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Research article

Preparation, characterization, and desalination study of polystyrene membrane integrated with zeolite using the electrospinning method

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ABSTRACT

The novel composite membranes of Styrofoam (polystyrene) integrated with natural zeolite particles were produced using the electrospinning method for desalination purposes. The product was then characterized using FTIR, XRD, and TGA. Subsequently, the effect of different zeolite concentrations, namely 0, 5, 10, 15, 20, and 30 wt.% on its morphological structure and effectiveness was determined. Desalination of artificial seawater was carried out using the gravitational filtration principle to confirm the membrane's capabilities, after which its performance was evaluated using 4 wt.% NaCl solution. The morphological analysis showed that the composite membrane obtained has a smooth surface with microdomains on a few parts, which indicates the presence of zeolite. The result showed that the product integrated with 30 wt.% zeolites had the best performance in the desalination of artificial seawater. This was determined by comparing the conductivity value of the seawater before and after the process. The conductivity led to an 82.63% decrease using 30 wt.% zeolites. The developed polystyrene membrane embedded with zeolite has good thermal stability, hence, it can be used for desalination.

1. Introduction

Water pollution is a serious problem faced by all populations in the world, specifically people who live in big cities. This has led to the development of various water treatment technologies, such as reverse osmosis, thermal vapor, and distillation. However, a suitable method with low energy consumption, environmentally friendly, and cost-effective is needed for long-term treatment [1]. Desalination is a process that can be used to prepare clean water from seawater. It is often carried out with the reverse osmosis (RO) method, which involves the use of a membrane.

A high-performance membrane is often produced by embedding a filler that has a nanoscale [2]. This helps to enhance its functional properties in separating and purifying water [3, 4, 5, 6]. Zeolite is one of the materials that has been extensively used as a filler for various purposes, but not limited to membrane production [7]. It can be modified through mechanical treatment to reduce the particle size into nano. The presence of a unique pore structure on its surface can also inhibit the transport of salt ions due to the sieving effect. Several studies have been carried out on zeolite integrated membrane. For example, high salt

rejection of brackish water using desalination led to a 98% enhancement after it was integrated into the polyamide membrane. Another study revealed that its presence increased hydrophilicity, water flux, and salt rejection. The important aspect that must be considered when preparing a membrane with zeolite as a nanofiller is the filler dispersion quality [8, 9, 10, 11, 12].

Styrofoam is one of the waste products that can easily be obtained and is often used for food and electronic packaging [13, 14, 15]. It is also known as polystyrene, a polymer that consists of styrene as the monomer. Polystyrene has several characteristics, including low cost, non-degr adable, and superior mechanical strength [15, 16], but the hydrophobic properties limit its application as a membrane for water treatment [6, 17, 18, 19, 20, 21]. However, this limitation still offers an advantage, because water molecules can pass through the membrane when the breakthrough pressure is attained. The product obtained is often used a low surface tension solvent and for the filtration of gas.

Several studies reported that the use of zeolite as a nanofiller can enhance the hydrophilicity of polystyrene membranes, and they were suitable for water filtration [22, 23]. There are no studies on the

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Table 1. Composition of polystyrene/zeolite membrane.					
PNZ		Polystyrene – DMF (g)			
(wt. %)	(g)				
5 wt%	0.115 g	2.189 g			
10 wt%	0.226 g	2.031 g			
15 wt%	0.365 g	2.066 g			
20 wt%	0.530 g	2.120 g			
30 wt%	0.927 g	2.163 g			

production of these materials using polystyrene and natural zeolite as well as their effectiveness for desalination. This study aims to use the composite membrane of polystyrene/zeolite to obtain superior desalination performance and mechanical properties. The zeolite particles were prepared from the natural form, which was obtained from the Pahae region, North Tapanuli, North Sumatera, Indonesia. Previous studies explored its potential for separation purposes, including gas and desalination. The presence of nanopores in the zeolite structure inhibits the passage of hydrated salt ions.

Therefore, this study aims to investigate the performance of polystyrene membrane integrated with different zeolite concentrations, namely 5, 10, 15, 20, and 30 wt.% using XRD, FTIR, SEM-EDX, mechanical testing, TGA, and desalination process.

2. Experimental

2.1. Material

Styrofoam (polystyrene) was obtained from a used electronic package, while dimethylformamide, sulphuric acid (98 wt.%), sodium hydroxide, and sodium chloride were purchased from Merck. Furthermore, natural zeolite was collected from Pahae region, North Tapanuli, North Sumatera, Indonesia. All chemicals were used without any further treatment.

2.2. Sample preparation

Pahae Natural Zeolite (PNZ) microparticles were prepared using the milling and sieving processes. The powder obtained was then dispersed in 6 wt.% sulphuric acid and stirred at a speed of 350 rpm. The temperature of the solution was set at 70 °C, and the stirring was carried out for 4 h. Subsequently, the residue obtained was washed until the neutral pH was reached, and the drying process was performed at 105 °C for 1 h.



Figure 2. Diffractogram of polystyrene, zeolite, and the composites.

The membrane was prepared by dissolving 4.7 g of polystyrene in 20mL of 99.8 wt.% dimethylformamide, followed by stirring at 350 rpm. The temperature of the solution was set at 80 °C, and the stirring was carried out for 30 min [13, 24, 25]. After the solution reached room temperature, the zeolite microparticles were added with the compositions presented in Table 1.

The electrospinning device was set, as shown in Figure 1. The polystyrene-DMF solution was placed in a syringe with 0.8 mm needle size. The distance between the needle and collector was 10 cm. The flow rate of the solution was then set at 13 μ L/min along with a voltage of 15 kV. This procedure was then repeated for the mixture of polystyrene and zeolite.

2.3. Characterization

XRD analysis was used to determine the crystalline phase of the samples. Furthermore, the process was carried out using Panalytical Empyrean Model. The analysis of the functional group present in each sample was determined using FTIR - Agillent Technologies-Cary 630 at a range of 650–4000 cm⁻¹ and 16 cm⁻¹ resolution. The thermal stability of



Figure 1. Electrospinning device.



Figure 3. FTIR spectra of polystyrene and polystyrene/PNZ composites.

each sample was evaluated using Thermogravimetric Analysis (TGA) at a range of 30-450 °C, and the heat rate was set at 5 °C/min. The mechanical properties, such as tensile strength and elongation at break were measured using UTM - Tensilon RTF equipped with a 5-kN load cell at speed of 10 mm/min. The morphology of each sample was observed using Scanning Electron Microscopy - Zeiss with 1500x magnification. The instrument was equipped with Energy Dispersive X-Ray (EDX) to evaluate the element composition. The capability of the membrane for desalination was determined by filtrating artificial seawater. The solution was prepared by dissolving 4 g of NaCl in deionized water until the concentration was 4 wt.% [26, 27, 28, 29]. The conductivity of the solution was measured before and after desalination with the prepared membrane using a digital conductivity meter - EZDO 6022. The ion exchange capacity of the product obtained was determined by immersing the membranes in 1 M NaCl solution for 24 h. Immersion was performed in a shaker at room temperature. Subsequently, 3 drops of phenolphthalein were added to the solution, and titrated using 0.01 M NaOH. The ion exchange capacity was calculated by multiplying the concentration and volume of NaOH, followed by division by the weight of the dry membrane. The final unit of ion exchange capacity was mmol/g [30].

3. Result and discussions

Figure 2 shows the diffractogram of zeolite, polystyrene, and polystyrene/PNZ composites.

The XRD results of zeolite, polystyrene, and polystyrene/PNZ composites showed the characteristic peaks of zeolite at 20 10.24, 25.6, and 27.4°. The diffractogram of polystyrene fiber showed the presence of an amorphous pattern. Similar results were also obtained by a previous study at 20 9.23°, 16.85°, and 22.23° [31, 32]. The occurrence of a crystalline pattern was due to the presence of the additive. The type of polystyrene used in this study was the expanded form, which is often produced with the incorporation of different additives. The diffractogram of composites was dominated by zeolite, which indicates that its particles were homogeneously distributed in the polystyrene matrix, as shown in Figure 2. It also indicated that there was no strong specific interaction between the matrix and zeolite. However, additional interaction, such as non-specific interaction can occur between the hydroxyl group on zeolite surface and polystyrene. The increase of diffractogram intensities has a linear correlation with the amount of additive in the composite, which confirmed that it was incorporated into the matrix. The amorphous structure of zeolite

was due to the impact of mechanical treatment, namely ball milling, which enhanced lattice structure disorder [11, 13, 33].

This finding is consistent with the FTIR spectra as shown in Figure 3. The spectra of neat polystyrene and the composites have a similar pattern, and there were no significant changes in them. Generally, the FTIR spectra confirmed the presence of aromatic structure (C=C) in polystyrene at 3026, 1602, and 1446 cm⁻¹ [34].

Good mechanical strength is one of the properties that must be possessed by membranes used for desalination because they have to endure the water pressure for specific periods. The tensile strength and elongation at break of polystyrene and polystyrene/PNZ composites are presented in Figure 4. It was observed that the tensile strength value of the membrane increased linearly with the number of zeolite particles incorporated. Meanwhile, the elongation at the break value decreased linearly with the addition of the particles.

The morphology of zeolite distribution in the fibers can be confirmed using SEM images, as shown in Figure 5. The fiber surface of the polystyrene/PNZ composites showed a microdomain due to the presence of unevenly dispersed zeolite in the matrix. The presence of the microdomain can be seen in almost all SEM images of the composites. Furthermore, their size is proportional to the number of zeolites added to the polystyrene matrix. The increase in size enhanced the surface roughness of fiber, which can be influenced by the aggregation among the particles. This indicates that the size of zeolite aggregate was bigger than the fiber diameter.

The EDX analysis showed that the natural zeolite used in this study has an average Si/Al ratio of 4.6, as shown in Table 2. The percentage of Si and Al in the composite linearly increased along with its addition to the polystyrene matrix. Combining the XRD and EDX data, the zeolite used can be categorized as the mordenite type, which has a high amount of silica. The percentage of Si was higher than Al, which indicated that it has hydrophobic characteristics as well as a high affinity for the hydrocarbon chain. This characteristic can help zeolite to be evenly distributed in the matrix, which was dominated by the hydrocarbon chain, as shown in Figure 5. The Si/Al ratio showed that the mordenite type has a good adsorption capacity. Furthermore, the increment of the ratio has a linear correlation to the adsorption capacity [35].

Figure 6 shows the thermal stability property of polystyrene and polystyrene/PNZ composites. The TG curve of the composites has a similar shape and number of degradation steps to neat polystyrene. The curve confirmed that polystyrene and the composites have one step of



Figure 4. Tensile strength and elongation at break of polystyrene and polystyrene/PNZ composites.



Figure 5. Morphology of (a) polystyrene and polystyrene/PNZ fibers (b) 5 wt.%, (c) 10 wt.%, (d) 15 wt.%, (e) 20 wt.%, and (f) 30 wt.%.

Table 2.	The	elemental	composition	of	polystyrene	and	polystyrene/PNZ	ob-
tained us	ing E	EDX.						

Sample	С	0	Si	Al
Polystyrene	98.17	1.69	-	-
Polystyrene/PNZ	96.77	2.32	0.73	0.18
Polystyrene/PNZ	91.63	3.01	1.05	0.28
Polystyrene/PNZ	94.53	3.92	1.27	0.28
Polystyrene/PNZ	91.51	6.04	2.03	0.42
Polystyrene/PNZ	88.52	7.98	3.07	0.70

degradation. The degradation temperature and weight loss of all samples are presented in Table 3. The early weight loss phenomenon, which occurred at a range of 30–300 °C indicated the evaporation of small molecules, such as water and organic solvent. The loss of weight caused by this process in all samples was between the range of 1–8 wt.%. The maximal temperature for the depolymerization step of polystyrene (Td_{max}) occurred at 342–438 °C, and this step was known as the first degradation step. The composites experienced a weight loss of 23–87% at Td_{max}. Comparing the second degradation step of neat polystyrene and



Figure 6. TGA curve of polystyrene and polystyrene/PNZ composites.

polystyrene/PNZ, the presence of zeolite in the matrix provided more thermal stability to the composites. However, a decrease occurred after its addition at a concentration of 20 and 30 wt.% due to the catalytic effect. Several studies reported the use of zeolite in polymer pyrolysis. These effects were caused by its natural properties, including acidity, pore structure, and size [36].

Desalination using gravitational filtration was used to evaluate the polystyrene/PNZ membrane's performance in reducing the number of salt and ions present in the sample. A digital conductivity meter was used to measure the conductivity of the seawater model sample, namely NaCl 4 wt.%, before and after desalination. Before the process, the initial conductivity was 1474 S cm⁻¹ Table 4 shows the value obtained for each solution after desalination as well as the ion exchange capacity of the membranes.

Table 4 shows data on the electrical conductivity value of artificial seawater, namely NaCl 4 wt.%. The incorporation of zeolite into the polystyrene matrix enhanced ion removal, which increased linearly with the amount added. The highest ion removal was observed after the addition of 30 wt.% zeolites. The electrical conductivity value of artificial seawater reduced up to 86.30% after using 30% polystyrene/PNZ as the membrane. This finding indicates that polystyrene/PNZ has the potential to be used for desalination. The results also showed that zeolite plays an important role in the process, and it is often used as an adsorbent due to the presence of micropores on its surface. Apart from the adsorption mechanism, it can also be assumed that the ion exchange occurred during desalination. However, ion exchange plays a minor role during the process, as shown in Table 4. This is because there was no linear correlation between the percentage of conductivity reduction and ion exchange capacity when it was assumed to be the main mechanism during desalination.

able 3.	Thermal	properties	of po	lystyrene	and	polystyrene/PNZ.	
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Sample	Td _{max} (°C)	Weight loss (%)
Polystyrene	408	87
Polystyrene/PNZ 5%	411	48
Polystyrene/PNZ 10%	424	56
Polystyrene/PNZ 15%	438	52
Polystyrene/PNZ 20%	428	45
Polystyrene/PNZ 30%	342	23

1

Table 4. The electrical conductivities after the desalination process and the ion exchange capacity of polystyrene and polystyrene/PNZ composites.

Sample	Electrical Conductivity (S.cm ⁻¹)	Percentage of reduction (%)	Ion Exchange Capacity (mmol/g)
Polystyrene	848	42.47	1.38
Polystyrene/ PNZ 5%	623	57.73	1.79
Polystyrene/ PNZ 10%	478	67.57	0.87
Polystyrene/ PNZ 15%	314	78.70	0.67
Polystyrene/ PNZ 20%	210	85.75	0.56
Polystyrene/ PNZ 30%	202	86.30	1.17

4. Conclusion

The combination of zeolite and polystyrene produced composites, which have the potential to be used for desalination. The diffractogram and morphology of the products indicated that the zeolite has an amorphous structure and was evenly distributed in the polystyrene matrix. Microdomains were also observed in some parts of the fiber. The tensile strength showed that the composites are more mechanically resistant. The result further showed that composites incorporated with 15 wt.% zeolites had higher thermal stability, while lower values were obtained from others with 30 wt.%. This finding indicates that the zeolite filler acted as a pyrolysis catalyst. The desalination process confirmed that polystyrene/PNZ has the potential to be used as membrane in the future. The electrical conductivity value of artificial seawater linearly decreased with the amount of zeolite in the polystyrene matrix.

Declarations

Author contribution statement

Yuan Alfinsyah Sihombing: Conceived and designed the experiments; Wrote the paper.

M. Zulham Efendi Sinaga, Rangga: Performed the experiments.

Rini Hardiyanti, Susilawati: Contributed reagents, materials, analysis tools or data.

Indah Revita Saragi: Analyzed and interpreted the data; Wrote the paper.

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Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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