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SYNTHESIS OF POLYSTYRENE FIBER MEMBRANES PREPARED BY ELECTROSPINNING: EFFECT OF $AgNO_3$ ON THE MICROSTRUCTURE

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ABSTRACT

Polystyrene (PS) is commonly employed in insulation, packaging, filters, and medical equipment, with recent studies exploring its potential in fiber membrane production. The electrospinning technique is discussed to synthesize PS fiber membranes with high porosity and controllable diameter. Additionally, incorporating silver nitrate into PS composite fibers is explored for enhanced functionalities such as catalytic activity, high electrical conductivity, and antibacterial activity. However, PS composite fiber membranes with silver nitrate ($AgNO_3$) metal variations are rarely observed. This research aims to modify the microstructure of PS fiber membranes produced using electrospinning by adding silver nitrate ($AgNO_3$) with varying concentrations. PS-Ag fiber membranes are produced using N,N-dimethylformamide (DMF) solvent, which serves as a solvent and a reducing agent for Ag. The results show that the effect of Ag affected the diameter of the PS-Ag fiber membrane, with an average diameter of around 3.67 - 6.93 micrometers. Degradation occurred in these samples at a strong broadening peak near ~1300 cm^{-1} until ~1600 cm^{-1} from the Raman results. The FTIR results show that the wavelength of ~3500 cm^{-1} indicated the presence of OH. The presence of OH indicates that the PS-Ag fiber membrane has the potential for water filtration application.

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INTRODUCTION

Polystyrene (PS) is a highly useful thermoplastic noted for its transparency, colorlessness, high electrical resistance, and low dielectric loss [1][2]. Common applications of PS materials include insulator materials [2], transparent packaging [3], filters [4], lampshades [5], and medical equipment [6]. Numerous studies have demonstrated the feasibility of manufacturing PS into fibers, significantly expanding its application across various fields [1]–[12]. Enhancing PS fiber membranes' functionality involves combining them with other polymers

or composite materials, which introduce many novel characteristics superior to single polymers [3], [11]. PS is commonly used as a constituent material in nanofiber composite structures produced via electrospinning, a method well-regarded for synthesizing fiber membranes [13]. Electrospinning offers the capability to create fiber membranes with high porosity (≥90%) and homogeneity [14]. This technique employs high voltage to induce a charge in the polymer solution. The needle and syringe pump serve as reservoirs and regulators for the solution, while the collector serves as the deposition site for the fibers. The fiber diameter can be controlled by adjusting electrospinning parameters, including solution, process, and environmental parameters. This enables the production of smooth and sustainable fiber membranes for various potential applications, such as composite materials [15].

PS materials, renowned for their versatility, have been the subject of extensive research, particularly when enhanced with additives to create composite fiber membranes. Such additives include ZnO [16], activated carbon (AC) [17], TiO_2 [18], and silver nitrate ($AgNO_3$) [5], [19]. Studies have demonstrated the incorporation of ZnO nanoparticles into PS fibers, producing superhydrophobic and anticorrosive surfaces on aluminum alloy substrates [16]. Additionally, PS fibers decorated with activated carbon have shown promising results as adsorbents for removing cationic dyes from polluted water [17]. At the same time, surface-tailored PS/TiO_2 composite nanofiber membranes have been effective for copper removal [18]. Silver nitrate, noted for its catalytic activity, high electrical conductivity, and antibacterial properties, has been incorporated into PS fibers, significantly enhancing their antibacterial activity, a desirable trait for applications such as air filtration masks [5]. Despite its advantages, the incorporation of silver nitrate at concentrations such as 1% has been found to reduce the thermal stability of PS-Ag composites compared to pure PS fibers [5]. Moreover, studies have shown that PS-Ag nanofibers possess antimicrobial properties against Gram-positive bacteria like *Staphylococcus xylosus* [19]. However, variations in the concentration of silver nitrate within PS composite fiber membranes produced via electrospinning have been scant. Therefore, this study explores different concentrations of Ag within PS composite fibers to better understand their impact on properties and potential applications.

By incorporating Ag at concentrations of 0.5, 1, and 2 wt.%, this study aims to investigate the microstructural changes in PS membranes. Previous research by Song et al. [19] demonstrated that adding Ag reduces the fiber diameter of PS membranes and sustains their antibacterial activity, suggesting a potential for safe and recyclable applications. Contrarily, the study by Mostafa et al. [5] did not address the structural and fiber diameter alterations caused by adding Ag nanoparticles. In this research, N, N-Dimethylformamide (DMF) was employed as the solvent, renowned for its efficacy in producing uniform PS fiber membranes [20], [21]. Utilizing DMF offers a safer and non-

toxic method for developing PS-Ag composite fibers [5]. The variation in Ag concentration is a critical parameter in the electrospinning process, significantly influencing the physicochemical properties of the resultant fiber membranes. The integration of Ag altered the microstructure of the PS fiber membranes. To analyze these modifications, fiber membranes with different Ag concentrations were examined using scanning electron microscopy (SEM) equipped with energy dispersive X-ray spectroscopy (EDS) mapping, providing insights into fiber diameter distribution. Raman and Fourier transform infrared (FTIR) spectroscopy were employed for molecular interaction characterization. The PS-Ag fiber membranes produced in this study hold promising potential for filtration applications.

METHODS

1. Materials

Polystyrene (PS) with an average molecular weight of 192,000 g/mol, silver nitrate ($AgNO_3$), and N,N-dimethylformamide (DMF) were procured from Sigma-Aldrich. DMF is a suitable solvent for reducing Ag and is non-toxic to humans.

2. Preparation of PS-Ag

The polymer solution was prepared by dissolving PS powder in DMF at a concentration of 25 wt.%. The precursor solution is ideal for electrospinning at this concentration, producing continuous fibers without beads. The solution was stirred on a magnetic stirrer with a hot plate set to 60 °C until it reached a homogeneous state. The variations in $AgNO_3$ concentration in the PS solution are detailed in Table 1.

Table 1. The ratio of silver nitrate ($AgNO_3$) in the PS solution

Mixing ratio (g) PS	$AgNO_3$	Ag in PS solution (wt.%)	Sample name
5	0	0	PS
5	0.025	0.5	PA1
5	0.05	1	PA2
5	0.1	2	PA3

The concentration selection was influenced by research that utilized only a single variation of Ag concentration at 1 wt.% [5]. Additional concentrations of 0.5, 1, and 2 wt.% were selected to explore a broader range of Ag concentrations and their effects. These concentrations were prepared into precursor solutions as outlined in Table 1 and stirred using a magnetic stirrer on a hot plate set to 60 °C until homogeneity was achieved. The inclusion of different $AgNO_3$ concentrations impacts the viscosity of the solution, with viscosity increasing as $AgNO_3$ concentration rises. This variation in viscosity notably affects the electrospinnability of the solution, where higher concentrations of $AgNO_3$ hamper the ability to form fine fibers. Moreover, the precursor solution with the highest $AgNO_3$ concentration exhibited a more intense brown color, indicating stronger silver interaction. Subsequently, these precursor solutions were subjected to electrospinning to fabricate PS-Ag fiber membranes. The process parameters were carefully adjusted to accommodate the varied viscosities and ensure the production of uniform fiber membranes.

3. Production of PS-Ag Composite Fiber Membranes

The precursor solution was loaded into a syringe equipped with a 21-gauge

needle as the initial step. The syringe was mounted on a syringe pump, initiating the electrospinning process. Electrospinning parameters were meticulously set, with the applied potential voltage at 12 kV, the flow rate at 0.5 mL/h, and the distance between the needle tip and the collector fixed at 20 cm. These parameter settings, illustrated in Figure 1, are derived from optimization experiments to achieve continuous fibers without bead formations, which is critical for successful fiber membrane production [22]. The relative humidity during the process was maintained at 60% to facilitate complete solvent evaporation. The resulting PS-Ag fiber membranes were collected on a collector lined with aluminum foil. After electrospinning, the fiber membranes on the aluminum foil were carefully removed from the collector for subsequent characterization.

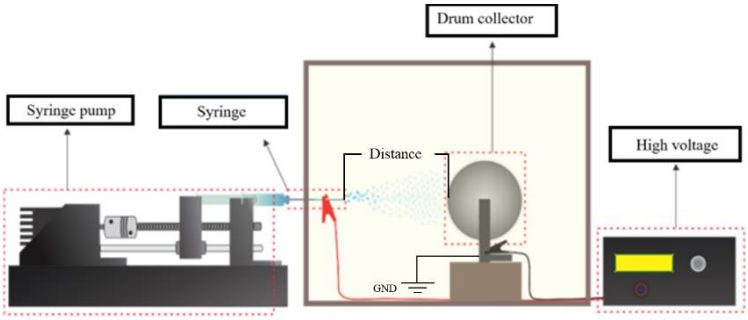


Figure 1. Schematic of electrospinning apparatus

4. Characterization of PS-Ag Composite Fiber Membranes

Fiber diameter and morphology were analyzed using Scanning Electron Microscopy (SEM). Measurements were conducted at 100 points across the SEM images to calculate the average fiber diameter and distribution, employing ImageJ software for image analysis. The coefficient of variation (CV) was calculated as follows:

$$CV = \frac{\sigma}{\mu} \qquad (1)$$

where $\sigma\sigma$ is the standard deviation, and $\mu\mu$ is the average fiber diameter [23][24].

Energy-dispersive X-ray Spectroscopy (EDS) mapping complemented the SEM analysis by assessing the distribution of silver (Ag) within the composite fibers. EDS results were correlated with SEM images to confirm the presence of Ag in the fibers. Raman spectroscopy was utilized to examine molecular vibrations, with a laser power output set at 5.0 mW to enhance the signal-to-noise ratio of the spectra. Fourier Transform Infrared (FTIR) spectroscopy further analyzed the molecular interactions between polystyrene (PS) and silver (Ag) within the fiber membranes, covering a wavenumber range from 1500 to 4000 cm^{-1}. Both Raman and FTIR spectra were compared to identify and confirm the functional groups in the fiber membranes, providing insight into how the inclusion of Ag influences the fiber diameter.

RESULTS AND DISCUSSION

1. Morphology of Fiber Membranes from PS-Ag

Figure 2 presents the Scanning Electron Microscopy (SEM) images of the PS, PA1, PA2, and PA3 samples. The

images reveal that all samples possess fibers with smooth surfaces and are devoid of beads, indicating that increasing concentrations of silver nitrate ($AgNO_3$) do not adversely affect the fiber morphology. Notably, the PA3 sample, as shown in Figure 2(d), exhibits white spots on the fiber surface, suggesting silver (Ag). Further characterization was conducted using Energy-Dispersive X-ray Spectroscopy (EDS) to substantiate the presence of Ag within the fibers.

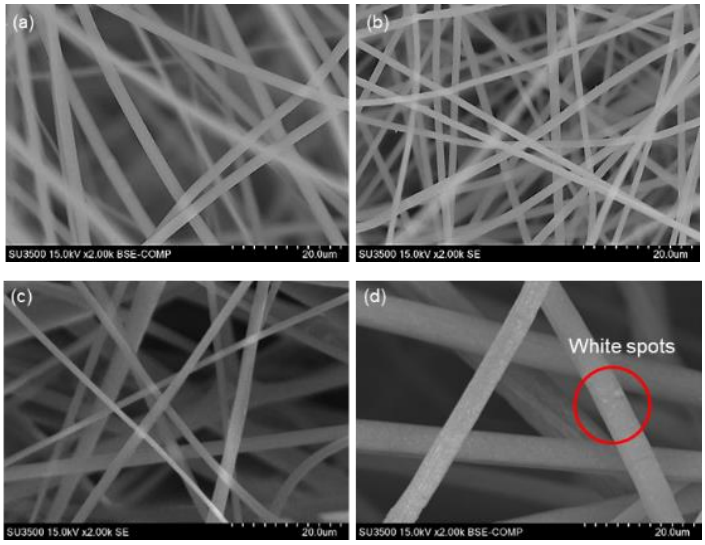


Figure 2. Characterization of SEM morphological results (a) PS, (b) PA1, (c) PA2, and (d) PA3.

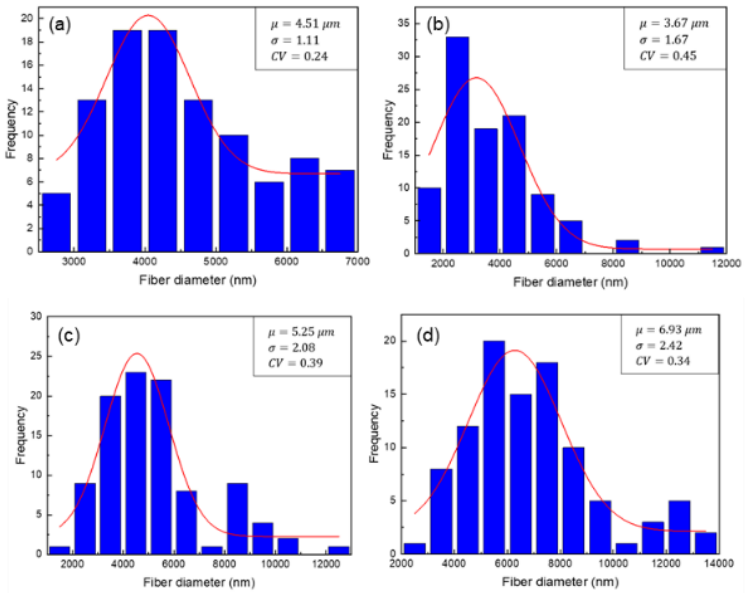


Figure 3. Graph of fiber membrane size distribution (a) PS, (b) PA1, (c) PA2, and (d) PA3.

Figure 3 illustrates the size distribution of the PS, PA1, PA2, and PA3 samples, determined from SEM image analysis using Image software and graphically represented using Origin software. This figure also indicates the average fiber diameter obtained

By analyzing 100 different points on each SEM image with ImageJ software. The distribution graph in Figure 3 demonstrates that the average fiber diameters of PS, PA1, PA2, and PA3 fiber membranes are 4.51 µm, 3.67 µm, 5.25 µm, and 6.93 µm, respectively. The coefficients of variation (CV) for the fiber membranes of PS, PA1, PA2, and PA3 are 0.24, 0.45, 0.39, and 0.34, respectively. A CV greater than 0.3 suggests that the PS-Ag fiber membranes exhibit non-uniform fiber sizes. However, this does not necessarily imply poor fiber quality; rather, it indicates less uniformity in size. The absence of beads on each fiber membrane sample attests to the good quality of the produced fibers, which are likely to possess robust mechanical strength in practical applications [22]. The fiber diameter trends observed indicate that the initial addition of Ag decreases fiber diameter for PA1, followed by an increase as Ag concentration is further elevated in PA2 and PA3. This pattern may be attributed to changes in the solution's viscosity and surface tension due to Ag content variations [25], [26]. Alterations in surface tension relative to viscosity can influence fiber formation, potentially leading to finer fibers when surface tension is more significantly impacted or thicker fibers when viscosity predominates. These dynamics are crucial for optimizing electrospinning parameters to ensure the processability and quality of the fibers [27]. Increasing fiber diameter with higher Ag concentrations suggests that adding Ag influences the electrospinning solution's properties, impacting the resultant fiber morphology.

Table 2. EDS Characterization Mapping Ag content in PA1, PA2 and PA3 samples

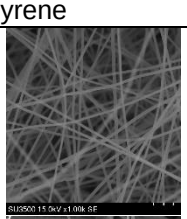
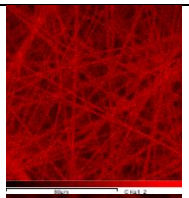
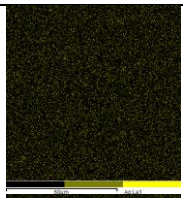
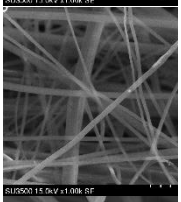
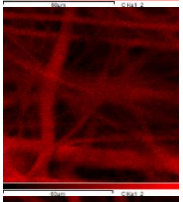
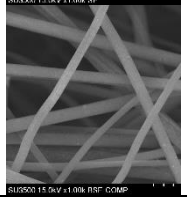
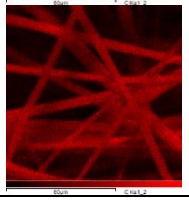
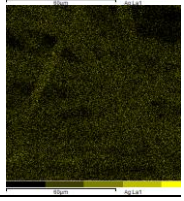
Sample	Fiber membranes of polystyrene	Carbon	Ag
PA1			
PA2			
PA3			

Table 2 presents the results from EDS characterization mapping for Ag content in the PA1, PA2, and PA3 samples. According to the table, yellow spots indicating the presence of Ag were detected in all samples. This observation aligns with the SEM image

analysis, where white spots also suggest the presence of Ag in the fiber membranes. Carbon (C) presence is also noted across all samples, marked by red spots. This finding is consistent with the expected chemical composition, as the fiber membranes primarily comprise polystyrene, which is predominantly Carbon. This EDS mapping thus confirms the incorporation and distribution of Ag within the polystyrene-based fiber membranes, reflecting successful composite formation.

Further characterization by EDS mapping, as depicted in Figure 4, substantiated the presence of Ag, evidenced by peaks around 3 keV in the PA1, PA2, and PA3 fiber membranes. The mapping data revealed that the PA1 sample, which contains 0.5 wt.% Ag in the PS solution exhibited an atomic Ag concentration of 0.12%—the PA2 sample, with 1 wt.% Ag content showed an atomic concentration of 0.38%—notably, the PA3 sample, which includes 2 wt.% Ag in the PS solution registered the highest atomic concentration of Ag at 0.73%. The increase in Ag concentration across the samples corresponds to the rising %At Ag values. In addition to Ag, the EDS analysis identified the presence of carbon (C) in all PS-based samples (PA1, PA2, and PA3) and the pure PS sample. This observation aligns with the primary chemical composition of polystyrene-based fiber membranes.

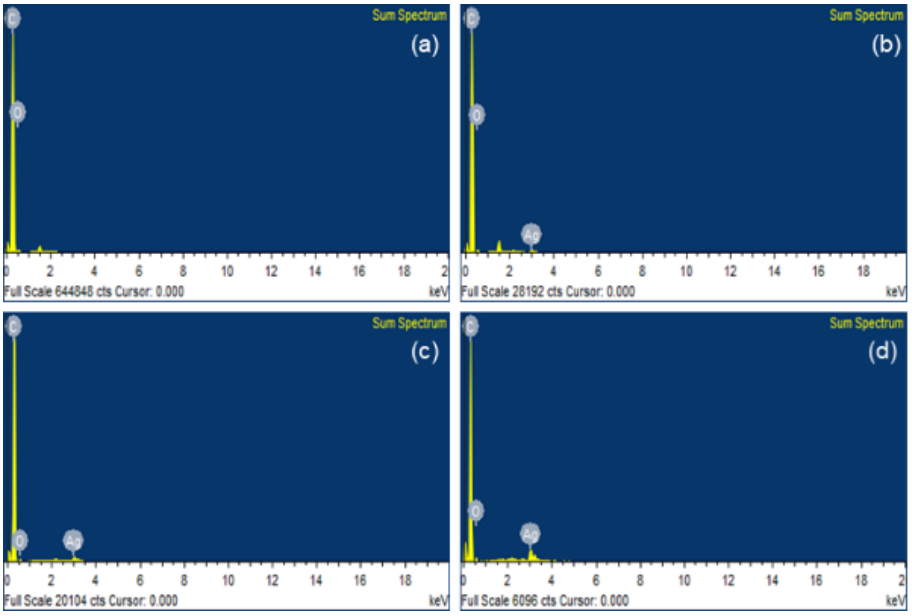


Figure 4. Quantitative analysis of EDS mapping of sample content characterization (a) PS, (b) PA1, (c) PA2, and (d) PA3.

2. Raman Spectroscopy Analysis

Raman spectroscopy analysis of the PS, PA1, PA2, and PA3 fiber membranes, as depicted in Figure 5, revealed distinctive spectral features. The presence of PS in the fiber membranes was confirmed by peaks at approximately 1000 cm^{-1}, with a specific peak at 1029 cm^{-1} attributed to C-H bending. The most intense bands observed at 1582 cm^{-1} and 1601 cm^{-1} indicate the C=C bonds and ring-skeletal stretching within the PS structure [28]. Moreover, a significant

broadening effect was noted in the range of ~1300 cm^{-1} to ~1600 cm-1, indicating an increase in intensity, which suggests the formation of COOH bonds due to sp3 hybridized carbons on oxidative cleavage [29]. This reaction might be catalyzed by Ag, which facilitates the coordination of the deprotonated form of carboxylate on the PS fiber surface [30].

The observed broadening also likely signifies the degradation of PS, a process typically catalyzed by metals with high redox potentials, such as cobalt. This study utilized Ag as the catalytic metal, resulting in weak bands between ~1200 cm^{-1} and ~1600 cm^{-1} under a laser power of 5 mW. This alteration contrasts with the Raman spectra of polystyrene fiber membranes before adding Ag. Thus, it can be inferred that the integration of Ag significantly influences the structural integrity and chemical properties of the polystyrene fiber membranes, impacting both the catalyst and the overall stability of the material.

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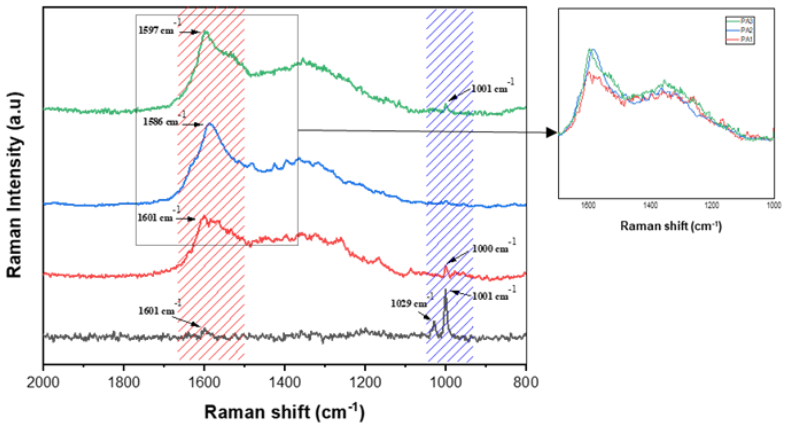

Figure 5. Raman Image Spectroscopic (a) PS, (b) PA1, (c) PA2, and (d) PA3

The degradation process in the porous fiber membranes likely involved oxidation. Oxygen within the fibers, combined with the high temperature generated by the 5 mW laser power, caused the polystyrene to undergo oxidative degradation, a phenomenon common in polymeric hydrocarbons. When exposed to laser power, Ag(I) is transformed into Ag(II) due to an oxidation reaction termed a laser-oxidative reaction, as evidenced by the appearance of carboxylic acid in the Raman spectra. This transformation induces instability and contributes electrons to the polystyrene, thereby promoting its degradation [31]. The proposed reaction mechanism is illustrated below [31].

$$-[CH\text{-}CH_2]- + Ag\ (II) \rightarrow -[\overset{\bullet}{C}\text{-}CH_2]- + H^+ + Ag\ (I)$$

$$-[\overset{\bullet}{C}\text{-}CH_2]- + O_2 \rightarrow -[OOC\text{-}\overset{\bullet}{C}H_2-]$$

$$-[OOC\text{-}\overset{\bullet}{C}H_2]- + H^+ + Ag\ (I) \rightarrow -[(OH)OC\text{-}CH_2]- + Ag\ (II)”$$

Data in Figure 5 also indicates that higher concentrations of Ag in the polymer result in more pronounced peak degradation. To mitigate the degradation of fiber membranes, a reduction in laser power to below 5 mW is recommended. However, this adjustment significantly extends the exposure time required to obtain the necessary graphs, thus necessitating further optimization.

3. Fourier-transform Infrared (FTIR) Spectroscopy

Fourier-transform Infrared (FTIR) Spectroscopy results, as depicted in Figure 6, reveal the spectra of fiber membranes doped with Ag. The IR spectra of PS fiber membranes and PS-Ag fiber membranes (in samples PA1, PA2, and PA3) show no significant differences. The characteristic bands ranging from 3100 cm-1 to 3000 cm-1 correspond to the =C-H stretching vibrations associated with aromatic rings [32]. Additionally, peaks at 2921 cm^{-1}, 2922 cm^{-1}, 2920 cm^{-1}, and 2916 cm^{-1} are attributed to symmetric and asymmetric stretching of CH_2 groups. Moreover, the aromatic bond (C=C) stretching vibration of the benzene ring is evident at the wave number 1682 cm^{-1} [33], [34].

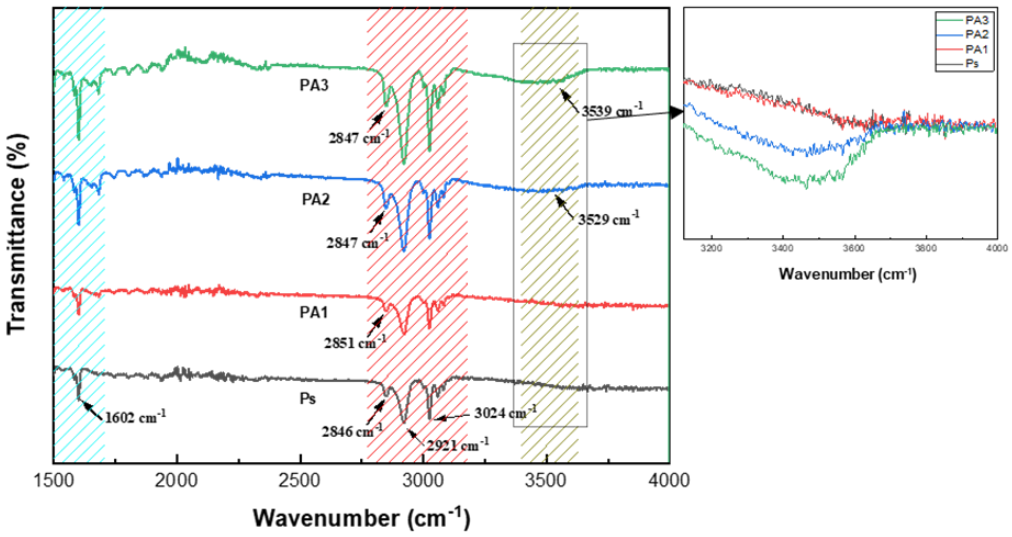

Figure 6. FTIR spectrum of fiber membranes (a) PS, (b) PA1, (c) PA2, and (d) PA3

It should be noted that the presence of OH groups is indicated by the peak at approximately 3400 cm^{-1}, demonstrating more pronounced buckling at 2% Ag concentration [31]. This increase in -OH groups is corroborated by Raman analysis, which suggests that the -OH groups may arise due to oxidative degradation from laser exposure during testing, indirectly signaling the presence of Ag as an electron donor [31]. However, FTIR analysis suggests that -OH could stem from external factors, such as environmental exposure. Related to the observations in Figure 6, the increase in -OH content correlates with an increase in fiber diameter in samples PA2 and PA3. This suggests that the -OH detected in the FTIR analysis might be attributed to external water

adsorbed during the aging process due to the microstructure of the fibers. This external water adsorption could increase the humidity within the fibers, thereby reducing their moisture resistance, which could lead to clogging and diminished performance in air filtration applications.

Moreover, more -OH groups in the fiber microstructure suggest increased hydrophilicity. This characteristic could enhance the filtration flux and improve performance [35]. Conversely, in air filtration applications, increased hydrophilicity could lead to higher moisture absorption by the fibers, potentially causing them to clump together and reduce the permeability to airborne particles, ultimately resulting in lower flux and poorer performance [34]. Therefore, while a hydrophilic membrane may be advantageous for water filtration, air filtration applications generally benefit more from hydrophobic fiber membranes.

CONCLUSION

The synthesis of 25% (w/w) polystyrene (PS) fiber membranes has been successfully carried out using various concentrations of silver nitrate ($AgNO_3$) viz. 0.5, 1, and 2 wt.% in PS solution. The addition of Ag has no impact on the morphology of the fiber membrane. Still, it has been proven to impact the membrane diameter of PA1, PA2, and PA3 fibers, which measure 3.67, 5.25, and 6.93 µm, respectively. From the EDS mapping result, the Ag was also well-distributed within the body of PS-Ag fiber membranes. However, Raman spectra results led to degradation in the fiber membranes, causing strong broadening near ~1300 cm^{-1} to ~1600 cm^{-1} with intensity. Addressing fiber membrane degradation involves reducing laser power to below 5 mW, although requiring significant optimization due to prolonged exposure times needed to acquire necessary data graphs. The FTIR results showed that the wavelength of ~3500 cm^{-1} indicated the presence of OH content in samples PA2 and PA3. The presence of OH indicates that the PS-Ag fiber membrane is hydrophilic, so it has the potential for water filtration application.

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