High-Efficiency Water Filtration by Electrospun Expanded Polystyrene Waste Nanofibers

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ABSTRACT: Nanofiber membranes were successfully synthesized from expanded polystyrene (EPS) waste with the addition of poly(vinylpyrrolidone) (PVP) for water microfiltration using the electrospinning method. The EPS-based nanofiber membranes exhibited a smooth morphology and were uniform in size. The concentration of the EPS/PVP solution changed some of the physical parameters of the nanofiber membrane, such as viscosity, conductivity, and surface tension. Greater viscosity and surface tension increase the nanofiber membrane diameter, whereas the addition of PVP results in hydrophilicity. Additionally, increasing the pressure increased the flux value of each variation of the nanofiber membranes. Furthermore, the rejection value was 99.99% for all variations. Finally, the use of EPS waste for nanofiber membranes is also beneficial for decreasing the amount of EPS waste in the environment and is an alternative to the current membranes available in the market for water filtration applications.
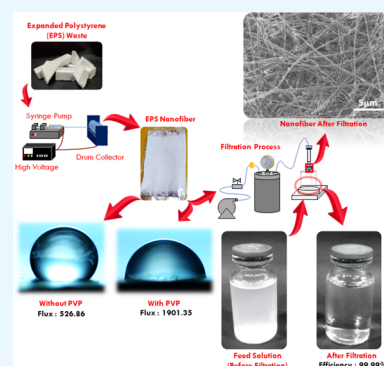
1. INTRODUCTION

Plastic waste originating from commonly used products has attracted global concern.[1] The increasing volume of plastic waste corresponds with the increasing global population, which unfortunately does not match the amount of recycling or reduction efforts.[2−4] Plastic waste is categorized as a highly nonbiodegradable material that requires hundreds or even thousands of years to degrade.[5] The World Bank has reported that approximately 760,000 tons per day of plastic waste is generated in Asia, which is predicted to increase up to 1.8 million tons or approximately 5.2 million m³ per day by 2025.[6] Typically, plastic waste is treated by burying it in the ground (landfill) or by burning it (incineration).[7] However, both these methods have a negative impact on the environment owing to air, soil, and water pollution.[8,9]

Expanded polystyrene (EPS) is a plastic waste containing harmful substances, such as benzene and styrene.[10−12] EPS is the second largest volume polymer after polyethylene, with more than 43 million tons in 2015 worldwide,[13] and it is extensively used in food packaging, electronics, craft materials, decorations, and building materials.[14] EPS has several properties, such as versatility, low cost, dimensional stability, low thermal conductivity, and low density.[15,16] As a plastic material, it is difficult to achieve degradable and disposable properties.

Thus, researchers have attempted to modify EPS into beneficial materials, one of which is nanofiber membranes prepared using an electrospinning method with various solvents such as N,N-dimethylformamide (DMF), D-limonene,

cyclohexane, 1,2-dichloroethane, tetrahydrofuran, dichloromethane, and N,N-dimethylacetamide.[16] Electrospinning is a method of fiber spinning that utilizes electrified polymers, both in solution and in melt form.[17] Several studies have confirmed that electrospinning is one of the most economical, simple, and facile methods for fabricating nanofibers.[17−21] Moreover, this technique allows the modification of the size,[22] morphology,[23] and composite formation of the fibers with other materials to enhance membrane functionalization.[24] Rajak (2020) synthesized an electrospun EPS nanofiber membrane using a DMF solvent for application in air filters, which demonstrated that EPS nanofiber membranes have a high air filtration efficiency of approximately 99.99%.[15] The versatility of electrospinning has been exploited to achieve high-quality water filtration systems.[25−30] Considering the broad applications of EPS nanofiber membranes, it is essential to conduct studies on their potential use in water filtration applications. However, owing to the low wettability of EPS nanofiber membranes, it is necessary to add a hydrophilic material to achieve sufficient wettability. Poly(vinylpyrrolidone) (PVP) is a promising material for achieving hydrophilicity.[31] PVP reduces the water contact angle (WCA) and accelerates the formation of

the Wenzel state of the nanofiber membrane.[32] In this study, the synthesis of nanofiber membranes from EPS waste with different PVP contents as a water filtration medium is discussed. The EPS nanofiber membranes with varying PVP content are fabricated using the electrospinning method.

2. MATERIALS AND METHODS

The source of EPS waste used in this study was electronic packaging, and PVP was supplied by Sigma-Aldrich (molecular weight 1,300,000), while DMF was used as the solvent. The EPS waste was pretreated prior to dissolution in DMF. To ensure that the EPS waste was adequately clean, thorough cleaning with distilled water was performed, followed by drying at ambient temperature. After drying, the EPS waste was cut into small pieces, approximately 1.5 cm in length. The EPS waste was dissolved in DMF using a magnetic stirrer for 6 h at 50 °C. Additionally, in this study, EPS waste with added PVP was prepared. Table 1 lists the detailed composition of the EPS waste with added PVP.

Table 1. Composition of the Samples

sample name	EPS waste (g)	PVP (g)	DMF (mL)
E1P0	1.5		8.5
E2P1	1	0.5	8.5
E1P1	0.75	0.75	8.5

The nanofiber membranes were synthesized using an electrospinning apparatus (ILMI-N101, Integrated Laboratory of Materials and Instrumentation, Department of Physics, ITB, Bandung, Indonesia). The precursor solution was poured into a 10 mL syringe with a 0.5 mm internal diameter needle and a flow rate of 0.7 mL h^{-1}. The needle was connected to a high-voltage source (12 kV), and the distance between the syringe pump and the drum collector was 10 cm. The drum collector had a diameter and length of 5.5 and 12 cm, respectively. The drum was wrapped with aluminum foil as the substrate to collect the fabricated EPS nanofibers. Electrospinning was continuously performed for 6 h at a temperature of 25 °C and relative humidity of 60% to obtain nanofibers of similar thickness.

The viscosity and surface tension of the as-synthesized precursor solutions were measured at 25 °C using a Fenske-Oswald viscometer (Fisher) and du Noüy tensiometer (Fisher), respectively. The crystallinity of the EPS/PVP nanofiber membrane was characterized using room-temperature X-ray diffraction (XRD, Miniplex 600, Rigaku, Japan) in the 2θ range of 10−70° using Cu Kα radiation as the X-ray source. The functional groups in the EPS and EPS/PVP nanofiber membranes were analyzed using Fourier transform infrared spectroscopy (FTIR, NICOLET IS10, Thermo Fisher Scientific) in the spectral range of 500−4000 cm^{-1}. The absorbance of the EPS and EPS/PVP nanofiber membranes was characterized using a double-beam ultraviolet−visible (UV−vis) spectrometer (LUS-B13, Labtron, U.K.). Moreover, a UV−vis spectrometer was used to measure the antacid concentration before and after the permeability experiments. The wettability of the EPS and EPS/PVP nanofiber membranes was evaluated by measuring the WCA using a contact angle measurement apparatus (Nachriebe 320, Center for Aerosols and Analytical Instrumentation, Department of Physics, ITB, Indonesia). The morphology of the EPS and EPS/PVP nanofiber membranes was characterized using

scanning electron microscopy (SEM, SU3500, Hitachi, Japan). Subsequently, the obtained SEM micrographs were processed using ImageJ software to determine the average fiber diameters and conduct statistical analyses.

3. RESULTS AND DISCUSSION

The electrospinning parameters used to synthesize nanofiber membranes included voltage, flow rate, humidity, and the distance between the syringe nozzle and collector, while the properties of the solution included conductivity, viscosity, and surface tension.[21,33] The characteristics of nanofibers depend on the properties of the solution. Table 2 lists the properties of

Table 2. Properties of the EPS−PVP Solution at 25 °C

sample name	conductivity (μS/cm)	viscosity (cP)	surface Tension (dyne/cm)
E1P0	14.76 ± 0.01	30.16 ± 0.244	41.03 ± 0.01
E2P1	13.56 ± 0.00	49.03 ± 0.275	41.95 ± 0.01
E1P1	10.78 ± 0.01	60.79 ± 0.814	42.36 ± 0.01

the solution prepared for the synthesis of the nanofiber membranes. The viscosity of E1P0 was 30.16 cP and increased twofold with the addition of PVP (E1P1). The increase in viscosity was owing to the higher molecular weight of PVP compared with that of EPS. A higher viscosity produces fibers with larger diameters because of the stability of the jet.[34] Conversely, when the EPS/PVP ratio was two (E2P1), the viscosity decreased to 49.03 cP. Moreover, the addition of PVP influenced the solution conductivity, which changed from 14.76 to 10.78 μS/cm for E1P0 to E1P1, respectively. This trend is in agreement with other studies, which shows a decrease of conductivity with increasing the amount of PVP.[35,36] In contrast to the viscosity, the higher conductivity causes higher stretchability, resulting in a smaller fiber diameter. Although PVP addition also affected the surface tension of the solution, the difference in surface tension was insignificant. Therefore, the conductivity and viscosity of the solution are more likely to influence the final morphology of the fibers.

Figure 1 shows SEM micrographs of the nanofiber membranes synthesized from the EPS waste, and it was concluded that the nanofiber membranes were successfully synthesized by electrospinning. As discussed previously, the fiber morphology depends on the properties of the solution. As expected, the average fiber diameter of E1P1 (1322 ± 374.5 nm) was higher than those of the other samples owing to the high viscosity of the E1P1 solution, as listed in Table 2. The average fiber diameter of E1P0 was 589 ± 106.4 nm, which was the lowest value owing to the low viscosity and high conductivity of the solution. Meanwhile, the fiber diameter of E2P1 was 701.7 ± 106.6 nm. The uniformity of the fiber diameter was estimated by calculating the coefficient of variation (CV) using eq 1.

$$CV = \frac{\sigma_f}{\mu_f} \tag{1}$$

where σ_f is the standard deviation and μ_f is the average fiber diameter. As shown in Figure 1, the distributions of E1P0 and E2P1 were significantly narrow, indicating a CV of <0.2. According to Matulevicius et al., a CV of <0.3 is considered to be indicative of a uniform fiber.[33] Although E1P1 had a CV of 0.28, which indicates a uniform distribution of the fiber
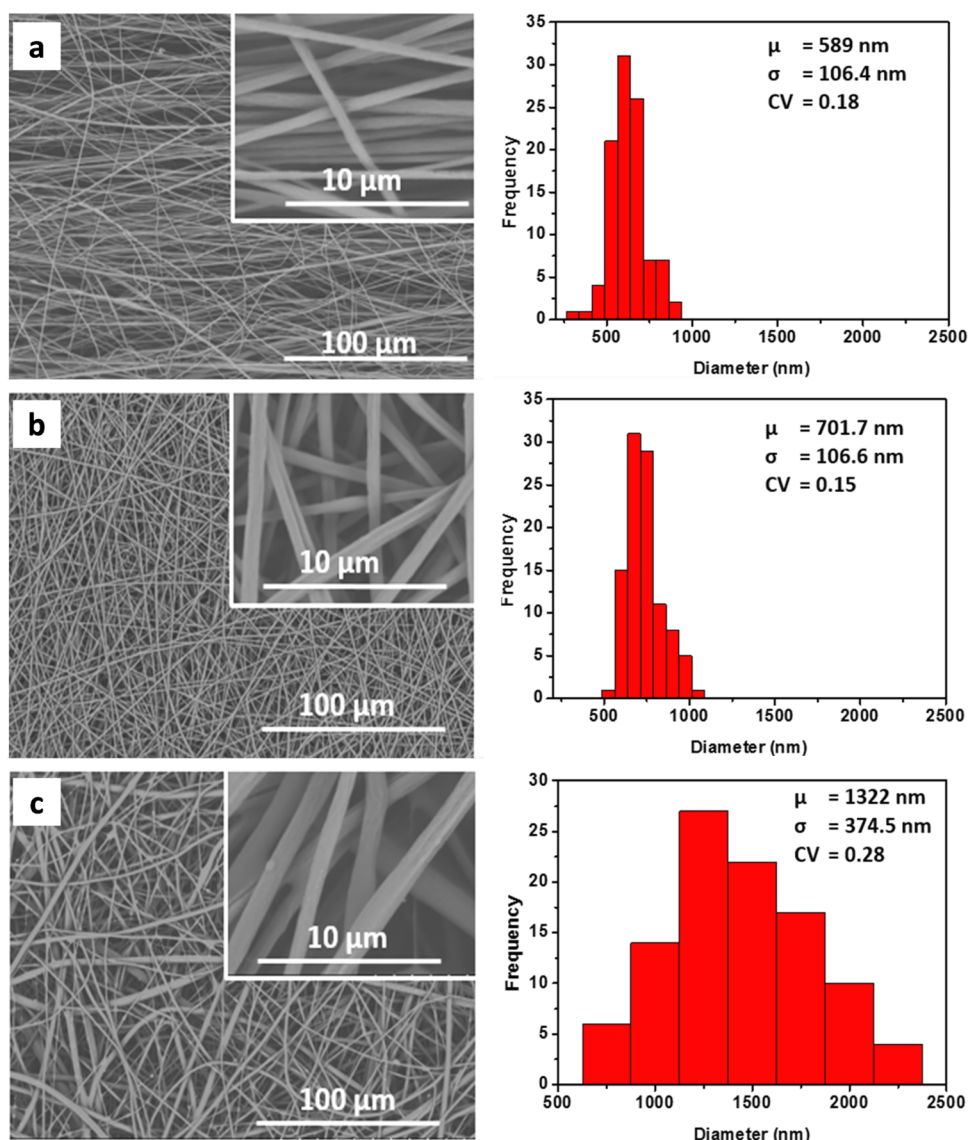
Figure 1. SEM micrographs of the nanofiber membranes (a) E1P0, (b) E2P1, and (c) E1P1, including the average fiber diameter and its distribution.

diameter, the variance was significantly large compared with E1P0 and E2P1. Porosity is another important property of nanofiber membranes. In this study, analytical calculations were used to determine the porosity of the nanofiber membranes[37] using eq 2.[37−40]

$$\Phi = 1 - \frac{V_{solid}}{V_{total}} \times 100\% \qquad (2)$$

where Φ is the nanofiber membrane porosity, V_{solid} is the nanofiber volume, and V_{total} is the membrane volume. The porosity nanofiber membranes of E1P0, E2P1, and E1P1 were 63.43 ± 0.46, 64.45 ± 6.00, and $65.57 \pm 0.80\%$, respectively. Although the fiber diameter of E1P1 increased by more than two times compared with that of E1P0, the porosity was not significantly different.

As shown in Figure 2, the XRD patterns indicate the crystallinity of the samples. As expected, all of the samples were in the amorphous state, which is typical of polymer materials.[15] However, relatively sharp peaks were observed in the range of $18-20°$ for E1P0, which may be attributed to polystyrene,
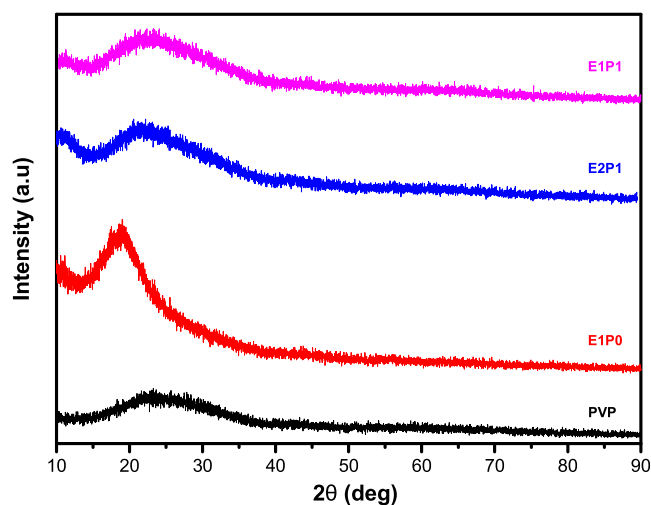

Figure 2. XRD spectra of the EPS and EPS/PVP nanofibers.

although the peaks associated with bulk EPS exhibited a higher intensity.[41] The addition of PVP clearly altered the E2P1 and E1P1 peaks. However, the difference between E2P1 and E1P1 was indistinguishable by XRD. The typical amorphous structure of the nanofiber membranes may be caused by the rapid phase change from liquid to solid during the electrospinning process.[42]

Figure 3 shows the FTIR spectra of the nanofiber membranes. The characteristic peaks of polystyrene were
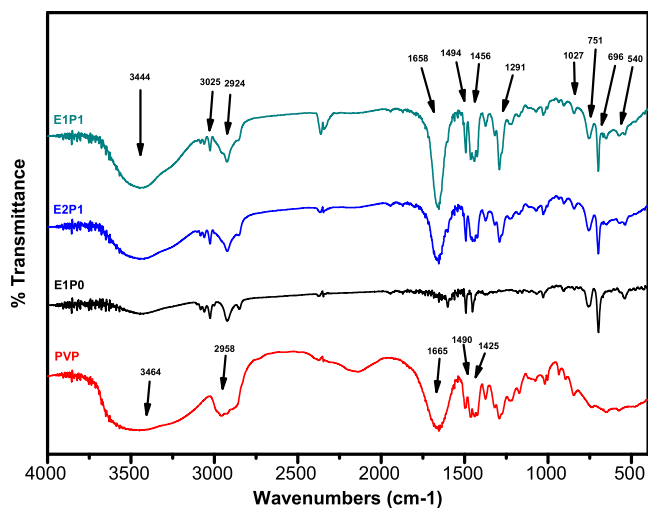

Figure 3. FTIR spectra of E1P0, E2P1, and E1P1, with the FTIR spectrum of PVP for comparison.

observed in the nanofiber membrane. The FTIR range from 3100 to 3000 cm^{-1} represents the C−H aromatic stretching vibration of the benzene ring, and the peaks at 2924 and 3025 cm^{-1} demonstrate the asymmetric and symmetric stretching vibrations of CH$_2$. Other characteristic peaks were observed in the range of 1658−1456 cm^{-1}, attributed to the C=C bond stretching vibration of the benzene ring.[15] The peaks at 1300−1000 cm^{-1} were attributed to C−H in-plane bending vibration, those at 900−600 cm^{-1} were attributed to C−H out-of-plane bending vibration, and those at 751, 696, and 540 cm^{-1} were attributed to C−H deformation vibration of the monosubstituted benzene ring.[32,43] Conversely, PVP exhibited the characteristic peaks for the O−H stretching of the hydroxyl group (3464 cm^{-1}), asymmetric CH$_2$ stretching vibration (2956 cm^{-1}), carbonyl (C=O) group (1665 cm^{-1}), and bending vibration of the C−H bond (1490−1425 cm^{-1}). The EPS−PVP solution exhibited a new peak and a sharper peak at 3444 and 1658 cm^{-1}, which were attributed to the stretching of the O−H and C=O bonds, respectively. Another study has

also reported that strong hydrogen bonds are generally indicated by the presence of a C=O strain band 1658 cm^{-1}.[44] Moreover, this may be owing to the interaction of the carboxyl group in the pyrrolidone ring with the hydrocarbon groups of benzene.[45] It is noteworthy that no trace of DMF was detected in the E1P0, E2P1, and E1P1 samples, indicating that it completely evaporated during the electrospinning process. Notably, the FTIR peaks for DMF are typically observed at 1673, 1389, 1256, and 1096 cm^{-1}.[46]

The hydrophobicity or hydrophilicity of the nanofiber membranes was measured using the water contact angle on the surface of the membranes, as shown in Figure 4. Hydrophobic properties are indicated by a contact angle of ≥90°. In contrast, a contact angle <90° indicates a hydrophilic membrane.[20,21] The E1P0 membrane exhibited hydrophobic behavior with a water contact angle of 140.46° (Figure 4a), and the addition of PVP altered the water contact angle to 84.70° (Figure 4c). Thus, PVP addition modifies the EPS membrane surface from hydrophobic to hydrophilic. The hydrophobic behavior of the nanofiber membrane returned to its original state upon increasing the EPS content, as observed for E2P1 (Figure 4b), with a water contact angle of 130.42°. The addition of PVP reduced the water contact angle and accelerated the formation of the Wenzel state of the nanofiber membrane, thus facilitating the filtration process.[31,47,48]

In addition to the characteristics of the nanofiber membranes, their performance was examined by pure water flux (PWF) and particle rejection. PWF is a measure of the ability of pure water to pass through the nanofiber membrane. The PWF was obtained from experimental data by considering the following parameters: permeate volume (V), membrane area (A), and filtration time (t). The following equation expresses the experimental approach to flux[49,50]

$$J = \frac{V}{A.\, t} \tag{3}$$

Figure 5a shows the PWF of the nanofiber membrane at a fixed pressure of 0.25. The average PWF for the E1P0, E2P1, and E1P1 nanofiber membranes were 526.86, 1175.25, and 1901.35 L m^{-2} h^{-1}, respectively. This result is extraordinary; with the addition of PVP, the PWF of E1P1 was significantly increased by more than three times that of E1P0. This suggests that surface modification caused by the presence of PVP plays an important role in enhancing the PWF. As discussed previously, the presence of PVP alters the membrane behavior from hydrophobic to hydrophilic, which is more likely to affect the PWF. Moreover, although E2P1 behaved similarly to E1P0 in terms of the water contact angle, the presence of PVP increased the PWF of E2P1 by more than two factors compared with that of E1P0. Porosity is another property that
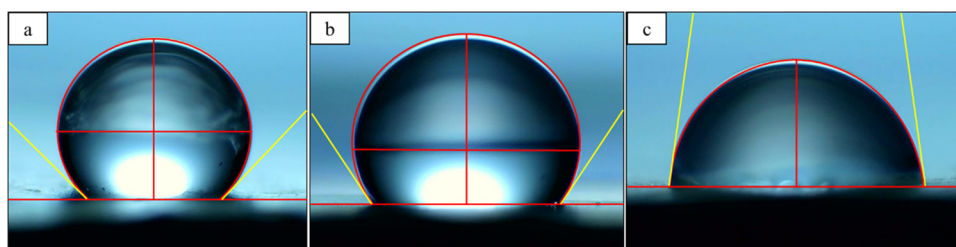

Figure 4. Images of a water droplet on the surface of the (a) E1P0, (b) E2P1, and (c) E1P1 nanofiber membranes, showing that the hydrophilic behavior changed to hydrophobic owing to PVP addition.
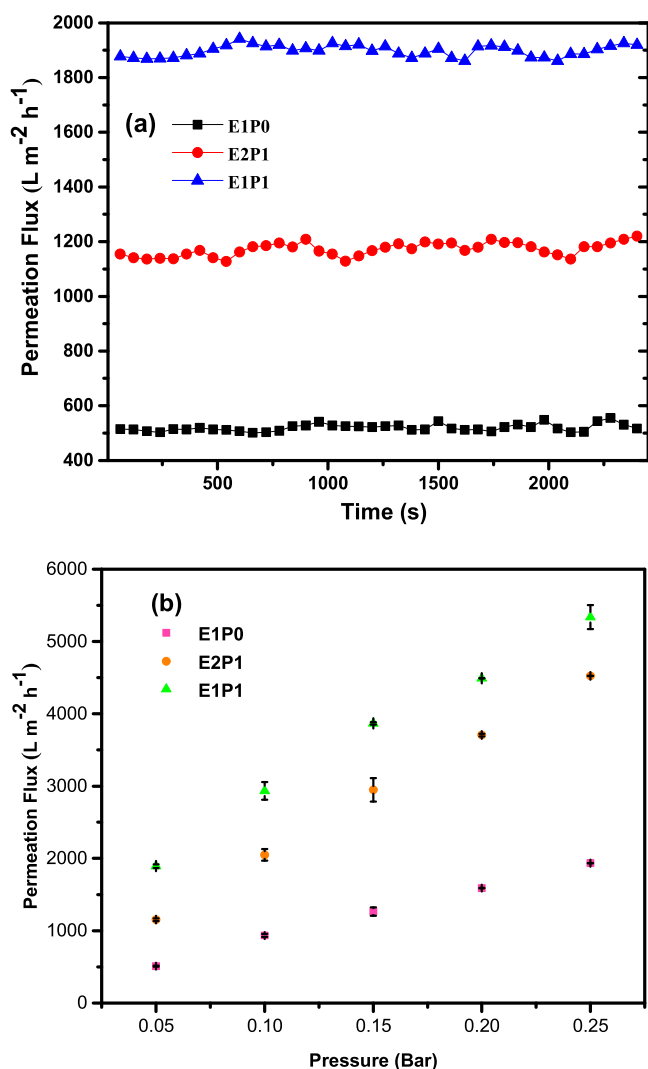
Figure 5. Permeability Evaluation: (a) Permeation flux over time at 0.25 bar; (b) permeation flux at different pressures.

may influence the PWF; however, the porosity of the samples was not significantly different, that is, 63−65%. Therefore, the effect of porosity on PWF is negligible in this study.

Figure 5b shows the PWF of the nanofiber membranes at various pressures. The PWF increased with increasing applied pressure, indicating that the relationship between the flux and pressure is in agreement with Darcy's law, as expressed by eq 4[50,51]

$$J = \frac{K}{\mu} \frac{\Delta P}{\Delta x}$$

(4)

where K is the permeability constant, ΔP is the differential pressure, μ is the dynamic viscosity, and Δx is the membrane thickness. Notably, the addition of PVP exhibited similar behavior during the permeation flux test. The slopes of E1P1 and E2P1 were almost identical, with values of 180.79 and 171.15°, respectively, independent of the EPS content. In contrast, the slope of the linear fit for E1P0 was 68.99°. This result indicates that the addition of PVP affects the surface interactions between water and the nanofiber membranes.

The rejection parameter was determined by passing an antacid suspension through the membranes. The antacid had an average particle size of 971 ± 234 nm, as determined by

particle size analysis. A calibration curve was used to calculate the unknown concentration from the permeate;[52] therefore, several solutions with different antacid concentrations (0, 317, 625, 1250, 1875, and 2500 ppm) were prepared for calibration. A 2500 ppm antacid feed solution was prepared to evaluate the particle rejection performance of the nanofiber membranes. Particle rejection (%) was calculated by comparing the antacid concentration in the feed (antacid concentration before filtration, C_f) and permeate (antacid concentration after filtration, C_p) suspensions as follows

$$\text{particle rejection (\%)} = \left(1 - \frac{C_f}{C_p}\right)$$

(5)

Figure 6 shows the permeation ability of the nanofiber membranes to withstand the antacid particles under an applied
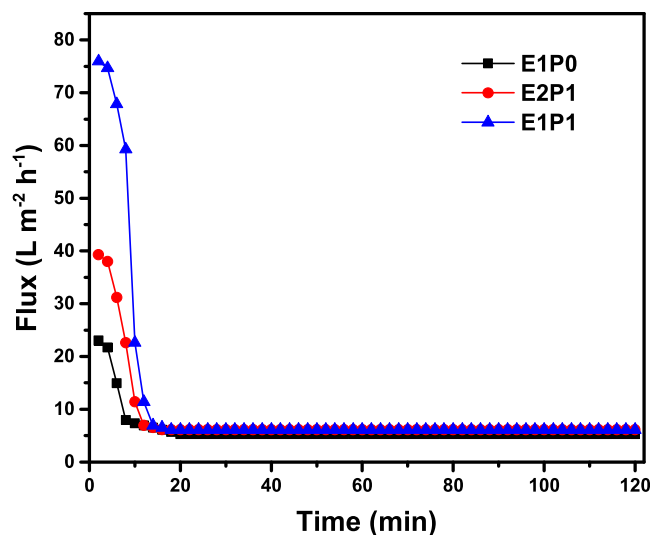

Figure 6. Permeation flux during the filtration process at 0.25 bar.

pressure of 0.25 bar. Notably, the particle rejection for the samples, determined using eq 5, was 99.99%. Considering the average particle size of the antacid, the performance of the nanofiber membranes falls within the category of microfiltration. As shown in Figure 7, the permeate collected after
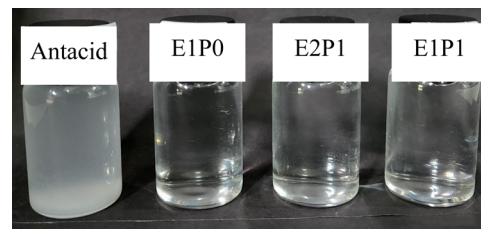

Figure 7. Images of water-suspended antacid solutions before and after filtration using the E1P0, E2P1, and E1P1 membranes (photo was taken by Alfian Noviyanto).

filtration was very clear, similar to pure water, which corresponds with the determined particle rejection of 99.99%. Although E1P0 had a lower flux than the other samples, its antacid rejection performance was similar to that of E1P1, which has a higher flux. Notably, a higher flux is preferred because the energy consumption is lower than that of a low-flux membrane.[53−55]
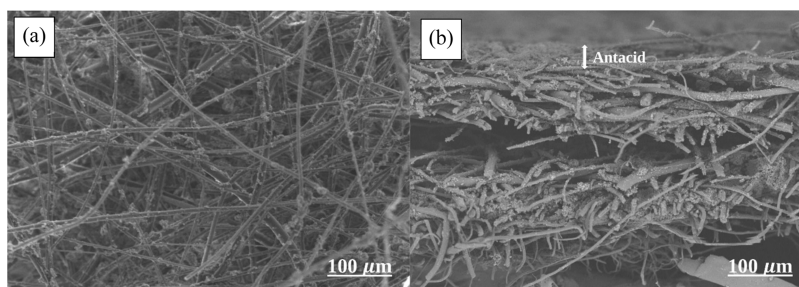
Figure 8. SEM micrographs of E1P1 after filtration: (a) surface and (b) cross section.

Table 3. Published Reports for Microfiltration Processes[b]

precursor solution (polymer/solvent)	fiber diameter (nm)	porosity (%)	flux (L m^{-2} h^{-1})	efficiency (%)
CA/acetic acid−acetone[56]	500−1700	N.A.	27,900[a]	87.7
PAN/DMF[57]	N.A.	72−79	1600	98.5
PAN/DMF[58]	100	75	71.2	99.3
PAN/DMF/DETA[59]	67−858	>90	68.98	<99
PVA/distilled water[60]	100−182	N.A.	5417[a]	95
PVA/PP[29]	2240	N.A.	32.346	96
PES/NMP[61]	550−1300	N.A.	16,000[a]	100
PVDF/DMAc/acetone[62]	N.A.	N.A.	35−50	>99
PVDF/DMF/acetone[63]	N.A.	N.A.	19.3−22.5	>90
PVDF-HFP, DMF, THF, acetone[64]	1285	72−87	34	>97
SBS/DMF-THF[65]	235−457	81	11.2	<99
E1P0	589	63	526.86	99.99
E2P1	701.7	64	1175.25	99.99
E1P1	1322	65	1901.35	99.99

[a]L m^{-2} h^{-1} bar^{-1}. [b]CA, cellulose acetate; PAN, polyacrylonitrile; PVA, polyvinyl alcohol; PES, polyether sulfones; PVDF, polyvinylidene fluoride; SBS, styrene butadiene styrene; NMP, N-Methyl-2-pyrrolidone; THF, tetrahydrofuran.

Figure 8 shows the SEM micrographs of E1P1 after filtration of the water-suspended antacid. As shown in Figure 8a, the particles were attached to the surface of the fiber. As shown in Figure 8b, the filtering process occurred in the depth layer. Therefore, it was concluded that initially, the antacid particles are suspended on the surface of the membrane. The smaller antacid particles migrate through the inner layer and are trapped by other nanofibers, resulting in a clear permeate, which agrees with the 99.99% particle rejection. Although the EPS-based nanofiber membrane achieves a high rejection efficiency and filters particles rapidly, in practice, deep clog cleaning must be performed regularly to maintain the performance of these nanofiber membranes. Notably, for a membrane that regularly undergoes deep clog cleaning, the evaluation of the mechanical properties of the membrane is essential to determine its durability. In summary, EPS/PVP nanofiber membranes are an alternative to current membranes for water filtration applications because of their high rejection efficiency.

Table 3 lists published reports on microfiltration processes. To the best of our knowledge, the use of EPS waste as a source for synthesizing nanofiber membranes has rarely been reported thus far. Moreover, the addition of PVP for the surface modification of EPS has not been reported previously. Therefore, this study presents the first report that utilizes EPS waste combined with PVP for water filtration applications. As listed in Table 3, the fiber diameters of E1P0, E2P1, and E1P1 were relatively larger than those of the other precursors, especially PAN. However, compared with other raw materials, EPS waste is a promising and inexpensive material. Therefore, the use of EPS waste to produce nanofiber membranes is

industrially valuable. Moreover, the performance of the membrane was excellent, with a particle rejection rate of 99.99%. Compared with the other membranes, the flux was considerably higher, although the PWF analysis was performed at a low pressure (0.25 bar). Therefore, these nanofiber membranes are suitable for household applications, such as laundry wastewater treatment. Finally, the use of EPS waste for nanofiber membranes is also beneficial for decreasing the amount of EPS waste in the environment, and it is an alternative to the current membranes available in the market for water filtration applications.

4. CONCLUSIONS

The EPS/PVP nanofiber membranes were successfully synthesized via electrospinning. The nanofiber membranes exhibited a smooth morphology and were uniform in size. The percentage of PVP in the solution influenced the nanofiber properties; for example, the nanofibers changed from hydrophobic (E1P0) to hydrophilic (E1P1). Moreover, the addition of PVP increased the nanofiber diameter from 589 ± 106.4 nm (E1P0) to 1322 ± 374.5 nm (E1P1), which was attributed to the increase in viscosity of the E1P1 solution. The PWF was influenced by PVP addition, resulting in an increase of 1901.35 L m^{-2} h^{-1} in E1P1 compared with that of E1P0 without PVP (526.86 L m^{-2} h^{-1}). Notably, the particle rejection of all of the nanofiber membranes was 99.99%. Finally, the use of EPS waste combined with PVP for nanofiber membranes is an alternative solution to decrease the amount of EPS waste in the environment, and the developed membranes may be used for high-performance water filtration.

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Notes

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