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Effects of Electrospinning Parameters on the Morphology of Electrospun Fibers

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Abstract

Hydrophobic electrospun membranes have a lot of applications in different fields. It is very difficult to increase the hydrophobicity of membranes for a specific application. This study investigates the effects of various electrospinning parameters on the morphology and hydrophobicity of polystyrene (PS) electrospun membranes. Polystyrene fibers were used as a reference for the study. Different parameters such as polymer concentrations, diameter of needles, and applied voltage were tested to study the influence on the hydrophobicity of electrospun fibers. Polystyrene fibers were electrospun at different concentrations from 5 to 20 wt.%, needles with a diameter from 0.5 to 3 mm were used, and voltage was applied between 8.06–16.05 kV. The surface morphology of polystyrene fibers and hydrophobicity were studied with a scanning electronic microscope and contact angle measurements. Based on the results of the study, higher polymer concentrations and voltages produce thinner fibers and more hydrophobic membranes. The results of this paper can be applied to the fabrication of different characteristic membranes for specific applications like water conservation, purification, and other fields.

1. Introduction

Oil pollution of water is one of the biggest problems nowadays. Oil-water separation is considered an important part of the water purification industry. There are many methods for oil-water separation, and one effective approach is the use of hydrophobic electrospun membranes. These membranes are particularly popular due to their unique properties, such as high surface area, tunable porosity, and inherent hydrophobicity. These characteristics make hydrophobic electrospun membranes highly suitable for a wide range of applications, including efficient oil-water separation. However different parameters are the main guarantee for the use of membrane fibers in different fields. Because one of the main surface properties of electrospun membrane fibers is their morphology, various parameters strongly affect the morphology of electrospun membrane fibers. Different polymeric materials with controlled hydrophobicity, achieved by adjusting their parameters, can be utilized in various fields. These applications range from construction materials and water conservation tanks to the purification of water from organic pollutants. To effectively use these materials across different applications, their specific surface characteristics must align with the requirements of each particular field [1–4]. Based on the research of some scientists, the humidity of the environment is an important factor affecting the morphology of electrospun polymer fibers [5]. The results of this study highlight the importance of adjusting the environmental conditions during the electrospinning process in creating the final fiber structure. In addition, in addition to humidity and environmental conditions, the molecular weight of the polymer is also the main factor that strongly affects the morphology and diameter of fibers [6]. Thus, it has been shown that the molecular weight of the polymers is very important to control the diameter and morphology of the fiber in the electrospinning process.

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Moreover, various solvent systems used to dissolve polymers during the electrospinning of polymer solutions are also important in forming the surface morphology of the prepared membrane fibers. Based on the research it was found that a good solvent system and special properties of solvents have different effects on the surface morphology of the produced membrane fibers [7, 8]. The results of this study show how important it is to choose a suitable solvent system to achieve the target fibers with the best hydrophobicity and characteristics of the membranes.

In addition to environmental conditions and solvent properties, other parameters during the electrospinning process may play an important role in shaping fiber morphology. For example, many studies report the importance of adjusting parameters such as applied voltages and electric field distribution to control the sizes and morphologies of electrospun fibers [9–11]. Based on these studies, it can be concluded that by adjusting various electrospinning parameters, researchers can modify the morphology of membrane fibers for a specific application.

Furthermore, the morphology of the fibers can be influenced by selecting the appropriate polymers and additives used in the electrospinning process. The reason for this is that many studies have shown different morphologies of electrospun fibers were created by adding nanofillers or using other special polymer mixtures [12–15]. As mentioned above, we found that understanding how different polymers, additives, and other environmental conditions interact during electrospinning is critical for designing fibers with the desired morphology. In general, the formation of the surface morphology of electrospun fibers is a complex process based on various parameters, and it was mentioned above that membranes with different surface properties can be obtained by changing only one parameter.

Apart from the above-mentioned conditions that influence hydrophobicity, many other factors contribute to this morphological formation. Nevertheless, very few studies have been dedicated to this issue. Therefore, the present paper explores how various parameters such as polymer concentrations, needle diameters, and applied voltage affect fiber morphology leading to its varying hydrophobicity. With precise control of these parameters, electrospun fibers can be tuned morphologically for different applied fields such as tissue engineering, sensor technologies, and oil-water separation.

2. Experimental details

2.1 Materials

Polystyrene (PS, average M_W = 192.000) and N,N-Dimethylformamide (DMF, 99.7%) were purchased from Sigma-Aldrich and a Russian manufacturing company "Chemservice" respectively.

2.2 Electrospinning process

To fabricate the polystyrene electrospun fibers, depending on the target polymer concentration a particular amount of PS spheres were completely dissolved in 20 mL of DMF resulting in 5 wt.%, 10 wt.%, 15 wt.%, and 20 wt.% PS in DMF solutions. After, those solutions were poured separately into a 5 ml syringe with a needle diameter of 1 mm, which was placed in the electrospinning machine. The following electrospinning parameters were used: constant pumping rate of 1.0 mL/h, and the needle tip-collector distance of 17 cm. Three spinning voltages were applied such as +8.06 kV, +12.05 kV, and +16.05 kV, while the collector applied voltage was kept constant (-5 kV). Moreover, needles with different diameter sizes were used during those electrospinning experiments. The schematic view of the electrospinning process is illustrated in Fig. 1.

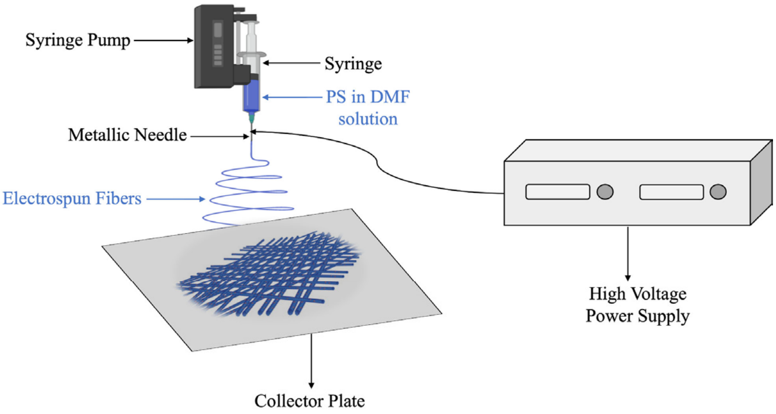

Fig. 1. Schematic diagram of the electrospinning process.

2.3 Characterizations

Scanning electron microscopy (SEM, ZEISS Crossbeam 540) technique was used to retrieve the morphology information about the electrospun fibers.

The water contact angle (WCA) values of the fibers were determined by a goniometer (Dataphysics OCA 15 Pro) using approximately 5 µL of the deionized water with a dosing rate of 1 µL/s (medium level) for all measurements.

3. Results and discussion

3.1 Effects of applied voltage

The electrospinning process is driven by the applied voltage. The thin fibers are mostly generated by the stretching and accelerating of the jets caused by the strong electric field [16]. The changes in the applied voltage during the electrospinning process can affect the electrospun fibers' morphology and their surface wetting behavior. In several studies, it is commonly claimed that as the applied voltage increases, the fibers' diameter steadily decreases [17–20]. On the contrary, in other cases opposite results were reported, particularly it was stated that the applied voltage can affect other process parameters including the distance between the needle and collector, which leads to an increase in the fiber diameter [21].

The morphology of the obtained PS electrospun fibers at three different voltage values are shown in Figs. 2 (A1-C2) below. As can be seen from Fig. 2 (A1), (B1), and (C1), at all three voltage values the fiber mats were generated. Moreover, the changes in the applied voltage did not affect significantly the bead formation, and the beads of different sizes were obtained at all three voltage values (see Fig. 2 (A2), (B2), and (C2)). Although no significant differences in the morphology of PS electrospun fibers at three different voltage values can be observed, there are some differences in the wetting surface behavior of the electrospun fibers. As the applied voltage was increased from 8.06 kV to 16.05 kV, the water contact angles of PS fibers increased slightly from 128° to 129°. This might indicate that the fiber diameter decreased with the increase in the voltage, which further led to the increased water contact angles as it was reported by Yohe et al. [22].

3.2 Effects of needle diameter on fiber morphology and its surface wetting behavior

SEM images of PS nanofibers obtained using different needle diameters at a constant pumping rate and the same applied voltage are depicted in Figs. 3 (A1-C2). Figures 3 (A1), (B1), and (C1) show that the diameter of fibers mostly varied from 311.7 nm to 563.1 nm for all three needles with diameter sizes of 0.5 mm, 1 mm and 3 mm, indicating that the needle diameter did not affect significantly the fiber diameter. There are also other studies stating that the needle diameter does not have a great effect on the diameter of fibers. Sencades et al. found out that for the poly(vinylidenefluoride–trifluoroethylene) nanofibers the decrease in the needle diameter did not significantly change the fiber mean diameter, but led to a broader fiber diameter distribution [23]. Macossay et al. evaluated the relationship between three needle sizes and the average nanofiber diameter of poly(methyl methacrylate) electrospun and stated that the needle diameter does not significantly affect the fiber diameter [24]. Similarly, a lack of correlation between the needle size and the fiber diameter was determined by Albertan et al. by using two different needles [25].

As shown in Figs. 3 (A2) and (B2), a smaller needle diameter can lead to the formation of larger numbers of beads but smaller in size. The reason for this is that the finer needle raises the surface tension of the polymer solution at the needle tip, and it becomes harder to form a stable jet causing the generation of the micro-beads. Conversely, larger beads in less amount were formed when the needle with a larger needle diameter was used (see Fig. 3 (C2)). Moreover, the agglomeration of coarse beads contributed to the lower porosity of PS electrospun fibers, which resulted in a lower water contact angle of 125°. It can be observed from Figs. 3 (A2), (B2), and (C2) that with the decrease in the needle diameter, the water contact angle values increased to 134° for PS fibers. As the needle diameter decreased from 3 mm to 0.5 mm, the smaller beads were formed, which led to the higher porosity of the electrospun fibers. Higher porosity commonly contributes to the increased surface roughness, which can further improve the hydrophobicity of the fibers.

3.3 Effects of polymer concentration

The polymer concentration can be one of the important factors determining the spinnability of a solution, which indicates whether or not a fiber forms. The concentration of the polymer affects the solution's surface tension and viscosity, two crucial aspects of the electrospinning process. If the polymer solution is too diluted, the polymer filaments

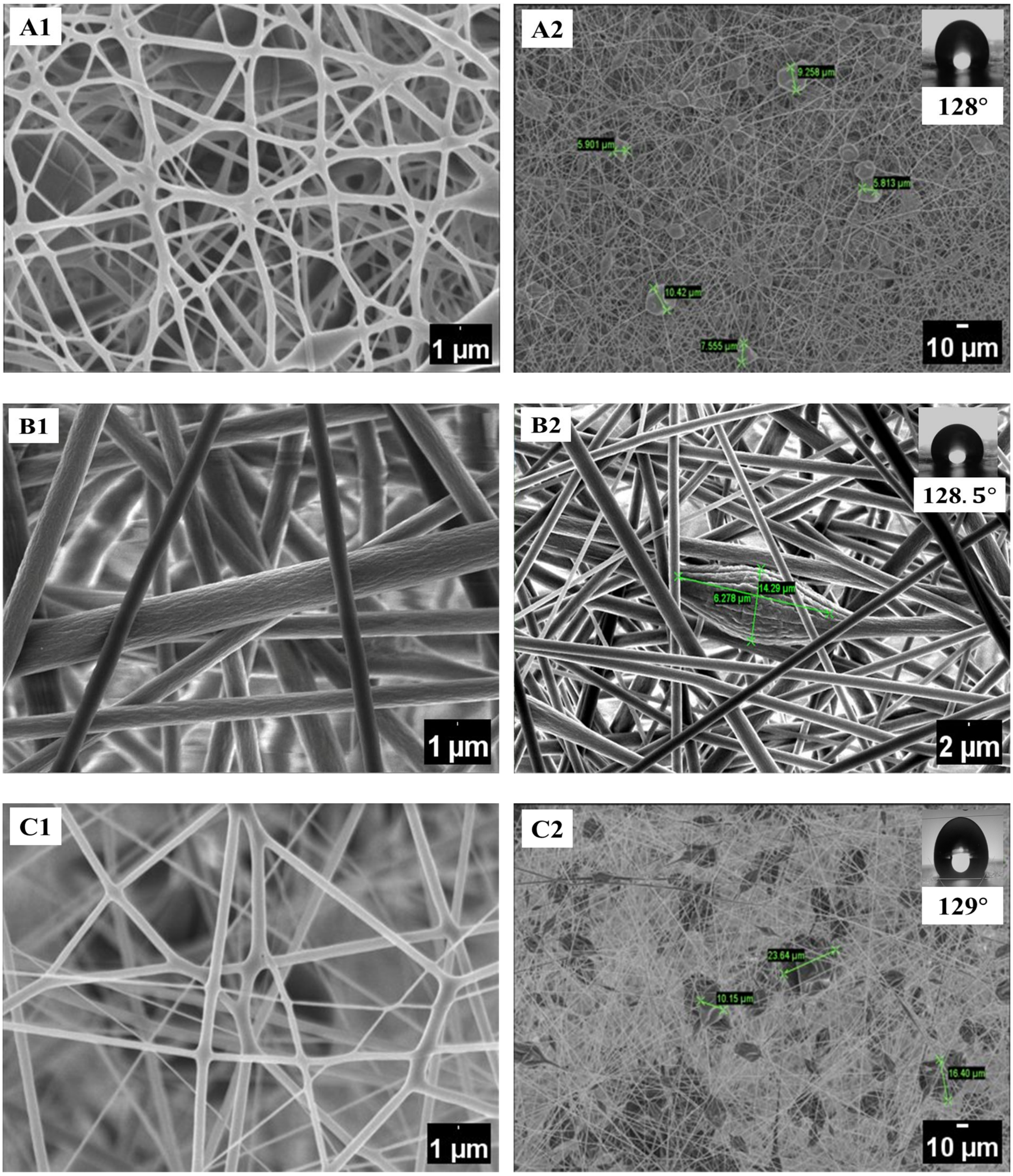

Fig. 2. SEM images of PS fibers obtained at different voltage values: (A1-A2) 8.06 kV, (B1-B2) 12.05 kV, and (C1-C2) 16.05 kV.

will break into droplets before it will reach the collector, and no fibers will be formed. Whereas, the excessive concentration of the solution prevents fiber formation because of its high viscosity, making it challenging to regulate the solution's flow rate through the tubes and the needle. The appropriate polymer concentration for the spinnability of a solution depends on the polymer molecular weight and the type of solvent used [26].

It can be seen from Fig. 4 (A) that at a lower polymer concentration of 5 wt.% PS in DMF the fiber structure was not obtained, and the polymer droplets of different sizes were generated. As the concentration increased to 10 wt.%, the electrospun fibers were generated (Fig. 4 (B)). When polymeric solutions with higher concentrations such as 15 wt.% and 20 wt.% PS in DMF were put into the electrospinning machine, thicker electrospun fibers were produced (Figs. 4 (B)

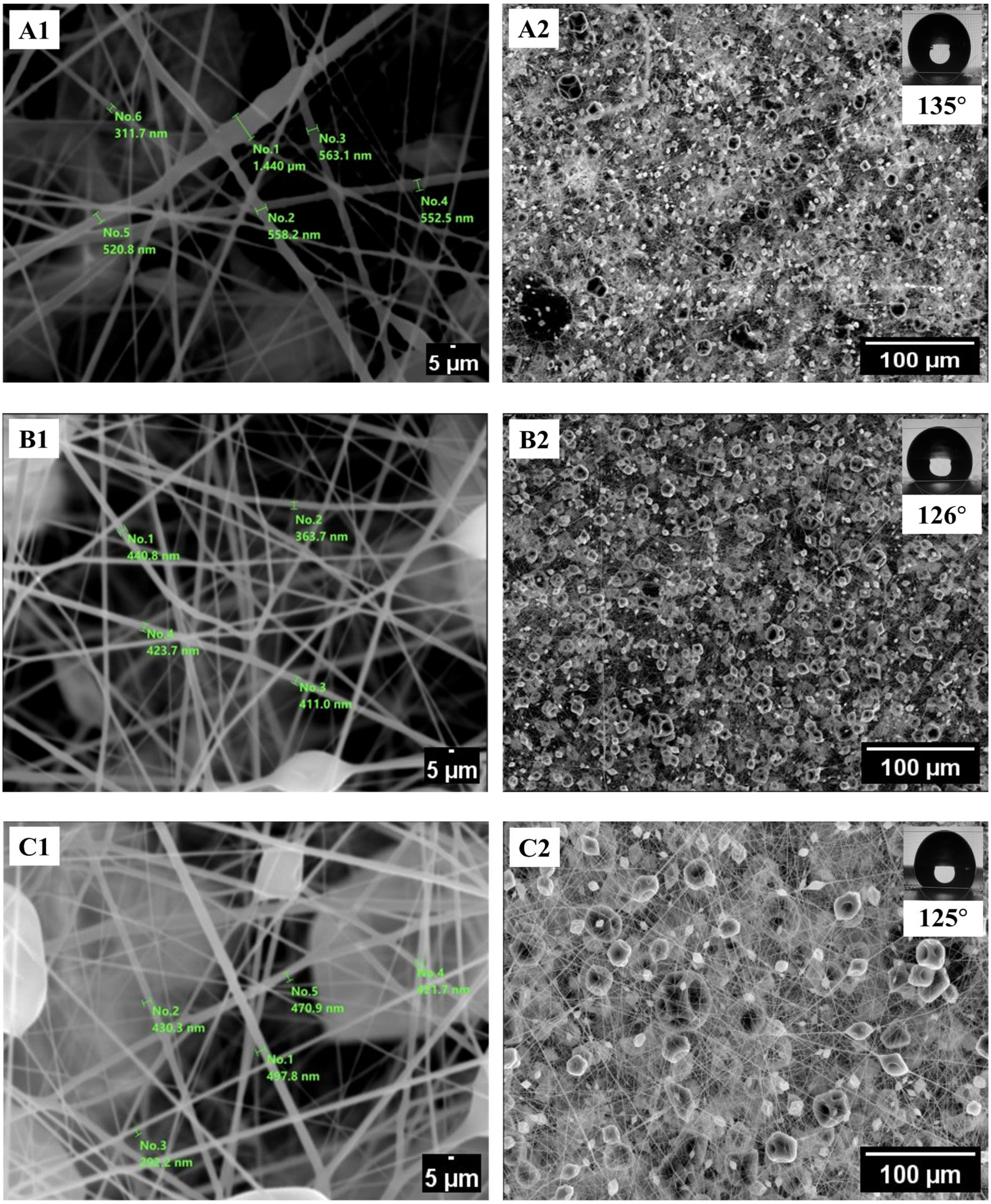

Fig. 3. SEM images showing the morphology of PS electrospun fibers obtained using needles with three different diameter sizes: (A1-A2) 0.5 mm, (B1-B2) 1 mm, and (C1-C2) 3 mm.

and (C)). The higher weight percentages of PS in DMF solutions (larger than 20%) were not studied because dense solutions could not pass through the needle. Therefore, the polymer concentration of 20 wt.% can be considered an optimal concentration among all PS in DMF solutions for obtaining a good fiber structure.

In other studies, particularly in the study developed by Hekmati et al., the electrospun fibers from the polyamide 6 in formic acid solutions with different concentrations such as 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.%, and 25 wt.% were investigated [27]. They determined that it was not possible to spin a low

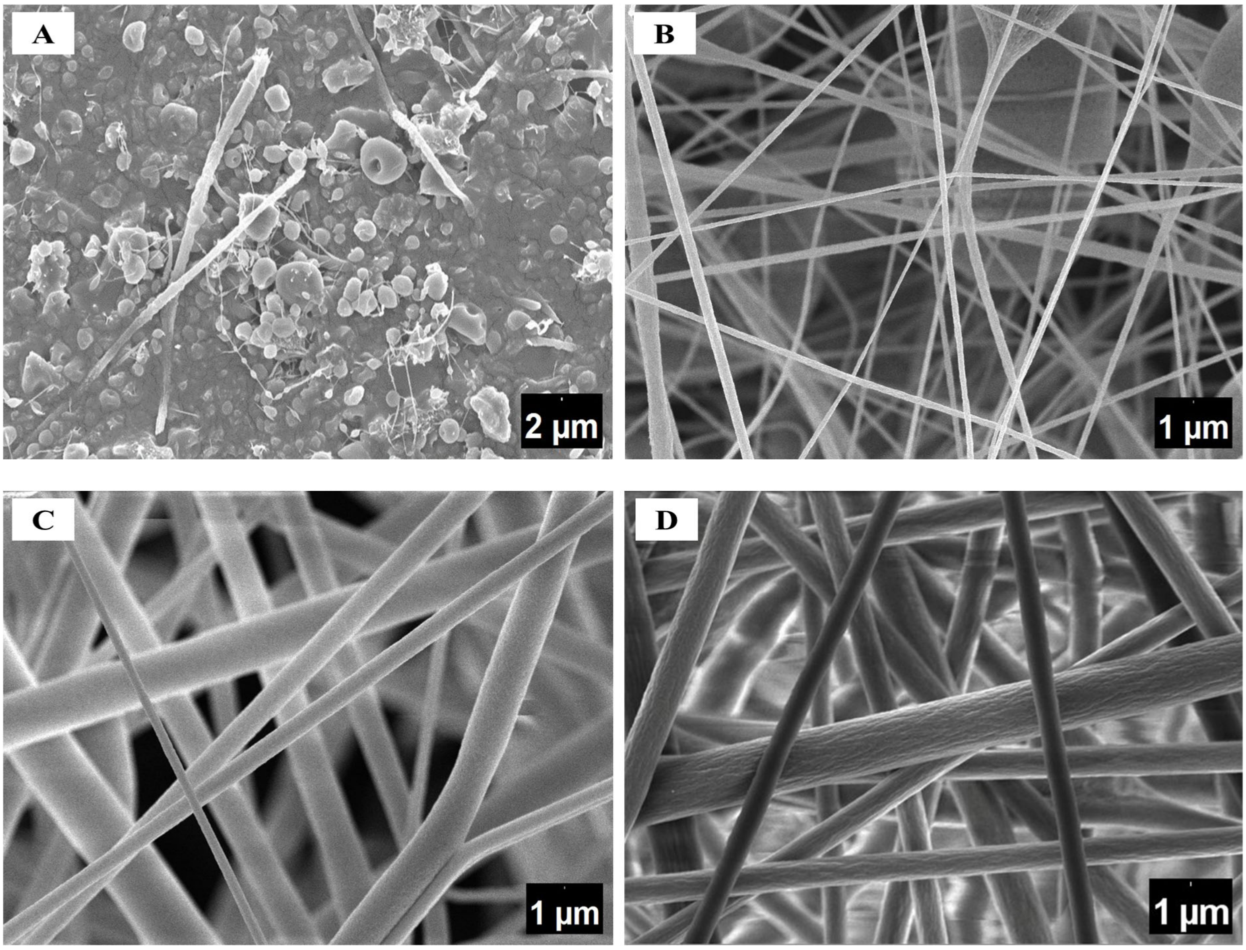

Fig. 4. SEM images of PS electrospun fibers obtained from different solution concentrations: (A) 5 wt.%, (B) 10 wt.%, (C) 15 wt.%, and (D) 20 wt.%.

concentrated polymer solution (5 wt.%), and only small polymeric droplets were produced. At higher concentrations (10 wt%, 15 wt.%, 20 wt.%, and 25 wt.%) of polyamide 6 in formic acid solutions, the electrospun nanofibers were obtained. The polymer concentration led to homogeneous, bead-free and non-branched nanofibers generation [20]. Similarly, Tarus et al. determined that under a certain concentration polymeric drops will be generated instead of fibers [28]. Thus, the effectiveness of electrospun fibers is influenced by the electrospinning parameters, which determine the fibers' morphology and surface properties. Ideally designed materials can be useful in water purification in local areas, in the development of water-repellent concrete composites for water storage, and in understanding the wetting behavior in various applications [29–33].

4. Conclusion

In conclusion, this study successfully investigated the key parameters influencing the morphology and hydrophobicity of electrospun polystyrene (PS) fibers. The results revealed that higher polymer concentrations and increased applied voltages tend to produce thinner fibers with enhanced hydrophobicity. Specifically, polymer concentrations of 5 wt.% or higher were found to consistently generate fibers. As the applied voltage was raised from 8.06 to 16.05 kV, the water contact angles of PS fibers showed a slight increase from 128° to 129°. Fiber diameters generally ranged from 311.7 to 563.1 nm across needle diameters of 0.5 mm, 1 mm, and 3 mm, suggesting that needle diameter had minimal impact on fiber thickness. However, a decrease in needle diameter increased water contact angle values, reaching 134° for PS fibers. These findings underscore the importance of precise control over polymer concentration, needle diameter, and applied voltage in the fabrication of electrospun fibers with tailored properties. The insights gained from this study can guide the optimization of electrospun fiber production, facilitating their effective application in areas such as water conservation, purification, and various other fields.

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