

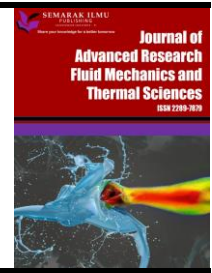


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Evaluation of Polymer Inclusion Membrane Containing Copoly Eugenol Divinyl Benzene 10% as a Carrier for Phenol Transport

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ABSTRACT

Phenol is a toxic and organic compound that often contaminates water, posing significant environmental hazards. To resolve this issue, we conducted research on phenol transport using the Polymer Inclusion Membrane (PIM) method, employing Copoly Eugenol Divinyl Benzene (Co-EDVB) 10% as a carrier compound. The primary objective of this research was to assess the stability and capability of the PIM membrane. Our findings revealed that membrane stability was achieved with a plasticizer concentration of 3.32%. The addition of NaNO_3 salt to the source phase yielded the most promising outcomes compared to other types of salt, facilitating the transport of 85.84% of phenol. When NaNO_3 0.01 M was introduced in both the source and receiving phases, the transport efficiency increased to 89.93% and 86.76%, respectively. In order to evaluate the durability of the PIM membrane during repeated use, transportation experiments were conducted five times. Without washing, the membrane successfully transported phenol in amounts of 72.19%, 69.23%, 62.71%, 55.91%, and 48.16% during each successive run. Conversely, with regular washing, the membrane maintained its transport efficiency, achieving 70.85%, 64.10%, 56.72%, 49.26%, and 27.19% for each cycle. Notably, the membrane's lifetime reached only 29 days without the addition of 0.1 M NaNO_3 salt. However, with the inclusion of NaNO_3 0.1 M salt, its longer extended to 58 days.

1. Introduction

The advancement of the industry has given rise to various new challenges, including environmental pollution, such as industrial waste generating hazardous pollutants like phenol [1,2]. Phenol stands as a primary water pollutant prevalent in diverse industrial sectors such as petroleum, pharmaceuticals, paints, electronics, and even hospitals [3]. Therefore, further treatment of liquid waste containing phenol and its derivatives is imperative before disposal into water bodies. One effective technology for this purpose is Liquid Membrane Technology [4].

Liquid membrane separation technology is increasingly being applied [5,6]. Various studies in this field have evolved, ranging from selectivity transport and capabilities to mathematical modeling for

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transport. Some researchers have recently published their membrane-related research in international journals [7,8]. Polymer Inclusion Membrane (PIM) is one such liquid membrane method [5]. The PIM method is considered to enhance membrane stability compared to Supported Liquid Membrane (SLM) due to the presence of a base polymer (supporting polymer) and a plasticizer [2,9]. The base polymer, such as polyvinyl chloride (PVC), is expected to address carrier compound leakage. The carrier compound is a component in the membrane that facilitates the separation process and aids in transporting the target compound through the membrane. The carrier compound reacts with the targeted components in the source phase, moves across the membrane, and releases these components in the receiving phase. In addition to the supporting polymer and carrier compound, another component in PIM membrane formation is the plasticizer. The plasticizer used, such as dibenzyl ether (DBE), functions to make the membrane system more elastic [10,11].

Carrier compounds based on natural compounds, such as polyeugenol derived from eugenol polymerization, have been explored. Eugenol is a chemical component found in clove leaf oil. However, polyeugenol has not been effectively utilized as a carrier compound due to its low interaction capability with target compounds, possibly attributed to its low molecular weight. Polymers with low molecular weights may have limited active sites [12]. Increasing the molecular weight of a polymer can be achieved through polymerization using vinyl compounds. The majority of vinyl compound polymerization reactions are addition polymerizations. Addition polymerization occurs mainly at the chain ends with double bonds, resulting in a high molecular weight. Co-EDVB is the product of polymerization or cross-linking between eugenol and divinyl benzene (DVB). The double bonds in this diene compound exhibit reactivity, allowing synthesis processes to occur at room temperature [8].

Kiswandono *et al.*, [13] has conducted a similar investigation, evaluating membranes using the PIM method with a Co-EDVB 4% as a carrier compound, resulting in a 60% transportation of phenol with an optimum plasticizer weight of 0.3100 gr. Based on the theory that increasing the molecular weight enhances active sites, makes phenol transport more optimal, this research evaluates the stability and capability of the membrane using the PIM method with Co-EDVB 10% as a carrier compound. It is anticipated that higher carrier compound concentrations will yield more optimal stability and membrane performance. Parameters tested in this study include the effects of plasticizer concentration, types of salt, salt concentration in the source and receiving phases, repeated use tests, and lifetime tests.

2. Methodology

2.1 Phenol Transport Equipment and Materials

The instruments utilized in this research include a Fourier Transform Infrared (FTIR) spectrophotometer (Perkin Elmer 99951, USA), a pH meter (Metrohm 827, Swiss), a UV-Vis spectrophotometer (Hitachi U-2010, Japan), and a Scanning Electron Microscope (SEM) (JSM 6360LA, Japan). Additionally, other tools employed comprise a magnetic stirrer, magnetic bar, analytical digital balance (Mettler Toledo AB54-S, USA), dropper pipettes, spatula, reaction tubes, separating funnel, stand and clamp, a set of phenol transport apparatus (chamber with a diameter of 3.5 cm, thickness gauge (Mitutoyo 7301, Japan), measuring glasses, chemical glasses, stirring rods, spatulas, round-bottom flasks, and measuring flasks. The PIM membrane is installed between two transport pipes to facilitate transportation. One pipe contains 50 mL of NaOH solution as the receiving phase, and the other contains 50 mL of 60 ppm phenol as the source phase. Subsequently, the phenol concentrations in the source and receiving phases are analyzed using a UV-Vis spectrophotometer at a maximum wavelength of 456 nm with the 4-aminoantipyrine method [14].

The procedure is as follows: 5 mL of the sample in the source phase, receiving phase, and standard phenol with various concentrations are added to 5 mL of deionized water, resulting in a final volume of 10 mL. The solution's pH in the source phase and standard phenol is adjusted to 10 ± 0.2 using NH_4OH 1 M and phosphate buffer, while the pH for the receiving phase is adjusted with HCl 0.5 M, and then 1 mL of 4-aminoantipyrine 2% and potassium ferricyanide 8% are added. The solution is allowed to stand until it changes color to pink. After the color change, the solution is transferred to a separating funnel and supplemented with 5 mL of chloroform. The separating funnel is shaken and left to stand for a few minutes until separation occurs, after which the organic layer or chloroform layer (bottom) is separated. The chloroform extract obtained is measured for absorbance using a UV-Vis spectrophotometer at a wavelength (λ) of 456 nm. The concentration of phenol in the source and receiving phases is obtained from the calibration curve. Each measurement is repeated three times, and the %RSD data values are observed [14].

2.2 PIM Membrane Fabrication

The composition of Co-EDVB 10% as the carrier compound, polyvinyl chloride (PVC) as the base polymer, and Dibenzyl Ether (DBE) as the plasticizer is in a ratio of 10%:32%:58%. 10 mL of Tetrahydrofuran (THF) were added as a solvent to the components of the PIM membrane. The dissolution process occurred over 30 minutes, followed by a 3-day period of standing to allow for the natural evaporation of the solvent [12]. The composition of the PIM membrane is detailed in Table 1.

Table 1
Composition of the membrane [12]

No.	Polyvinyl chloride (PVC) (g)	Co-EDVB 10% (g)	Dibenzyl Ether DBE (g)
1	0.1728	0.0540	0.3032
2	0.1728	0.0540	0.3100
3	0.1728	0.0540	0.3132
4	0.1728	0.0540	0.3200
5	0.1728	0.0540	0.3232

2.3 Effect of Plasticizer Concentration

PIM membranes were prepared with varying concentrations of the plasticizer (Dibenzyl Ether). The employed plasticizer concentrations were 3.21%, 3.28%, 3.32%, 3.39%, and 3.42% (w/w), with sequentially added masses of DBE being 0.3032 g, 0.3100 g, 0.3132 g, 0.3200 g, and 0.3232 g [12]. After weighing the membranes, they were placed on the phenol transport pipe. Subsequently, in the source phase column, 50 mL of 60 ppm phenol with a solution pH of 5.5 was filled, and in the receiving phase, 50 mL of NaOH 0.1 M was added. The transport pipe was sealed and stirred using a magnetic stirrer for 48 hours at room temperature. After 48 hours, the PIM membrane was removed, air-dried for 48 hours, and then reweighed. The phenol concentration in the receiving and source phases was analyzed using a UV-Vis spectrophotometer at a wavelength of 456 nm.

2.4 Effect of Salt Type

PIM membranes with optimum plasticizer composition from the previous procedure were initially weighed. Subsequently, these membranes were placed on the phenol transport pipe. In each source phase column (5 chambers), a mixture of 50 mL of phenol (60 ppm, pH 5.5) with salt 0.01M was filled.

The salts used were KCl, NaCl, NaNO₃, Na₂SO₄, and KNO₃. Meanwhile, 50 mL of 0.1 M NaOH was introduced into the receiving phase. The transport pipe was sealed and stirred using a magnetic stirrer for 48 hours at room temperature. After 48 hours, the PIM membrane was removed, air-dried for 48 hours and reweighed. The phenol concentration in the receiving and source phases was analyzed using a UV-Vis spectrophotometer at a wavelength of 456 nm.

2.5 Effect of Salt Concentration in the Source and Receiving Phases

PIM membranes with optimum salt type compositions were initially weighed. Subsequently, these membranes were placed on the phenol transport pipe. In the source phase column, 50 mL of 60 ppm phenol with a solution pH of 5.5, supplemented with optimal salt types at concentrations of 0; 0.001; 0.010; 0.100; and 0.500 M, was filled. Simultaneously, in the receiving phase column, 50 mL of NaOH 0.1 M was added. The transport pipe was sealed and stirred with a magnetic stirrer in the source and receiving phases for 48 hours at room temperature. After 48 hours, the PIM membrane was removed, air-dried for 48 hours and reweighed. The phenol concentration in the receiving and source phases was analyzed using a UV-Vis spectrophotometer at a wavelength of 456 nm. The same procedure was followed for salt concentration variations in the receiving phase. Salt concentrations of 0 M (control); 0.001; 0.010; 0.100; and 0.500 M in the receiving phase. After stirring, samples were taken from the source and receiving phases, and the phenol concentration was analyzed using a UV-Vis spectrophotometer at a wavelength of 456 nm.

2.6 Repeated Use of PIM Membrane

In the repeated use test, two variations were conducted: repeated use without washing and with washing. PIM membranes with optimum plasticizer composition were initially weighed. Subsequently, these membranes were placed on the phenol transport pipe. In the source phase column, 50 mL of 60 ppm phenol with an optimum pH of 5.5 was filled, and in the receiving phase column, 50 mL of 0.1 M NaOH was added. The transport pipe was sealed and stirred with a magnetic stirrer in the source and receiving phases for 48 hours at room temperature. After 48 hours, the PIM membrane was removed, air-dried for 48 hours, and then reweighed. The phenol concentration in the receiving and source phases was analyzed using a UV-Vis spectrophotometer at a wavelength of 456 nm. Subsequently, the PIM membrane was reused for phenol transport with five repetitions using a new phenol solution. Furthermore, the PIM membrane was washed with deionized water for 30 minutes, and then it was reused for phenol transport with five repetitions using a new phenol solution.

2.7 Lifetime Testing

Lifetime testing aimed to determine the endurance of PIM membranes with or without the addition of salt [15]. PIM membranes with optimum plasticizer composition were placed in the center of the phenol transport pipe. Subsequently, in the source phase column, 50 mL of phenol with a concentration of 60 ppm and a pH of 5.5 was filled, with five variations

- (a) Without salt
- (b) Using NaNO₃ 0.1 M
- (c) Using NaCl 0.1 M
- (d) Using Na₂SO₄ 0.1 M
- (e) Using KNO₃ 0.1 M

In the receiving phase column, 50 mL of 0.1 M NaOH was added. The transport pipe was sealed and stirred with a magnetic stirrer in the source and receiving phases. The lifetime was determined by measuring the pH value in the source phase. An increase in the pH value in the source phase indicated membrane leakage. The pH in the source phase was periodically checked until the pH in the source phase reached approximately 9.0 [13].

3. Results

3.1 PIM Membrane

The produced PIM membrane is ready for phenol transport. The PIM membrane is a thin and flexible film with an average thickness of 0.31 mm. This membrane comprises a carrier compound, plasticizer, and supporting polymer [10,14,16].

3.2 Effect of Plasticizer Concentration

In addition to increasing membrane plasticizer, it can also enhance the transport capability of phenol. However, excessive concentrations of plasticizer may diminish the membrane's ability to transport phenol. Various concentrations of plasticizer were added during the fabrication of PIM, namely 3.21%, 3.28%, 3.32%, 3.39%, and 3.42%. Membranes with different plasticizer concentrations were used for phenol transport. Successively, the transported phenol concentrations in the receiving phase were 60.63%, 74.99%, 83.91%, 81.13%, and 63.43%. The optimum condition for the transported phenol concentration in the receiving phase occurred at a plasticizer concentration of 3.32%, with a percentage of transported phenol in the receiving phase reaching 83.91%. The addition of plasticizer to the membrane enhances transport speed and is likely to increase the diffusion of species through the membrane, influencing the stability of the membrane that functions to form or compact the membrane [12].

This addition, besides increasing membrane elasticity, can also act as an inhibitor for phenol to be transported to the receiving phase if the amount of plasticizer exceeds the required limit. However, the greater the addition of plasticizer, the more the transported phenol tends to decrease. This phenomenon is due to the migration or leakage of the plasticizer into the membrane/interface, creating an additional barrier of plasticizer [1,2]. The addition of plasticizer at concentrations of 3.21%, 3.28%, 3.39%, and 3.42% results in the partial loss of membrane components. The low percentage of ML Loss is attributed to the minimal leaching of components into the receiving phase, thereby minimizing the loss of membrane components. This is evidenced by the low percentage of phenol transported to the receiving phase [12]. The highest ML Loss percentage is observed with the addition of 3.21% concentration of plasticizer, amounting to 19.52%.

The influence of various plasticizer concentration variations on phenol transport and % ML Loss is illustrated in Figure 1.

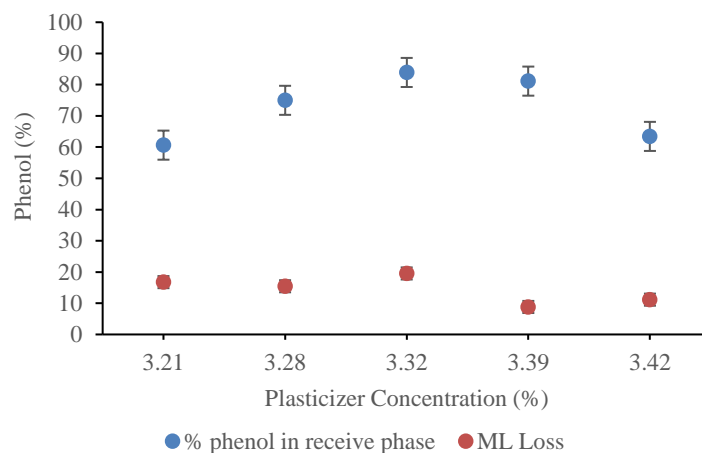


Fig. 1. Graphs illustrating the effect of plasticizer concentration on % phenol in the receiving phase and ML loss variations

3.3 Effect of Salt Types

The addition of various types of salts in the source phase can reduce the loss of membrane components, thereby enhancing membrane stability compared to the absence of salt addition [9]. The optimal result was obtained with the addition of NaNO_3 salt, where the transported phenol concentration reached 85.84%. NaNO_3 , with a lower quantity of cations compared to other salt types, may have limitations in binding to the active sites ($-\text{OH}$) in the carrier compound (Co-EDVB 10%). The low quantity of Na^+ ions is likely to restrict their interaction with the active sites. The addition of different salt variations also influences ML Loss, as depicted in Figure 2.

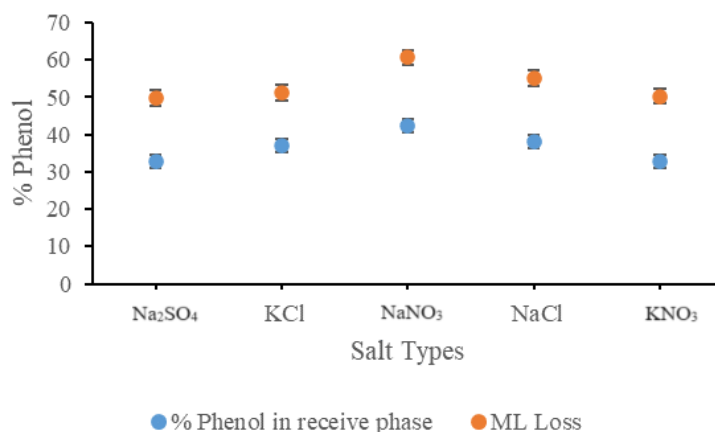


Fig. 2. Graphs illustrating the effect of salt types on % phenol in the receiving phase and ML loss graph for salt type variations

3.4 Effect of NaNO_3 Salt Concentration in the Source Phase

The optimal salt concentration in the source phase is 0.01 M, resulting in a transported phenol concentration in the receiving phase of 89.93%. The presence of NaNO_3 salt in the source phase disrupts the formation of hydrogen bonds between phenol and the carrier compound Co-EDVB 10%. The increasing concentration of cations from the salt in the phenol solution corresponds to a higher quantity of cations in that phenol solution. A higher concentration of salt ions in the source phase

leads to a smaller percentage of transport results because an increased number of ions hinders the phenol transport process. As the NaNO_3 concentration increases, there is a reduction in active groups in Co-EDVB 10%, resulting in a decline in phenol transport percentage [13,17]. The impact of various concentrations of salt in the source phase on phenol transport and % ML Loss is illustrated in Figure 3.

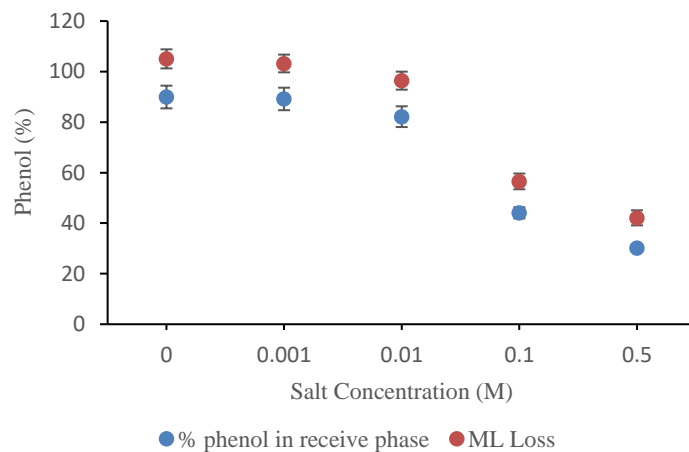


Fig. 3. Graphs illustrating the Effect of salt concentration in the source phase on the percentage of phenol in the source phase and the ML Loss graph for variations in salt concentration in the source phase

3.5 Effect of NaNO_3 Salt Concentration in the Receiving Phase

The optimal salt concentration in the receiving phase is 0.01 M, with the transported phenol concentration in the receiving phase being 86.76%. The addition of NaNO_3 concentration in the receiving phase results in a lower percentage of phenol compared to when NaNO_3 is added in the source phase. The addition of various concentrations of NaNO_3 in the receiving phase will increasingly hinder phenol from being transported from the source phase to the receiving phase. An increase in NaNO_3 concentration will decrease the percentage of phenol transported to the receiving phase because the Na^+ ions from NaNO_3 can reduce the solubility of NaOH . The Na^+ ions, which function to convert phenol to phenolate, will decrease, leading to a decrease in the percentage of phenol transported as the NaNO_3 concentration increases [12].

The addition of NaNO_3 salt in the receiving phase also causes % ML Loss to decrease with increasing NaNO_3 concentration added to the receiving phase. The influence of various concentrations of salt in the receiving phase on phenol transport and % ML Loss is shown in Figure 4.

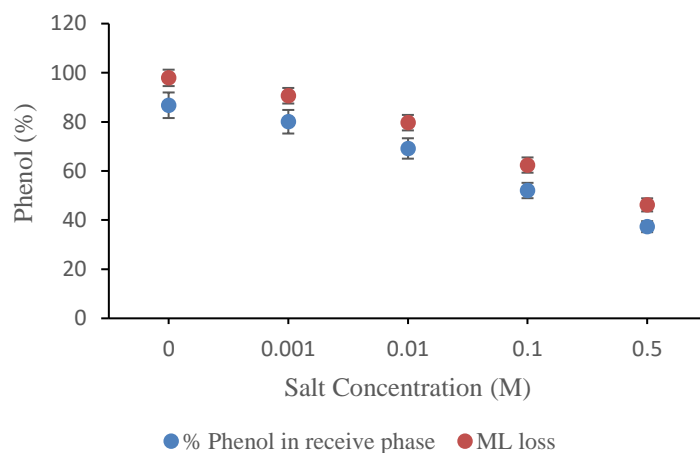


Fig. 4. Graphs illustrating the Effect of salt concentration in the receiving phase on the percentage of phenol in the receiving phase and the ML Loss graph for variations in salt concentration in the receiving phase

3.6 Repeated Use of PIM Membrane

The highest repeated use is the first repetition, both in variations without washing and with washing, with sequential percentages of transported phenol of 72.19% and 70.85%. The influence of repeated use of PIM membrane on the concentration of transported phenol and % ML Loss can be illustrated through Figure 5 and Figure 6.

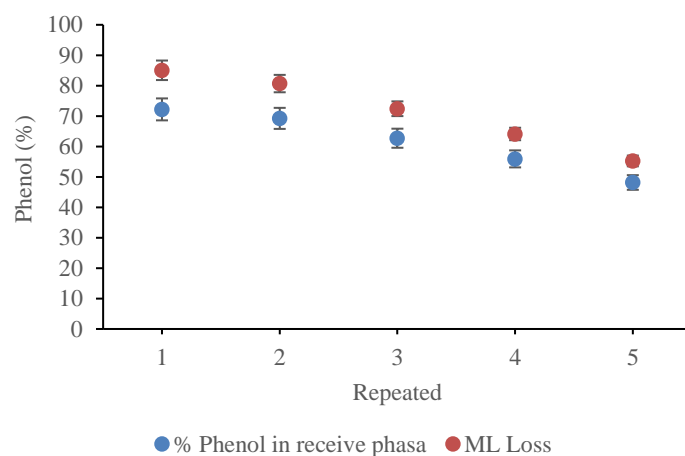


Fig. 5. Graph illustrating the effect of repeated use without washing of the PIM membrane on the percentage of phenol and ML Loss in the receiving phase

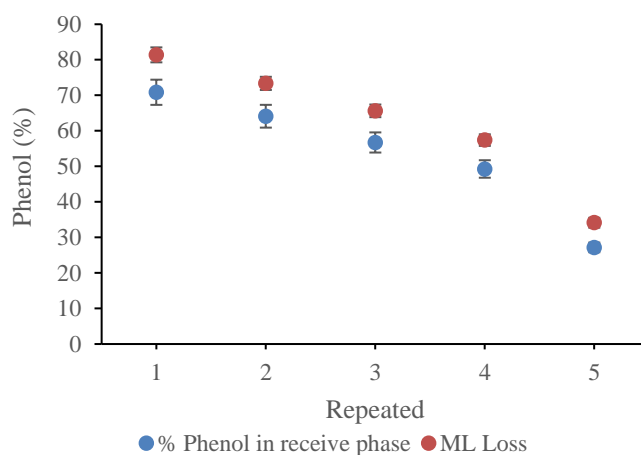


Fig. 6. Graph illustrating the effect of repeated use with washing of the PIM membrane on the percentage of phenol and ML Loss in the receiving phase

During the second, third, fourth, and fifth repeated use, the active groups on the membrane decrease. The decrease in the percentage of transported phenol is caused by the presence of leaching components. Leaching can originate from the carrier compound (Co-EDVB 10%), base polymer (PVC), and plasticizer (DBE). This is supported by the difference in the weight of the membrane before and after being used for transport, known as liquid membrane (LM) loss [1]. Successive large % ML Loss values are obtained in the first use, both without washing and with washing, amounting to 12.85% and 10.52%.

3.7 Lifetime

Lifetime with the addition of NaNO_3 salt has a longer duration, lasting for 58 days, compared to the lifetime of PIM membrane without the addition of salt or with the addition of other types of salt, as indicated in Table 2. A membrane is considered stable if it has a long lifespan and is considered to have experienced leakage if the pH in the source phase is approximately [13]. The graph depicting the relationship between the pH of the source phase and the membrane's lifespan is shown in Figure 7.

Table 2
 The lifetime of the PIM membrane

Days	pH				
	Without Salt	Na ₂ SO ₄	KNO ₃	NaCl	NaNO ₃
1	5.52	5.51	5.53	5.52	5.55
5	5.93	5.94	5.83	5.75	5.73
9	6.38	6.31	6.03	6.35	6.33
13	6.86	6.75	6.29	6.55	6.46
17	7.27	7.18	6.44	6.77	6.71
21	7.81	7.46	6.86	6.95	6.92
25	8.33	7.79	7.16	7.23	7.18
29	9.10	8.14	7.47	7.56	7.45
33		8.71	7.74	7.77	7.67
37		9.11	8.02	7.97	7.85
41			8.84	8.27	8.08
45			9.21	8.75	8.23
49				9.23	8.41
53					8.62
57					8.83
58					9.08

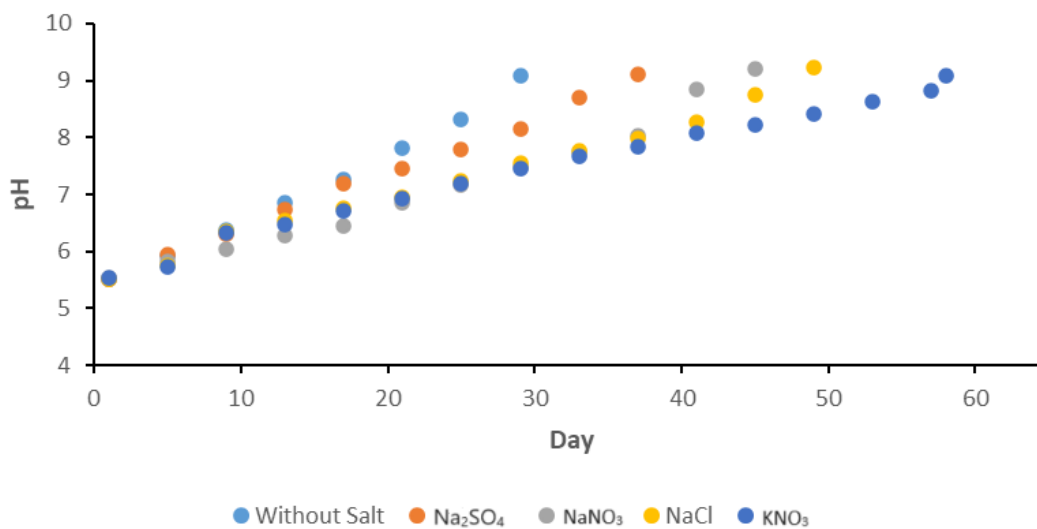


Fig. 7. Graph of pH measurement throughout the lifetime

3.8 Characterization of PIM Membranes

On the membrane before transport at a magnification of 1000x, the membrane surface is still covered by the plasticizer as its liquid medium [6]. The use of membranes for transport allows the components of the membrane to leach. This leaching of membrane components will result in the surface of the PIM membrane becoming porous. The leaching components can originate from the carrier compound, base polymer, or plasticizer. This is supported by the weight difference of the membrane before and after transport through weighing [1]. Characterization of PIM membranes before and after transport was analyzed using SEM and FTIR. The results of the SEM characterization can be seen in Figure 8.

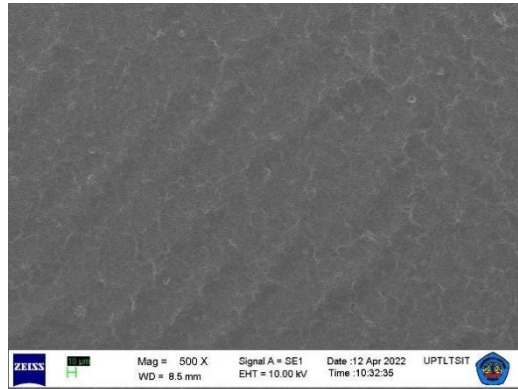


Fig. 8. Surface morphology of the PIM membrane before transport

This weight difference in the membrane is referred to as ML Loss. In the surface morphology of the PIM membrane after transport, at a magnification of 1000x. The surface morphology of the PIM membrane after transport (without the addition of salt) is shown in Figure 9 and the surface morphology of the PIM membrane after transport (with addition of NaNO_3 salt) is shown in Figure 10.

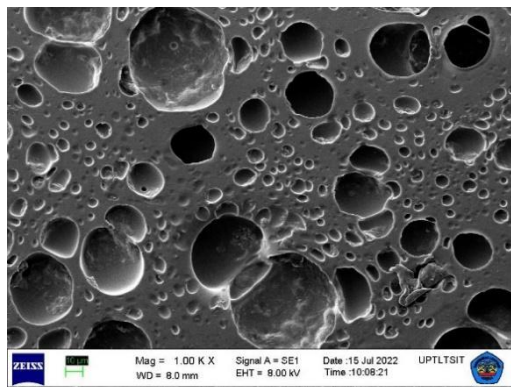


Fig. 9. Surface morphology of the PIM membrane after transport (without the addition of salt)

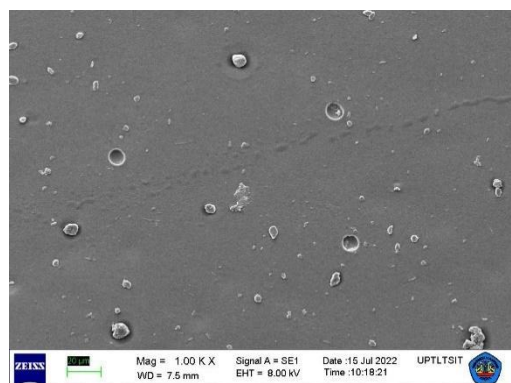


Fig. 10. Surface morphology of the PIM membrane after transport (with the addition of NaNO_3 0.1M)

On the surface of the membrane after transport with the addition of NaNO_3 in the source phase, pores are visible. However, the pores are not as large as those seen on the membrane surface after transport without the addition of salt. This is because salt can increase the stability of the membrane, making it more stable and less prone to leakage [17]. During the membrane characterization process, the influence of parameters such as mixed convection and the presence of nanoparticles can also affect the stability and morphology of the membrane. As shown in the study by Bakar *et al.*, [18], the addition of nanoparticles such as Cu, Al_2O_3 , and TiO_2 in a porous medium exhibits varying velocity distribution patterns, which can influence the membrane's surface properties, including stability and observed leakage, this method is suitable for wastewater treatment [19]. The FT-IR characterization was carried out on the PIM membrane before and after phenol transport under optimum conditions, as shown in Figure 11.

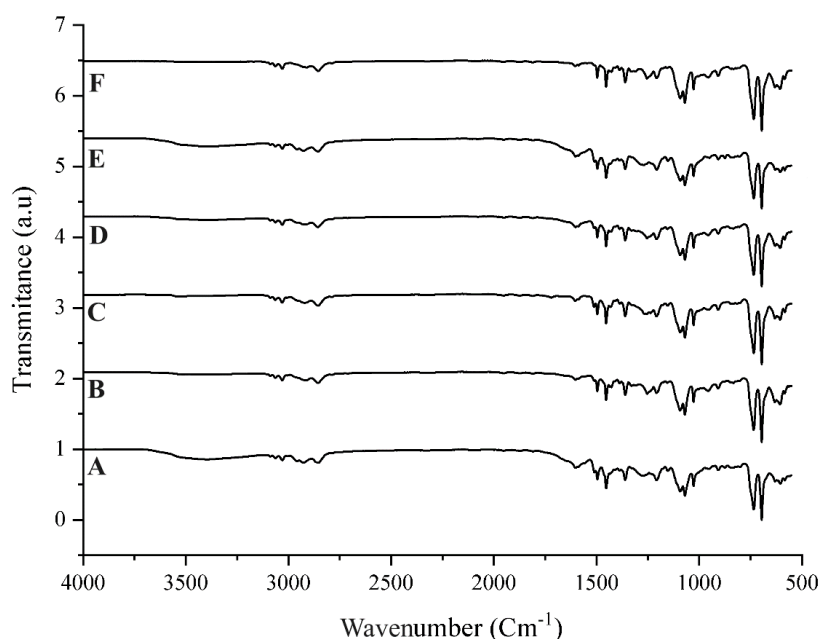


Fig. 11. Comparison of IR spectra (A) after transport with the addition of KNO_3 , (B) after transport with the addition of Na_2SO_4 , (C) after transport with NaCl , (D) after transport without the addition of salt, (E) after transport with the addition of NaNO_3 , and (F) before transport

The characterization results indicate that the PIM membrane before and after transport relatively did not experience a wavelength shift. Specific absorbance in Figure 10 identifies that the PIM membrane after transport still has the same functional groups as the PIM membrane before transport. Therefore, characterization can be conducted by observing changes in intensity in the obtained FT-IR spectrum. The decrease in intensity in the FT-IR results before and after transport indicates the loss or leaching of active sites from the membrane to the receiving phase. A comparison of the two FT-IR spectra of the membrane before and after transport shows a reduction in the peak of the $-\text{OH}$ stretching functional group in the PIM membrane after transport [12].

4. Conclusions

Based on the discussion of the research results, it can be concluded that the stability of the PIM membrane is achieved with the addition of a plasticizer concentration of 3.32%, and the addition of NaNO₃ 0.01M in the source and receiving phases makes the PIM membrane more stable by reducing the percentage of ML Loss. The optimum ability of the PIM membrane is achieved in the first use without washing, and the membrane has a lifespan of 58 days with the addition of NaNO₃ in the source phase. FTIR characterization shows a difference in intensity between the membrane before and after transport, indicating the leaching of membrane components during the transport process. SEM characterization reveals pores on the membrane surface after transport, indicating interaction between the –OH groups of phenol and the active sites of the carrier compound. This proves that an increased concentration of the carrier compound enhances the stability and performance of the PIM membrane, optimizing the transport of phenol.

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