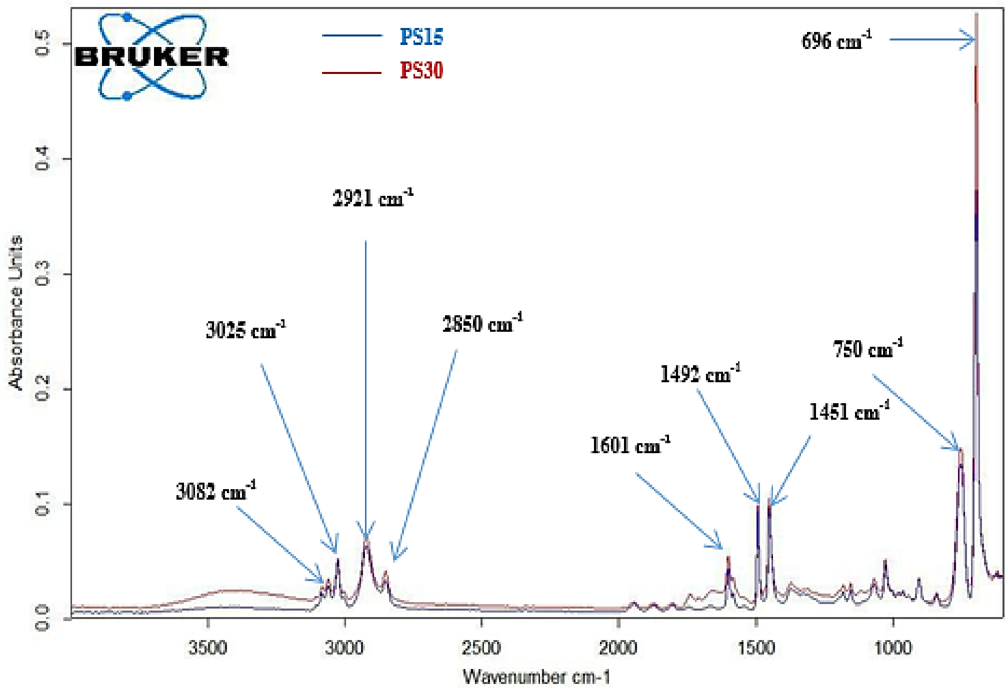

Figure 4: FTIR for (15 and 30%) polystyrene membranes.

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° to 116° 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°. These structures provide a benefit for improving membrane hydrophobicity [22].

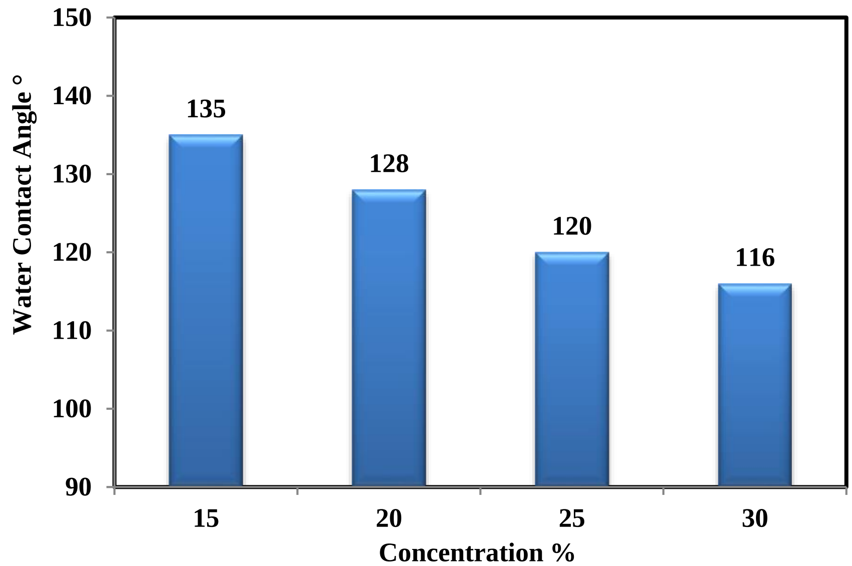

Figure 5: Contact angle for (15, 20, 25 and 30%) polystyrene concentration membranes, respectively.

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].

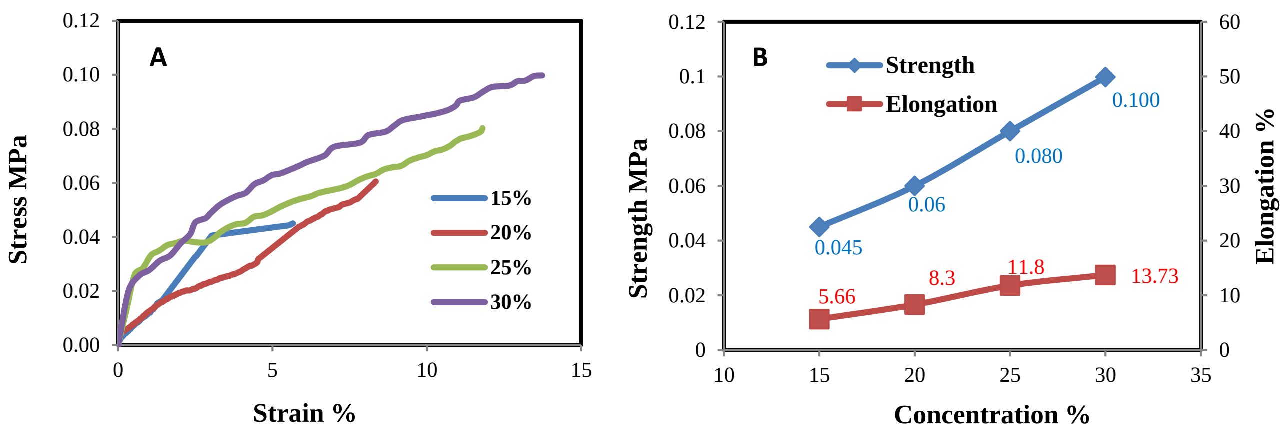

Figure 6: (A) Stress-Strain curves for polystyrene electrospinning membranes, (B) Effect of polystyrene concentration on tensile strength and elongation.

4. Conclusions

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.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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