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THE EFFECT OF COMPARATIVE CONCENTRATIONS OF POLY-BADGE AS A CARRIER ON THE POLYMER INCLUSION MEMBRANE METHOD OF PHENOL TRANSPORT

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ABSTRACT

Since it may rehabilitate phenol without creating secondary waste, the liquid membrane Polymer Inclusion Membrane (PIM) technique has recently been the focus of several investigations. This study employed the PIM approach to phenol transport utilizing the carrier Poly-Bisphenol A Diglycidyl Ether (Poly-BADGE) at 1:1 and 3:1 polyeugenol to BADGE ratios. Numerous phenol transport parameters were investigated, including the impact of pH on the phenol source phase, the concentration on the sodium hydroxide receiving phase, membrane thickness, and the time of the phenol transport process using the PIM technology. Dissolving the carrier, polyvinyl chloride, and dibenzyl ether in tetrahydrofuran solvent produced an elastic and porous PIM. UV-Vis Spectrophotometer was then used, along with the addition of a 4-amino antipyrine reagent, to ascertain the phenol content following transport. The wavelength at which absorbance was measured was 460 nm. The findings from the research suggest that, given optimal conditions such as a source phase 5.5 pH, a receiving phase concentration of 0.10 M sodium hydroxide, a membrane thickness of T_{54} type PIM membrane, and a transport time of 24 hours phenol can be effectively transported using PIM with Poly-BADGE carriers at ratios of 1:1 and 3:1, achieving transport efficiencies of 57.31% and 85.30%, respectively. The BADGE was examined using Fourier Transform Infrared (FTIR) and Scanning Electron Microscope (SEM) before and after transport.

Keywords: Membrane; Phenol; PIM; Poly-BADGE; Wastewater.

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INTRODUCTION

Wastewater from various industries, such as the chemical, pharmaceutical, paper, plastic, pesticide, and petrochemical sectors, frequently contains organic contaminants like phenol and its byproducts.[1] Phenol has a negative influence on both the environment and human health as one of the primary contaminants found in wastewater.[2] Phenol's potential to result in low blood pressure, liver damage, acute poisoning, and possibly death indicates how dangerous it is.[3,4] Maintaining environmental sustainability requires efficient treatment of these contaminants. Liquid membrane technology has emerged as a key area of study recently in an attempt to remove organic contaminants from wastewater without producing secondary waste. This approach offers great selectivity and efficiency in separating organic contaminants, as demonstrated by the study conducted by Kiswandono *et al.*[5] One of the best techniques for separation is liquid membrane technology as it operates without pressure. The fundamental idea of separation based on the variation in concentration between the source and receiving phases has created a lot of opportunities to address the wastewater purification issue.[6] Polymer Inclusion Membrane (PIM) is an intriguing technique in liquid membrane technology. It was noted by Keskin *et al.*[7] as a promising technique. Depending on the membrane composition, PIM membranes provide a higher selectivity and a bigger interfacial surface. Because of these benefits, the PIM approach has been effectively utilized to separate specific chemical substances while still being simple to apply.[8,9] The PIM technique is valued for its capacity to improve membrane stability, particularly because of the supporting polymer that prevents carrier leakage.[10-14] In PIM techniques to

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separate target compounds, carriers are crucial components.[15-19] Additional support, such as plasticizers and supporting polymers, is needed for these membrane components.[20-23] Eugenol was selected as the primary ingredient in the carrier synthesis in this study. The simplicity of phenol separation is demonstrated by the use of eugenol as a carrier. Nevertheless, there are still issues with the way eugenol interacts with target molecules when it is polymerized in direct synthesis using an acid catalyst. Consequently, polyeugenol's molecular weight may be raised to enhance the compound's carrying capacity by cross-linking it with vinyl compounds.[24,25] Large molecular weights are the outcome of addition polymerization, which is used in most vinyl compound polymerization processes. Several other researchers use vinyl compounds as poly eugenol cross-linking agents, including divinyl benzene (DVB), ethylene glycol dimethacrylate (EGDMA), and diallyl phthalate (DAF). Another option is to use the hydroxy groups in polyeugenol to polymerize it. The potential of the carrier to interact with the target ingredient is increased when epoxide compounds, such as Bisphenol-A-Diglycidyl Ether (BADGE), are used as a cross-linking agent. This results in BADGE polyeugenol molecules with a greater molecular weight.[25] Copoly(eugenol-diallyl phthalate) (Co-EDVB), which was employed by Kiswandono *et al.*[4] to overcome phenol pollutant contamination in wastewater utilizing the PIM technique, is a carrier that has been used in several prior research. Hence, this study utilized the PIM method to explore the phenol transport using carriers of Poly-Bisphenol A Diglycidyl Ether (Poly-BADGE) at ratios of 1:1 and 3:1. Various factors were investigated, including membrane thickness, transport time, sodium hydroxide concentration in the receiving phase, and the pH of phenol in the source phase. The objective is to provide valuable insights into the application of PIM techniques for addressing significant challenges in wastewater treatment.

EXPERIMENTAL

Material and Methods

The equipment employed in this investigation comprised an analytical balance (Mettler Toledo AB54-S), an automatic calibration B1900126 digital pH meter, a Fourier Transform Infrared (FTIR) Spectrometer (Perkin Elmer UATR two C999515) for membrane characterization and phenol concentration analysis, a Scanning Electron Microscope (SEM; Field Emission, FEI, Quanta FEG 650), and a UV-Vis Spectrophotometer (Thermo Scientific GENESYS 150). Three pieces of equipment are utilized for phenol transport: a stirrer, magnetic stirrer, and chamber. The compartment is cylindrical and is divided by a PIM membrane. The region directly in touch with the phenol solution, or the chamber's effective diameter, is a mere 3.5 cm, despite the chamber's overall diameter of 5 cm (Fig.-1).[26] Tissue, masking tape, distilled water, aluminum foil, distilled water, Poly-BADGE carrier powder at concentrations of 1:1 and 3:1, as well as Merck pro-analyst (pa) quality chemicals such as phenol (C_6H_5OH), sodium hydroxide (NaOH), dibenzyl ether (DBE), polyvinyl chloride (PVC), chloroform ($CHCl_3$), hydrochloric acid (HCl), ammonium hydroxide (NH_4OH), sodium sulfate (Na_2SO_4), 4-aminoantipyrine ($C_{11}H_{13}N_3O$), potassium ferricyanide [$K_4Fe(CN)_6$], dipotassium phosphate (K_2HPO_4), monopotassium phosphate (KH_2PO_4), sodium nitrate ($NaNO_3$), sodium chloride (NaCl), and potassium nitrate (KNO_3).

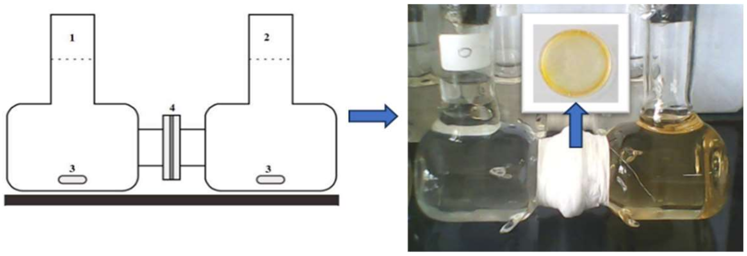


Fig.-1: Phenol Transport Pipeline, (1) SOURCE PHase; (2) Receiving Phase;(3) Magnetic Stirrer; (4) Membrane[26]

A 50 mL source phase solution with 60 ppm phenol is contained in one tube, while the other tube has a receiver phase solution of 50 mL of NaOH.

Variations in the pH of Phenol

The concentration of NaOH in the receiving phase is kept at 0.1 M, and the pH of phenol in the source phase is then varied to 3.5, 4.5, 5.5, 6.5, 7.5 and 8 using a PIM membrane that has been positioned in the middle of the transport pipe. The solution is then stirred for nine hours at room temperature using a magnetic stirrer in both the source and receiving phases.

Variations in the NaOH of Concentrations

By gradually increasing the NaOH concentration to 0.01, 0.05, 0.1, 0.25 and 0.5 M, the effect of NaOH on the acceptor phase was examined. The ideal pH for the phenol acquired in the previous process and the phenol in the source phase stays constant. Using a magnetic stirrer, the two solutions in the compartment were stirred for nine hours at room temperature.

Variations in the Thickness of the Membrane

PIM membranes are composed of basic components with three distinct total weights. Table-1 presents a comparison between the Poly-BADGE 1:1 and 3:1 carrier and PIM membrane components.[27,28]

Table-1: Comparing the Components of PIM Membranes

Membrane Thickness	10% Poly-BADGE (g)	32% PVC (g)	58% DBE (g)	Total Weight (g)
T_{27} (Thin)	0.027	0.0864	0.1566	0.2700
T_{54} (Normal)	0.054	0.1728	0.3132	0.5400
T_{108} (Thick)	0.108	0.3456	0.6264	1.0800

PIM membranes are printed with a carrier and available in three thicknesses: T_{27} (thin), T_{54} (normal), and T_{108} (thick). By positioning these membranes in the middle of the chamber pipe, they allow phenol to be transported under ideal circumstances, such as when the receiving phase's NaOH concentration and the pH of the phenol in the source phase are both at the proper levels. The compartment was then stirred for nine hours at room temperature using a magnetic stirrer.

Variations in the Transport Time

The influence of time on the phenol transport was examined taking into consideration the phenol's pH in the first phase the amount of NaOH present in the receiving phase, and the ideal membrane thickness from the previous approach. The solution was stirred for 4, 9, 24, 32 and 48 hours at room temperature using a magnetic stirrer.

RESULTS AND DISCUSSION

Variations in the pH of Phenol

The study found that the pH level of phenol in the source phase had a significant impact on the transport efficiency of phenol. This is explained by the effect that the differential in proton concentration between the source and acceptor phases has on phenol diffusion across the membrane phase. Figure-2 illustrates how, during the time of the nine-hour transport process, the phenol concentration in the receiving phase (%Cp) affects the phenol pH in the source phase using the Poly-BADGE 1:1 and 3:1 carrier.

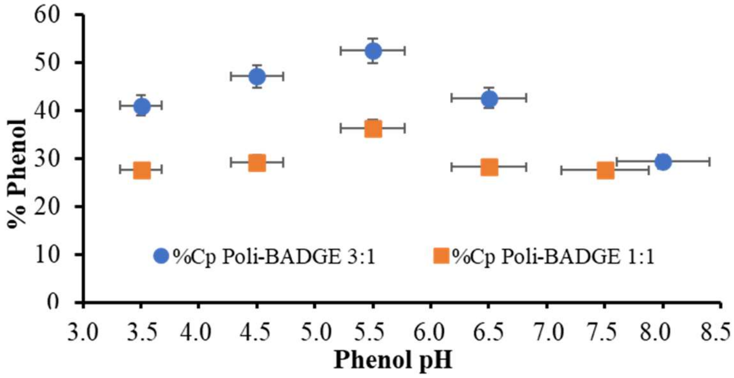


Fig.-2: Phenol pH Impact on the Percentage of Phenol Conveyed

%Cp Poly-BADGE 3:1 represents the amount of phenol present at the receiving phase. The data collected explains why the effectiveness of phenol transfer declines when the source phase's pH rises above pH 5.5.

Therefore, it can be concluded that at pH 5.5, with a %Cp of 36.33% for poly-BADGE 1:1 and 52.55% for poly-BADGE 3:1, the best phenol transport with the carrier poly-BADGE 1:1 and 3:1 occurs. This is because phenol is more soluble in the organic phase (membrane phase) at pH 5.5. It is, after all, more likely to exist in molecule form, where a greater number of hydrogen bonds may form. Due to phenol's propensity to dissolve more readily in the organic phase, there are more relationships between π-π and hydrogen bonding the phenol and the membrane phase carrier. The amount of phenol transferred increases as a result of this increased contact.[29,30]

Variations in the NaOH of Concentrations

Another crucial element is the quantity of NaOH in the receiving phase. In the receiving phase, phenol may be stripped using a chemical called NaOH to produce phenolic, which cannot re-enter the membrane phase.[26] Using the Poly-BADGE 1:1 and 3:1 carrier, Fig.-3 illustrates how the phenol content in the receiving phase affects the concentration of NaOH during the nine-hour transport method.

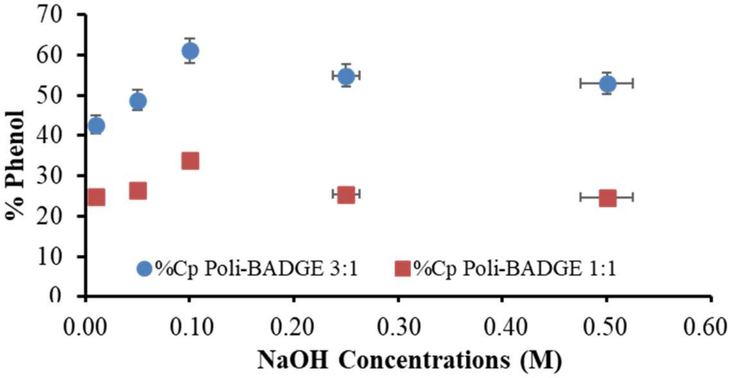


Fig.-3: Impact of NaOH Content on the Percentage of Phenol Conveyed

According to the results, the %Cp rises as the receiving phase's NaOH concentration increases. Nonetheless, the %Cp decreased at NaOH concentrations greater than 0.10 M. According to the study, phenol transport was most successful in the NaOH acceptor phase at concentrations of 0.10 M, with percentage changes of 33.90% for the carrier Poly-BADGE 1:1 and 61.12% for Poly-BADGE 3:1. The membrane-receiving phase contact will develop more quickly during the release of phenolic ions if the NaOH receiving phase solution has strong enough basic properties at a concentration of 0.10 M, increasing the amount of phenol transformed into phenolic ions. The observed phenol concentration increases with the amount of phenolic ions present in the receiving phase.[31] However, too concentrated or alkaline NaOH concentrations might cause the membrane's active site to leak into the receiving phase. As a result, the membrane has fewer active sites and is unable to phenol transport.[5,26]

Variations in the Thickness of the Membrane

Membranes with varying thicknesses will result from variations in the combined weight of the constituent parts. Next, a thickness gauge is used to measure the thickness of the manufactured PIM membrane. Because the solvent spontaneously evaporates throughout the membrane-making process, the final PIM membrane weighs less than the sum of its parts. Therefore, it's likely that some of the components that make the membrane will also evaporate, resulting in a membrane that weighs less overall than the components together.[5] Figure-4 shows how the thickness or total weight of the PIM membrane is affected by the concentration of phenol in the receiving phase during the nine-hour transport procedure when using the Poly-BADGE 1:1 and 3:1 carrier.

Changes in PIM membrane thickness can affect the proportion of phenol transport that has been conducted, as evidenced by the data acquired. Research on PIM membrane thickness changes varies based on the kind of membrane thickness. Based on the results of the research, the T_{54} membrane, characterized by a thickness of 0.26 mm and a total mass of 0.54 grams, achieves optimal phenol transport, registering a %Cp of 36.15% with the 1:1 Poly-BADGE carrier and a %Cp of 61.20% with the 3:1 Poly-BADGE carrier. This is because there is a very maximum number of phenol molecules interacting with the carrier, resulting in a membrane that is neither too thick nor thin. According to the data, the amount of carrier and phenol's barriers to passing through the membrane phase are balanced, allowing phenol to pass through the membrane phase more quickly and dissociate into phenolic at the receiving phase. The optimal thickness of T_{54} is the proportion of phenol present in the receiving phase.[26]

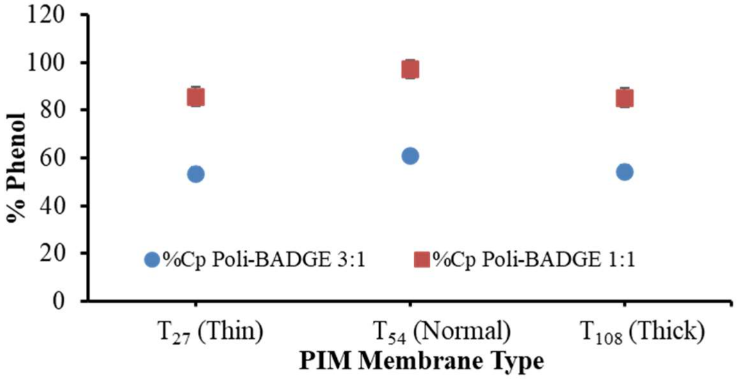


Fig.-4: Impact of different PIM Membrane Thicknesses on the Percentage of Phenol Conveyed

Variations in the Transport Time

Time is a critical factor in the transmission of phenol that must be considered. The effectiveness of the membrane in the phenol separation and recovery process is demonstrated by the transport time. The time of transport affects the phenol's velocity. The transport time and the duration of contact between the membrane carrier and the phenol in the source phase are closely correlated. Phenol in the source phase interacts with the membrane carrier via hydrogen bonding via π-π interactions.[26] The longer the transport time, the greater the interaction will be between the phenol and the membrane carrier. As phenol and the carrier on the membrane come into increasing contact, it is expected that the concentration of phenol transmitted to the receiving phase will increase. An efficient membrane is demonstrated by a brief transport time with the maximum amount of phenol transmitted.[25] The results of research that looked at how the transport time process affected the amount of phenol in the receiving phase during the 9-hour phenol transport method are shown in Fig.-5. The study employed Poly-BADGE 1:1 and 3:1 carrier.

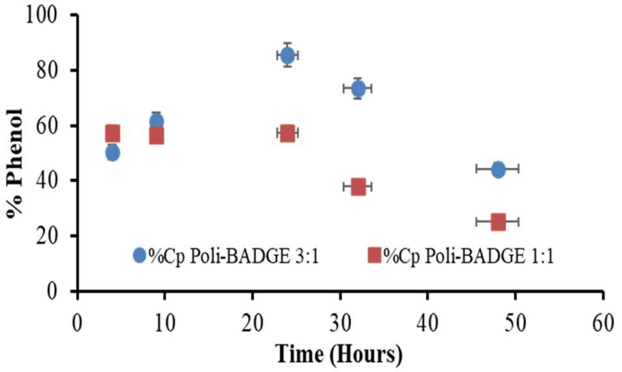


Fig.-5: Transport Time's Effect on the Proportion of Phenol Transported

24-hour transport period is found to be the most effective phenol transport technique, with a percentage change of 57.31% in the Poly-BADGE 1:1 carrier and 85.30% in the Poly-BADGE 3:1 carrier, according to the data generated. A quicker phenol transport process is the outcome of a longer time interval between the phenol and the carrier on the membrane during the transport phase. An increased release of phenolic ions into the receiving phase is caused by the extended interaction of phenol with the membrane's carrier.[32,33] This does not, however, apply to phenol transport time of 32 and 48 hours, which are longer than 24 hours. This is feasible because, if the transport time exceeds 24 hours, the receiving phase's NaOH may no longer be able to produce sodium phenolate in greater amounts. As a result, the longer contact time also reduces the components in the membrane phase (leaching), which lowers the membrane's active site.[25]

Characterization of PIM Membranes

The PIM membrane was then examined using FTIR and SEM both before and after the transport process. The infrared spectra of the characteristic peaks in the FT-IR spectrum for both Poly-BADGE 1:1 and 3:1 PIM membranes (Fig.-6) demonstrate that these two PIM membranes underwent minimal changes in wave number. The PIM membrane underwent an FTIR characterization test before and during transportation, yielding these findings.

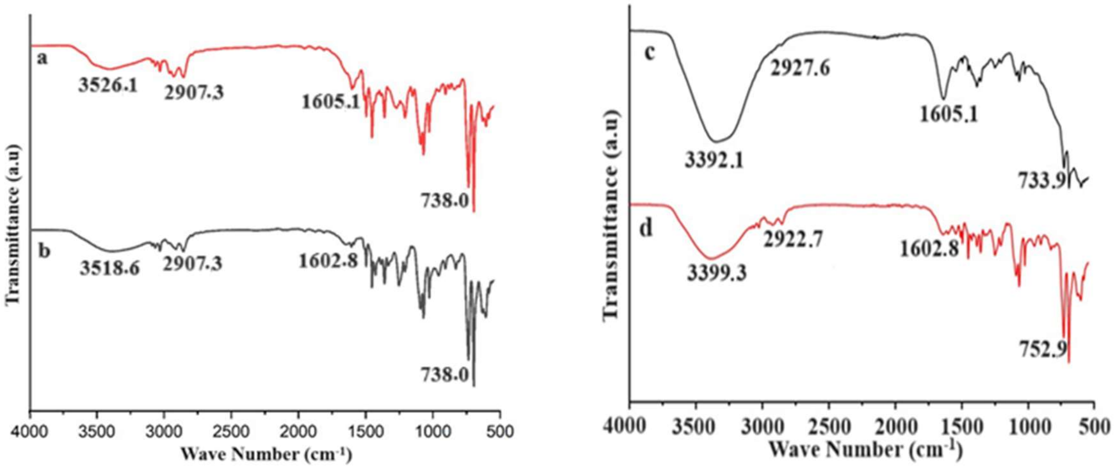
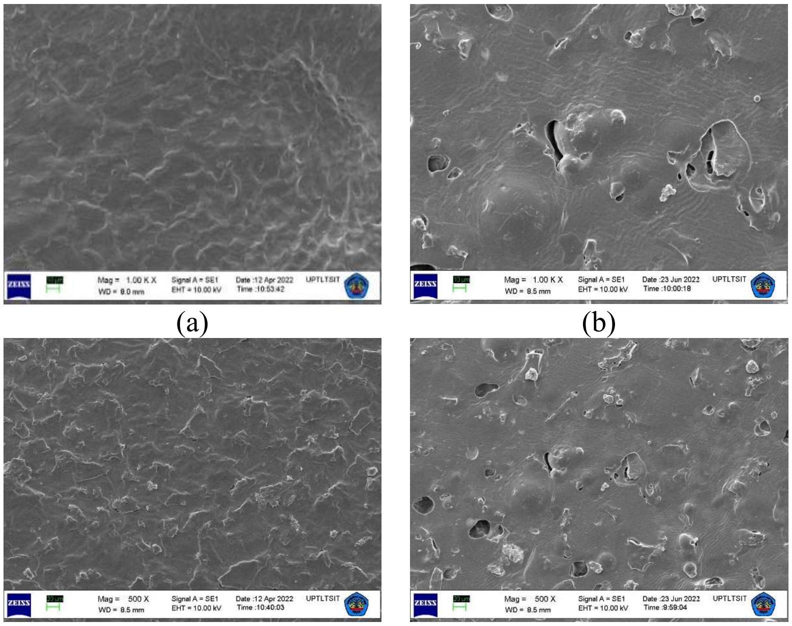


Fig.-6: Comparison of Infrared Spectra on Membrane PIM with (left) Poly-BADGE 1:1 carrier (a) after Transport and (b) before Transport; (right) Poly-BADGE 3:1 Carrier (c) after Transport and (d) before Transport

The IR spectra of Fig.-6 typically show the following characteristic peaks: vinyl (CH_2=CH-), aromatic rings (C=C), hydrogen bonds (-OH), and -C-H stretching. The PIM membrane contains the same functional groups both before and after transport, however, there are negligible variations in wave numbers, as seen by the specific absorption in both spectra. A comparison of the two spectra shows that the absorption of the -OH group on the membrane before transport experiences a leftward shift in the absorption band and a decrease in intensity at wave numbers 3518.6 cm^{-1} (left Poly-BADGE 1:1) and 3392.1 cm^{-1} (right Poly-BADGE 3:1). This suggests that hydrogen bonds are present. Due to the -OH groups on both phenol and the Poly-BADGE carrier, hydrogen bonding between the two -OH groups may develop. The bulk of the Poly-BADGE active site on the membrane is lost throughout the transport process as a result of hydrogen bonds formed between the Poly-BADGE carrier and the -OH phenol active site. Certain active areas on the PIM membrane are depleted during the transport process, according to the results of the SEM characterization of the membrane. Refer to Fig.-7 for the SEM characterization outcomes illustrating the surface morphology both before and after translocation of the PIM membrane. When examining the membrane of PIM before transportation using the carriers Poly-BADGE 1:1 at 1000x magnification (Fig.-7(a)(b)) and Poly-BADGE 3:1 at 500x magnification (Fig.-7(c)(d)), it is evident that the plasticizer, which serves as the liquid medium, is still covering the membrane's surface. After phenol transfer, the PIM membrane will have holes and an uneven surface. The PIM membrane's porous nature allows for the formation of holes on its surface during transport. These holes are the result of membrane components leaching a process that occurs throughout the transport phase.[29,30] Zhao *et al.*[34] stated in their research that when membranes are used for transportation processes, there is a possibility that the components that make up the membrane can experience dissolution. In other words, during the transportation process, the components that make up the membrane can dissolve in the membrane phase, raising considerations regarding the stability and sustainability of membrane performance in the longer term.[35]

After phenol transfer, there were notable morphological alterations in the PIM membrane. As pores develop, the membrane's surface becomes uneven. Liquid Membrane loss, the term for the loss of membrane components during the transport process, is what causes these holes to develop. This alteration in membrane morphology suggests leaching of the membrane's constituent parts. Plasticizers, base polymers, and transporters can all provide components for leaching membranes. As a result, these morphological modifications have an impact on how well the membrane transports phenol. Moreover, it is demonstrated by shifts in FTIR spectra and modifications in membrane morphology that hydrogen bonding interactions between the phenol and carrier are crucial for the passage of phenol across PIM membranes. These findings and characterization demonstrate that the research was successful in determining how different factors affect the flow of phenol across the PIM membrane. It is possible to enhance membrane performance in phenol separation and recovery applications across a range of industrial settings by comprehending these elements.



(a) (b)

(c) (d)

Fig.-7:Comparison of PIM Membrane Morphology with 1:1 Poly-BADGE Carrier (a) before Transport; (b) after Transport and with 3:1 Poly-BADGE Carrier (c) before Transport; (d) after Transport

CONCLUSION

This study demonstrates that a prudent use of Poly-BADGE-based PIM membranes can result in an effective transport technique to mitigate phenol wastewater contamination. The outcomes demonstrated how several factors, such as the source phase's pH, the receiving phase's concentration (NaOH), the thickness of the PIM membrane, and the length of contact, influenced the efficiency of phenol transfer. The ideal pH for phenol transfer was 5.5, and 0.10 M of NaOH was used. Additionally, we discovered that, with a %Cp value of 85.30%, a Poly-BADGE ratio of 3:1 was superior to a ratio of 1:1 in terms of boosting the proportion of phenol delivered. It should be mentioned, nonetheless, that an excessively lengthy contact period may cause the effectiveness of phenol transfer to decline. FTIR and SEM analysis of the membrane before and after transport revealed that a change in hydrogen bonds and a loss of membrane-active sites affected the membrane's performance in phenol transport. Overall, this study offers insightful information for the creation of effective PIM membranes that treat wastewater phenol contamination while accounting for a range of factors that affect the phenol transport process.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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