CHARACTERISTICS OF STYROFOAM WASTE-BASED MEMBRANE THROUGH VAPOR AND LIQUID-INDUCED PHASE INVERSION PROCESS

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Abstract

Polymeric membrane-based-Styrofoam waste in the form of a thin sheet was successfully prepared by a phase-inversion technique in different final solidification treatments, namely, immersion and evaporation. This study aims to identify the effects of different solidification processes on membrane properties such as hydrophobicity, pore configuration, porosity, and membrane temperature stability. Characterization was carried out using contact angle, SEM, FTIR, TGA, and porosity tests. The results showed that an increase in Styrofoam 18-30 wt.% in dimethylformamide (DMF) as solvent decreases the hydrophobicity by 9.5%. The average contact angle of 62–80° indicated that the obtained membrane was prepared by immersion treatment. The membrane subjected to evaporation treatment was hydrophobic. Moreover, the microscopy image shows that the immersed membrane was denser than the evaporated membrane. This showed that a higher exchange rate between the solvent and non-solvent (water) produced a tight membrane than free evaporation in air. The polystyrene membrane from Styrofoam exhibited excellent temperature stability up to 350 °C. In addition, the mechanical strength was affected by employing different solidification processes. The obtained results were also successfully tabulated from a statistical point of view to validate the conclusions. The following information can provide basic knowledge for modifying membrane-based-Styrofoam to optimize zero-waste goals.

***Keywords**: hydrophobic membrane, phase-inversion, polystyrene membrane, styrofoam waste*

Introduction

Membrane technology is a recommended solution that utilizes semi-permeable separation capabilities, inexpensive materials, low energy consumption, and ease of operation. Polymer-based membranes have been widely used because of their ease of manufacturing processes with membrane characteristics that can vary according to the desired performance (Jeon and Lee, 2015; Fang *et al.*, 2012). The membrane used in this study was a polymer membrane made from recycled Styrofoam to reduce the problem of Styrofoam waste. Styrofoam is a thermoplastic polymer of polystyrene from styrene monomers that is widely used in everyday life as food packaging, disposable utensils, and insulation materials. As a

result, Styrofoam waste has increased dramatically, which has a serious impact on the nature and health of living things, namely, the main formation of microplastics and suspected toxins (Yap *et al.*, 2021).

Polystyrene (PS) is a nonpolar compound that can be easily isolated using *n*-dimethylformamide (DMF), chloroform, and toluene solvents and then purified by precipitation (Baig *et al.*, 2019; Adamczak *et al.*, 2019). The use of Styrofoam in the manufacture of membranes can be a positive value because it has several advantages, including flexibility, is not easy to break, is hydrophobic, can help separate the water content in biogas, is not corrosive, and is relatively cheap (Ketut Sumarni *et al.*, 2013; Adamczak *et al.*, 2019). In membrane preparation, the phase-inversion process has been widely used as the best method for obtaining polymer membranes at an industrial level. The basic concept of the phase inversion process has been introduced in previous research (Kesting, 1985). The process of phase inversion in a polymer solution is not only thermodynamic, but also kinetic. Ternary diffusion, moving boundaries, and changes in the solvent concentration within the coagulation bath make the process very difficult to describe mathematically (Zheng *et al.*, 2006).

Based on previous research, polystyrene membranes prepared from Styrofoam waste were successfully fabricated using electrospinning, followed by evaporation as the final solidification process. Microscopically, the membrane was in the form of low-density nanofibers. However, some studies have also reported that phase inversion followed by immersion in water as a non-solvent produces polystyrene-based membranes with hydrophilic properties. Generally, the final membrane microstructure, including pore size, mean pore diameter, and porosity, is also determined by the rheology of the dope solution and coagulation non-solvent (Henson *et al.*, 2015). In addition, the type of polymer, concentration, thickness of the film, and precipitation method affect the pore membrane, which can be modified for microfiltration, ultrafiltration, nanofiltration, and reverse osmosis (Enger *et al.*, 2009). To the best of our knowledge, the use of both vapor- and liquid-induced phase inversion process methods on Styrofoam waste-based membranes has never been compared and fully reviewed in terms of the properties obtained by comparing the two methods.

Therefore, we studied a different final solidification for polymeric membrane preparation, that is, through vapor- and liquid-induced phase inversion processes, to obtain fundamental data to modify the membrane, which is also supported by data from statistical tabulation regarding the characteristics of polystyrene-based membranes.

Research Methods

Materials

Styrofoam is obtained from commercial stores as food packaging and is used as a basic polymer matrix for polystyrene (PS). N, N-dimethylformamide (DMF) (Merck-1033014,99%, Germany) was employed as the solvent, whereas water was used as the non-solvent to ensure the solidification of the membrane. The casting solutions were prepared with various compositions (w/w %) of Styrofoam in DMF, that is, 18, 22.5, 25, 27.5; and 30wt%.

Fabrication of styrofoam waste-based membrane

Polymeric membrane-based Styrofoam waste was prepared by a phase-inversion technique, which started with dope solution preparation (Nurherdiana *et al.*, 2021). The dope solution was prepared in six different concentrations from Styrofoam waste mixed with N, N-dimethylformamide. To

obtain homogeneous solutions, polystyrene (PS) solutions were stirred using a magnetic stirrer for 4 h, followed by ultrasonication to remove air trapped in the solution. This is because the air bubbles lead to the appearance of pore configuration defects. The ultrasonication process was run for 1 h, and the dope solutions were kept at room temperature for 24 h to achieve doping with free air bubbles (Sitorus *et al.*, 2022; Rahmiati *et al.*, 2018).

The dope was then casted on the flat glass support with a 0.5 mm- 1 mm thickness. Finally, the green membranes were subjected to two different solidification treatments, namely immersion and evaporation. During the immersion process, the membrane was immersed in water for 24 h at room temperature, while the remaining green membrane evaporated in air under room pressure and temperature (Iqbal *et al.*, 2018; Khadijah and Harun, 2016). Figure 1 presents the process of the phase-inversion method, highlighting that the green membranes were solidified by different treatments. Various concentrations of Styrofoam added to the solvent are listed in Table 1.

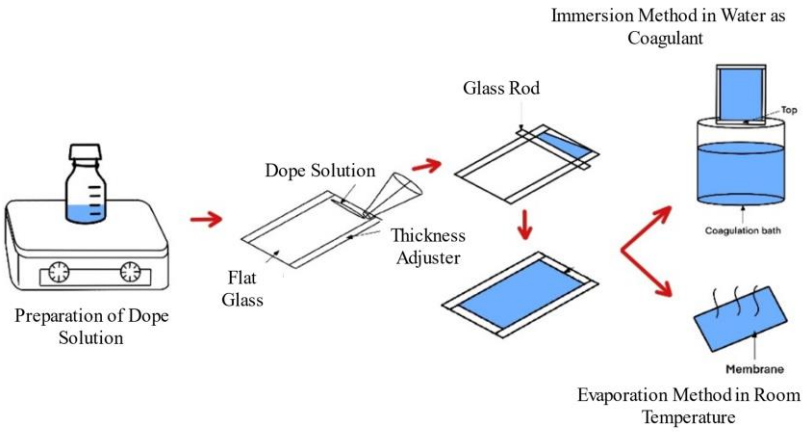


Figure 1. Phase inversion procedure for obtaining polystyrene membranes

Table 1. The various composition of styrofoam-based membranes.

Sample Code	Immersion	Evaporation
PS A	18	-
PS B	-	18
PS A22.5	22.5	-
PS A25	25	-
PS A27.5	27.5	-
PS A30	30	-

Characterization

The morphology of the PS membranes obtained from the Styrofoam waste was first investigated by scanning electron microscopy (SEM, Hitachi SU3500). The functional groups of the PS membranes were studied using Fourier-transform infrared spectroscopy (FTIR, Shimadzu). This provides information on the specific vibration characteristics obtained from molecular absorption and transmission spectra. Thermogravimetric analysis (TGA) is defined as a monitoring technique used to evaluate material stability in the presence of increasing temperature. The measurement conditions was employing temperature between 25-600°C with; heating rate, 5°C.min^{-1} under air flow. The water contact angle (WCA) of the membranes were measured manually by observing the sessile drop using images in JPEG format, which acquired using webcam software were opened with ImageJ, and then angles were determined using the angle option

(Gomes *et al.*, 2013). Five different regions of the membrane surface were measured to determine the average WCA values. The porosity of the membranes was calculated from five replicate measurements using water as the wetting agent. The results obtained in this manner were calculated using Eqs. (1) (Adamczak *et al.*, 2020).

$$\varepsilon = \frac{m_w - m_d}{A.L.p} \times 100 \qquad (1)$$

where ε is the porosity of the obtained membrane [%] and m_w and m_d are the weights of the wet and dried measured membranes [g], respectively. The membrane area was denoted as A [cm^2], the membrane thickness was measured using digital Vernier calipers [cm], and the water density was 0.998 $g.cm^{-3}$.

Physical analysis of the membrane mechanical strength was also performed using a universal testing machine (ASTM 882-91, 1996), namely, yield, tensile strength, Young's modulus, and elongation (Huan *et al.*, 2015).

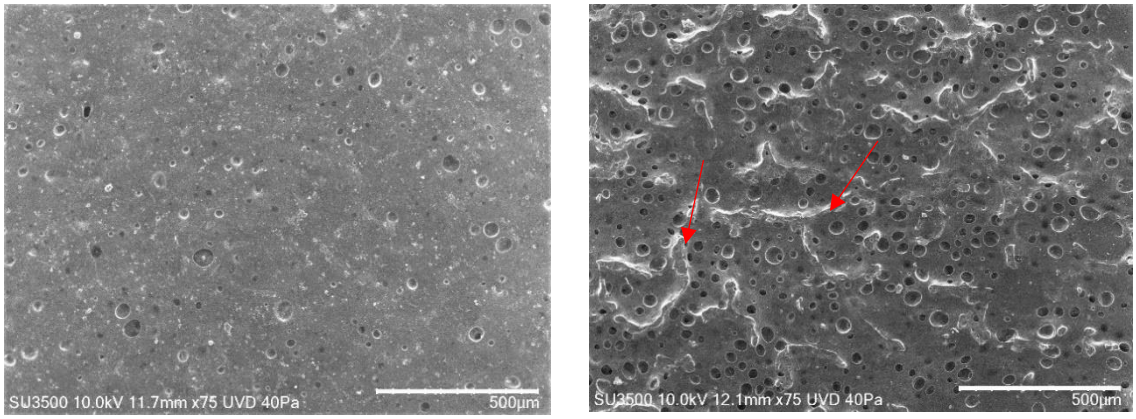
Results and Discussion

In this study, we aimed to provide basic information related to the different effects of the final stages of membrane solidification, namely immersion and evaporation, on the structure and characteristics of polystyrene membranes based on Styrofoam waste. Figure 2 presents images of the PS membranes obtained from both treatments. The results showed that the PS membrane prepared using the immersion technique was white and tight (Figure 2a). Figure 2b shows a more transparent form solidified in air at room temperature. Both membranes produce similar elasticity, which is slightly hard but still elastic.

(**a**) (**b**)

Figure 2. Images of polystyrene (PS) membranes from Styrofoam waste prepared by (a) immersion and (b) evaporation treatment.

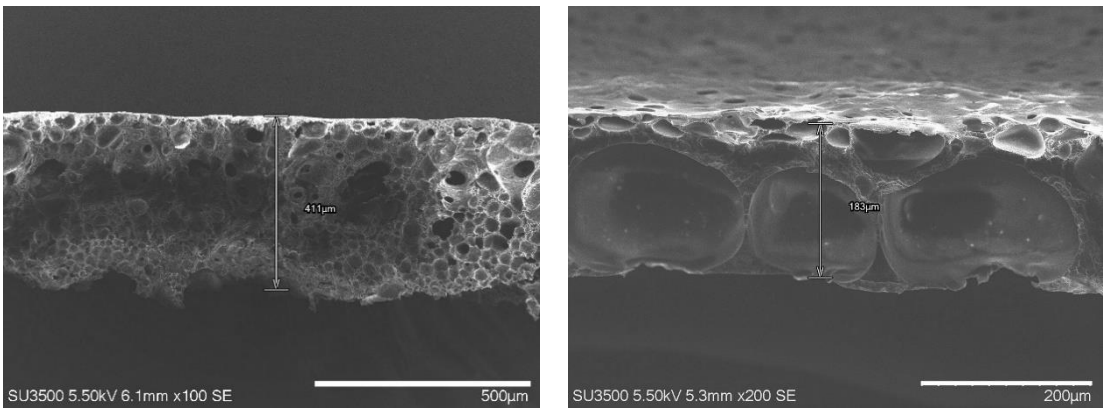
An overview of the microscopic analysis is shown in Figure 3, which provides the top-surface image information of the two solidification treatments. Figure 3a shows the top surface of the PS A membrane, which employs an immersion technique in water (nonsolvent) as a coagulant agent. This resulted in a smooth surface with no visible difference in surface roughness. While Figure 3b shows a distributed surface roughness, such as irregular waves, as indicated by the red arrow. The pore diameter on the surface was also measured manually, which showed an average size of 10–40 μm, whereas employing an evaporation membrane produced pore diameters of 24–46 μm. Moreover, the evaporation treatment resulted in the accumulation of spatial fluctuations on the membrane surface structure, as previously reported by applying an electrospinning technique followed by an evaporation process at the end of membrane solidification (Huan *et al.*, 2015). In addition, the surface of PS A is much denser than that of PS B, which is related to the faster exchange rate between the solvent and water than between the solvent and ambient air.



(a) **(b)**

Figure 3. SEM images of top surface polystyrene (PS) based-membranes using final solidification treatments of (a) immersion and (b) evaporation

Figure 4 shows cross-sectional images of the two membranes. The PS A membrane showed a uniform pore configuration that was sponge-like, whereas the PS B membrane (Figure 4b) showed a dominant pore configuration, such as a macrovoid. This shows that the rate of exchange of solvent and water in the immersion technique is faster than that of solvent and air; thus, this lag leaves various macropore sizes, as described in a previous study (Nurherdiana et al., 2019). The cross-sections of the membranes resulted in pores diameters of 9.25 to 50µm for immerse treatment (Figure 4a) and 49 to 154 Âµm for the evaporation treatment (Figure4b).



(a) **(b)**

Figure 4. The cross-section images of membranes prepared by treatment, (a) immersion and (b) evaporation

The contact angle properties were characterized by deep analysis of the membrane as hydrophilic membrane mats, the results of which are shown in Figure 5. The contact angle measurement was performed with five repetitions of the analysis to validate the results. In addition, a statistical review is presented to determine the significant effect of the membrane solidification process on the characteristics of the resulting membrane. Statistical testing was performed using Analysis of Variance (ANOVA). Several tested categories were the effect of differences in the solidification process, hydrophilicity of the top and bottom surfaces of the membrane in both solidification processes, and effect of differences in the concentration of wt% Styrofoam added to DMF solvent. The

error was 5%, as used in the general performance test.

Figure 5 shows that the PS A membrane has an average contact angle of 79.7 ± 0.489°, while the PS B membrane is 96.8 ± 2.969°. The results showed that the PS A membrane was hydrophilic, while the PS B membrane was hydrophobic. It is shown in the literature that the hydrophilicity values with a contact angle of < 90° are hydrophilic, while > 90° are hydrophobic. The existence of these differences can be caused by the presence of surface roughness, which contributes to the increase in the contact angle, as previously reported (Lalia *et al.*, 2013; Ramos-Olmos *et al.*, 2008; Deka *et al.*, 2019). Increasing the irregularity of the membrane surface can be recommended for regulating the hydrophobicity of the membrane using a simpler method. Macroscopically, the form of hydrophilicity is shown in Figure 5, which is equipped with an angle to measure the contact angle of water on the membrane.

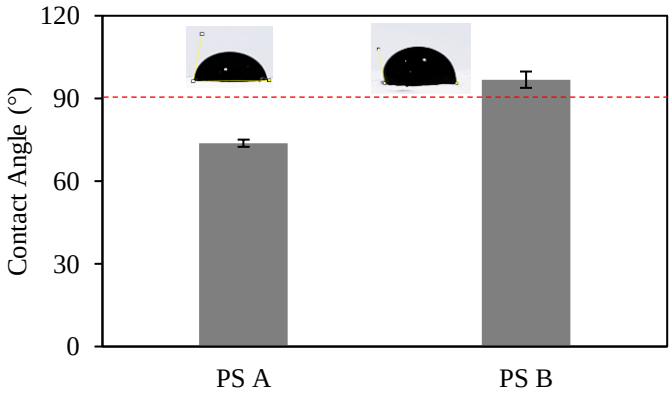


Figure 5. The contact angle value of PS A and PS B membranes.

Figure 6 shows a decrease in the contact angle of the PS A membrane with increasing Styrofoam composition added to the DMF solvent. The decrease in the contact angle values sequentially the average value was 79.7 ± 0.489°, 70.5 ± 0.110°, 69.2 ± 0.370°, 69.8 ± 0.606° and 62.3 ± 0.705° respectively from 18, 22.5, 25, 27.5 and 30 wt% Styrofoam in DMF. This decrease in value is in line with previous research which contact angle values were between 50-60° with a significant decrease in value with increasing Styrofoam weight (Adamczak *et al.*, 2019).

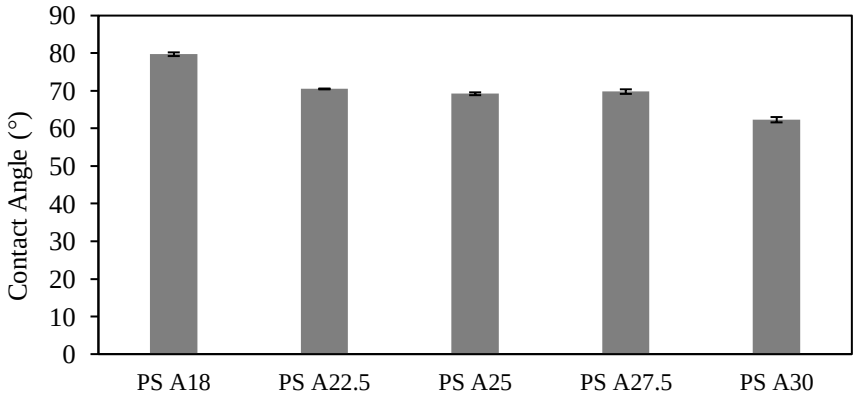


Figure 6. Contact angle value of the membrane PS A with various wt% Styrofoam addition in DMF.

The results of the ANOVA test for the four categories are presented in Table 2 regarding 4 different categories. The results show that the variations in solidification treatment, namely immersion and evaporation at 18 wt% Styrofoam in DMF as solvent, and variations in wt% Styrofoam in the immersion treatment, concluded that there was a significant difference in the resulting contact angle values. This is evidenced by the values of Fcalculated > Fcritical and PValue < 0.05 (error value) so that the initial hypothesis (H0) is rejected (Nurherdiana *et al.*, 2019). However, the two treatments, namely immersion and evaporation, did not show significant differences in the contact angle of the membrane on the top and bottom surfaces, as evidenced by Fcalculated < Fcritical and PValue > 0.05 (error value); thus, the initial hypothesis (H0) was accepted with a 95% confidence level.

Membrane porosity is an initial analysis for identifying active sites based on the surface area, predicting flux, and treating membrane fouling. Porosity measurements were carried out with five repetitions of data collection, and the results are shown in Figure 7. The PS A membrane had a porosity value of 24.85 ± 3.529%, while PS B had a porosity value of 9.84 ± 0.895%. This indicates that the porosity of the PS A membrane is greater in accordance with the regularity of the sponge-like pore configuration, thus indicating a larger pore surface area compared to that of the PS B membrane, which is dominated by macrovoid pores. Figure 8 shows that the percentage of porosity increases with increasing percentage of wt% Styrofoam, which predicts that the pores formed are smaller with high regularity, which also supports the results of a larger contact angle. This indicates that the addition of wt% Styrofoam increases the densification and configuration regularity and decreases the pore size of the membrane, as previously reported (Bakeri *et al.*, 2012; Hubadillah *et al.*, 2018).

Table 2. ANOVA analysis result

Categories	$F_{calculated}$	$F_{critical}$	P_{Value}
The effect of different solidification treatments, namely immersion and evaporation at 18 wt% Styrofoam in DMF.	284.51	3.239	4.4×10^{-14}
Immersion treatment: WCA of the upper and lower membrane surfaces.	0.00121	4.414	0.973
Evaporation treatment: WCA of the upper and lower membrane surfaces.	2.368	4.412	0.141
The effect of the difference in wt% Styrofoam in the solvent on the immersion treatment.	252.17	5.318	2.5×10^{-7}

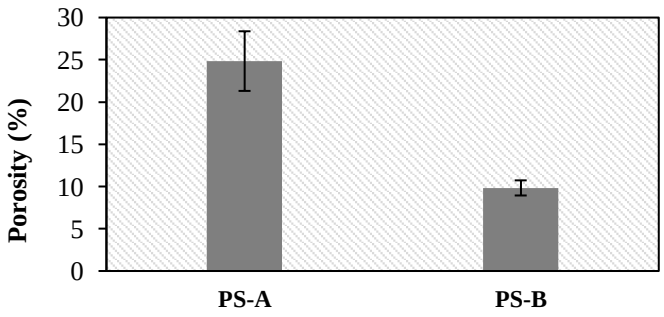


Figure 7. The percentage of porosity of polystyrene (PS) membrane-based Styrofoam waste with immersion (PS A) and evaporation (PS B) treatment.

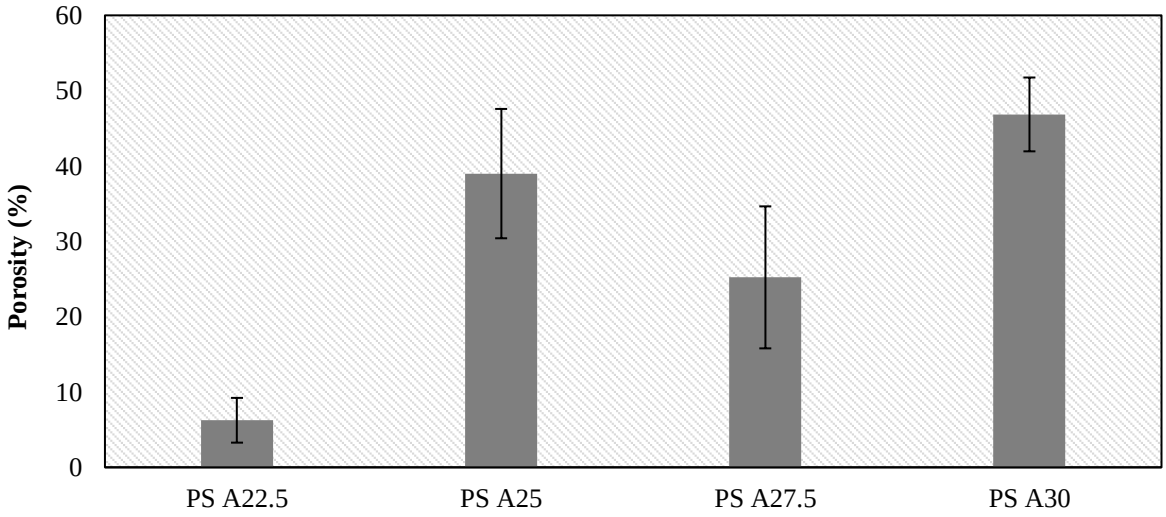


Figure 8. Porosity of the PS A membranes with various Styrofoam contain in DMF (wt%).

Figure 9 shows the results of thermogravimetric analysis, which can provide information on thermal stability and membrane degradation at certain temperatures. It was performed between 25 and 600°C at a heating rate of 5°C.min-1 under air flow at room temperature. It shows A typical high resolution thermogravimetric curve for PS B membrane, there is a decrease in mass around 140-155°C which indicates the evaporation of DMF solvent on the membrane. This appears to be different from membrane A, which does not show a decrease in mass at this temperature or at a water evaporation temperature of approximately 80-100°C. Therefore, the oven dehydration carried out in the methodology was considered effective in evaporating the trapped solution in the PS A membrane, but not yet effective in evaporating the trapped solution on the PS B membrane. Furthermore, there was a significant decrease in mass at temperatures between 380-400°C on both membranes. This indicates that the stability of the membrane prepared from Styrofoam waste can only survive at a temperature of approximately 350°C before being degraded. This shows that the crystallinity and densification of the membrane were the same, even though different solidification methods were employed.

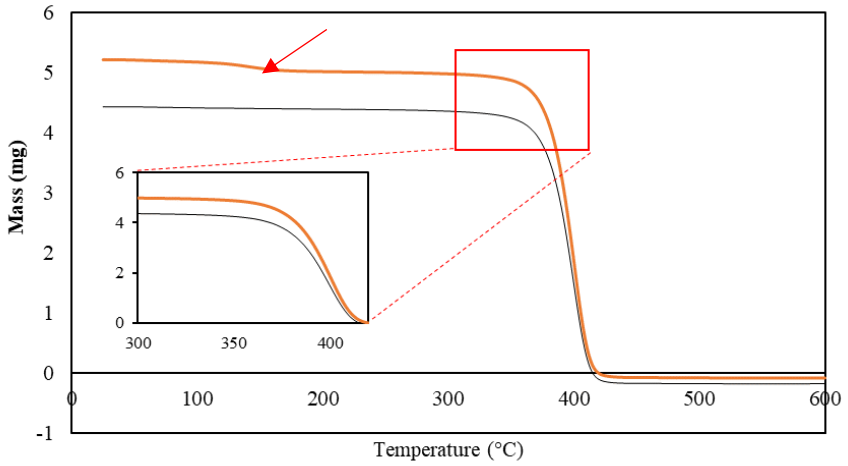


Figure 9. Typical thermogravimetric analysis for membranes: (a) immersion (black) and (b) evaporation (red).

Figure 10 shows the results of functional group analysis using FTIR showing the similarity between the immersion membrane and evaporation membrane, namely that there was an absorption peak in the wave number region of 2922.25 – 2850.88 cm^{-1} which was a stretching vibration, C-H, and an absorption peak in the region of wave number 3082.35 – 3059.20 cm^{-1} from stretching vibration = C-H. In addition, there are also stretching vibrations C=C indicated by absorption peaks in the wave number region of 1600.97 cm^{-1}, 1492.95.10 cm^{-1}, and 1450.52 cm^{-1}, as well as very sharp absorption in the wave number region of 756, 12 and 698.25 cm^{-1} which indicated the presence of a monosubstituted aromatic group. In addition to the absorption peaks mentioned above, there is also a wide absorption peak in the 3485.49 cm^{-1} region, which corresponds to the absorption of the hydrogen-bonded -OH group (Table 3). Based on the above analysis of the FTIR spectra, it can be concluded that the FTIR spectra of the immersed and evaporated membranes are similar to the FTIR spectra of standard polystyrene.

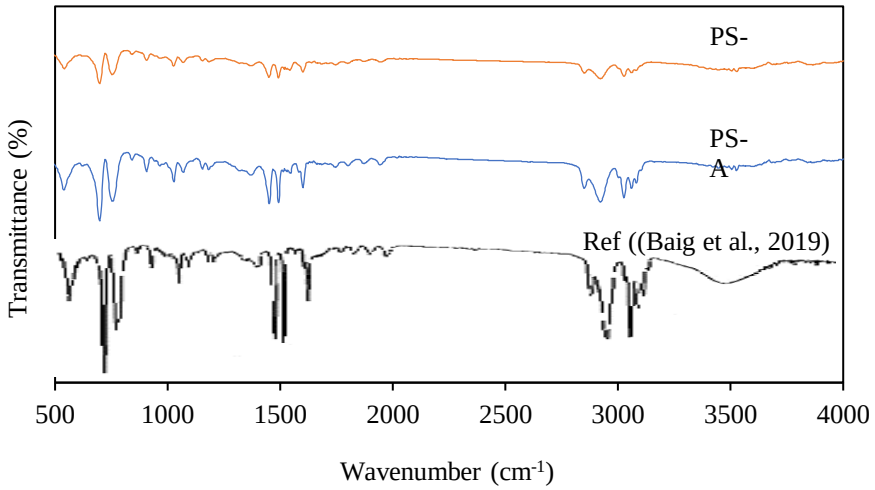
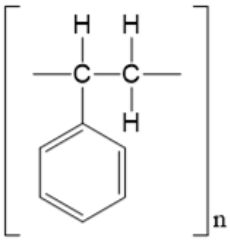


Figure 10. Infrared Spectrum of polystyrene membrane from Styrofoam waste.

Table 3. Infrared absorption band (Baig *et al.*, 2019)

Polystyrene Structure	Functional Group	Wavenumber (cm^{-1})
	–OH	3000–3700
	=CH	3000–3300
	–CH	2800–3000
	C=C	1600–1700
	Substituent compound aromatic	650–900

The physical characteristics, which also provide a crucial part of the membrane, are utilized optimally. Some influential values are the yield strength, tensile strength, Young's modulus, and elongation. The yield strength is the maximum transition value from an elastic polymer to a plastic polymer, and the tensile strength provides information on the maximum tensile force that the polymer can withstand until it breaks. In addition, Young's modulus indicates the magnitude of the force required for the polymer to increase in length per unit area of the sample. The elongation indicates the accuracy of the flexibility of the membrane to be stretched (Yap *et al.*, 2021).

Table 4. Mechanical strength of the PS membrane in various solidification process.

Membrane	Yield Strength (MPa)	Tensile Strength (MPa)	Young's Modulus (MPa)	Elongation (%)
PS A (Immersion in water)	0.42	0.47	37.76	233.59
PS B (Evaporation)	0.48	1.42	47.96	213.24
(Huan *et al.*, 2015) (DMF: THF)	-	0.2 (75:25)	-	-
	-	1.5 (50:50)	-	-
	-	0.4 (25:75)	-	-

Table 4 shows that the yield, tensile strength, and Young's modulus values of the PS A membrane subjected to immersion in water are lower than those of the PS B membranes aerated at room temperature. This tensile strength value is in accordance with previous studies, which showed that the use of solvents and solidification treatments resulted in different membrane strengths. However, the elongation value of PS A was higher than that of PS B, which was confirmed by the physical appearance of the membrane, which was less brittle.

Conclusions

Polystyrene (PS) membrane-based-Styrofoam waste has been successfully synthesized using a phase inversion technique with different final solidification processes, namely immersion in water (non-solvent) and evaporation in air at room temperature. From the discussion above, it can be concluded that the membranes prepared by immersion and evaporation resulted in significantly different hydrophilicity values. This result was also supported by statistical analysis using ANOVA. The immersion membrane had smaller pores than the evaporated membrane, which contributed to the formation of macrovoids in the pore configuration. Thus, the PS A membrane had a higher porosity than the evaporation membrane, and the porosity of the immersion membranes was in the range of 40–50 %, while that of the PS B membrane. Based on the results of the statistical analysis using the ANOVA method, it was found that various solidification treatments and concentrations of wt. % Styrofoam affected the magnitude of the contact angle. The different solidification processes employed contribute to the different tensile strengths and elongations of the membrane. The resulting data can provide a fundamental theory for modifying Styrofoam waste-based membranes.

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Conflict of Interest

The authors have no conflict of interest.

Author Contribution Statements

SDN and BW conceived of the presented idea and design the experiments. MJS and AK carried out the experiments and analysis. RRY and MJJ contributed to the interpretation of the experiment result. HF developed the theoretical framework. All authors contributed to the final manuscript.

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