Jurnal Sains Nuklear Malaysia, 2026, 38(Special Issue): 25 – 36
ISSN: 2232-0946

FABRICATION OF POLYVINYLIDENE FLUORIDE-BASED POLYMER INCLUSION MEMBRANES FOR THE SELECTIVE EXTRACTION OF RARE EARTH ELEMENTS FROM ELECTRONIC WASTE LEACHATES

Fathiyyah Nadhirah Abdul Fauzi[1], Maziati Akmal M.H.[2*], Wan Syasya Nabilah[3], Wafiuddin Ismail[1] and Ahmad F.B.[3]

[1] Department of Chemistry, Kulliyyah of Science,
International Islamic University Malaysia (IIUM),
25200, Kuantan, Pahang, Malaysia
[2]Department of Science in Engineering, Kulliyyah of Engineering,
International Islamic University Malaysia (IIUM), Jalan Gombak,
53100 Kuala Lumpur, Malaysia
[3]Department of Chemical Engineering & Sustainability, Kulliyyah of Engineering,
International Islamic University Malaysia (IIUM), Jalan Gombak,
53100 Kuala Lumpur, Malaysia
[*]Correspondence author: maziatiakmal@iium.edu.my

ABSTRACT

Rare earth elements (REEs) are important materials of modern technologies, but these elements are difficult to obtain from conventional mining due to both environmental and geopolitical reasons, which has led to the exploration of secondary sources such as e-waste. Herein, we report on the early-stage development and evaluation of PIMs, prepared using PVDF, with the aim of extracting REEs. Membranes were fabricated using three different solvents, namely dimethylacetamide (DMAc), N-methyl-2-pyrrolidone (NMP), and dimethylformamide (DMF), using the NIPS technique and then quenched with either water or air. The membranes were characterized by analyzing the mechanical strength, thickness, and morphology, and the results were analyzed to determine the influence of fabrication conditions on the structural properties relevant to REE transport. The results indicated that water-quenched membranes were generally thicker than air-quenched ones, whereas membranes made with NMP had the highest tensile strength, at approximately 4.3 MPa. Scanning Electron Microscopy (SEM) images of the membranes confirmed that those fabricated with DMAc had the most porous structure, followed by DMF and NMP, which is consistent with the lower swelling index (40.68%) and higher water absorption (8.94%) of these membranes, allowing extractants to diffuse more readily through the membrane. Overall, these results demonstrate that the choice of solvent and quenching method is critical to the determination of the membrane strength and porosity, two properties that are critical to the efficient extraction of REEs, and future work will incorporate ionic liquid extractants and assess the transport of REEs from e-waste leachates.

Keywords: Rare Earth Elements; Polymer Inclusion Membranes; Electronic waste; Polyvinylidene Fluoride; Nonsolvent Induced Phase Separation

INTRODUCTION

Rare earth elements (REEs) form a collection of 17 chemically similar elements that can be found in the central region of the periodic table (atomic numbers 21, 39, and 57–71) (Bashiri et al., 2022). They are critical in both traditional and emerging industries, with applications ranging from consumer electronics and renewable energy to lighting and medical technology (Ali et al., 2022a; Balaram, 2019; Liu et al., 2019). These elements are essential in a variety of technologies, including fluorescent lights, lasers, superconducting magnets, atomic power sources, and turbine engines (Dev et al., 2020; Golroudbary et al., 2022; Haque et al., 2014). Interestingly, while they are called "rare," these elements are not rare at all, as they appear in the Earth's crust in higher abundance than precious metals like gold or platinum (Croft et al., 2024). The challenge is extracting them, which is technologically problematic and costly because REEs appear in small quantities, usually in parts per billion (ppb). Their principal mineral sources include monazite, xenotime, lanthanide, allanite, bastnäsite, loparite, and phosphate minerals (Ali et al., 2022).

Ironically, despite the fact that REEs are critical for modern and green technologies, the industrial extraction of REEs from ores often leads to high environmental contamination and production costs (Hammache et al., 2021; Liang et al., 2024). Furthermore, the separation of REEs is extremely difficult because of the similarity on chemical and physical properties, which has made it necessary to develop sustainable and environmentally friendly methods for recovery (Filho et al., 2023). Numerous techniques such as solvent extraction, ion exchange, precipitation, crystallization, and adsorption have been explored over the years to recover REEs from various sources, including mineral ores, industrial byproducts, and even environmental samples (Pavón et al., 2022; Quijada-Maldonado & Romero, 2021; W. Zhang et al., 2020). Although these techniques have been useful for selective separation, they have limitations, including low selectivity and purity of the recovered elements, the large amount of waste generated, and the large number of chemical reagents used (Chen & Zheng, 2019). Taken together, these challenges emphasize the need for greener and more efficient strategies for REE recovery.

In place of conventional solvent extraction techniques, polymer inclusion membranes, or PIMs, have shown great promise. The components of these membranes are a plasticizer, a carrier (extractant), and a base polymer (Croft et al. in 2018). Among these materials, PVDF-based PIMs stand out as promising choices. The homopolymerization or copolymerization of vinylidene fluoride monomers produces PVDF, which is known for its exceptional flexibility, wear resistance, and resistance to impacts and environmental contaminants (Kazemi and Yaftian, 2024; Turgut et al. 2017; Zheng and colleagues, 2018). Polymer inclusion membranes (PIMs) are more stable than many traditional liquid-liquid extraction systems, but they are still not durable enough to be used on a large scale in industry (Kazemi & Yaftian, 2024; Kogelnig et al., 2011). As a result, recent research has increasingly focused not only on improving membrane stability but also on optimizing other critical performance factors, such as extraction rates and extractive capacity.

In this study, the properties of PVDF-based PIMs fabricated by the phase inversion technique were examined in relation to the combined effects of solvent type, quenching method, and PVDF concentration. Additionally, this work addresses difficulties in membrane fabrication by analyzing significant mechanical, structural, and physical parameters. Physical characterization involves tensile strength tests as well as the analysis of morphology, swelling index, and water uptake. On the other hand, chemical characterization focuses on identifying functional groups using Fourier Transform Infrared Spectroscopy (FTIR).

MATERIALS AND METHODS

Materials

Polyvinylidene fluoride (PVDF) with an average molecular weight of 180,000 g/mol was obtained from Sigma-Aldrich and used as the base polymer in this study. Aliquat 336 ($C_{25}H_{54}ClN$). Additionally, a quaternary ammonium salt with a molecular weight of 404.16 g/mol, was also sourced from Sigma-Aldrich and employed as an extractant. As solvents, N, N-dimethylformamide (DMF, C_3H_7NO; 73.09 g/mol), N,N-dimethylacetamide (DMAc, C_4H_9NO; 87.12 g/mol), and dimethyl sulfoxide (DMSO, $(CH_3)_2SO$; 78.13 g/mol) were all purchased from Sigma-Aldrich. Deionized water with a resistivity of 18 MΩ·cm, produced in-house using a laboratory-grade purification system, was used for all experimental procedures.

Fabrication of PVDF-based PIM via Phase Inversion

The membranes were fabricated using the non-solvent induced phase separation (NIPS) method, with PVDF as the base polymer. To evaluate the influence of processing conditions, formulations were prepared with different solvents (DMAc, NMP, and DMF) and at varying PVDF concentrations. For each formulation, 3.00 g of PVDF pellets were dissolved in 30 mL of solvent, maintaining a ratio of 1 mL of solvent per 0.1 g of polymer (Hoque et al., 2019). The solutions were stirred at 60 °C for approximately 2 hours until complete dissolution was achieved, then cast evenly onto a glass plate supported with non-woven fabric.

Two quenching methods were applied. In the water quenching process, the cast film was immediately immersed in a precipitation bath at room temperature. In the air quenching method, the film was briefly exposed to ambient air before immersion. After quenching, the membranes were dried for 24 hours and subsequently cut to appropriate sizes for further analysis.

Characterization of PVDF-based PIM

A combination of four analytical techniques was employed to evaluate the physical, chemical, structural, and mechanical properties of the fabricated membranes.

Functional group analysis

Fourier Transform Infrared Spectroscopy (FTIR) was used to identify functional groups present in the membranes. Spectra were collected in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1}, using transmittance mode and 32 scans at room temperature.

Morphological analysis

The surface morphology of the membranes was examined using Scanning Electron Microscopy (SEM). Prior to imaging, the pre-dried PVDF membranes were coated with platinum. Observations were carried out at a magnification of 3000×.

Mechanical properties analysis of the PVDF-PIMs

The tensile strength of the PVDF-PIMs was measured following the procedure described by Baczyńska et al. (2018). Tests were conducted at room temperature using a universal testing machine (SHIMADZU), as shown in Figure 1. Membrane samples were cut into strips measuring 5 cm in length and 0.5 cm in width, and each strip was clamped vertically between two pairs of 50 mm forceps. To ensure reliability, three replicates were tested for each sample, and the average value was reported.

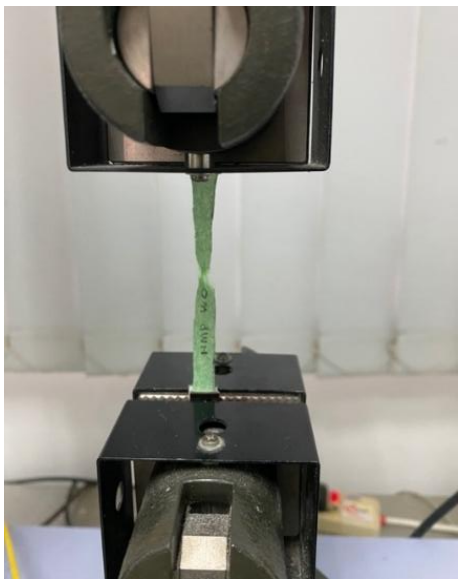

Figure 1: Tensile strength test using Universal Tensile Machine (UTM)

Physical properties analysis of the PVDF-PIMs

Water uptake (WU, %) and swelling degree (SD, %) were determined to assess the physical characteristics of the membranes. Dried PVDF-PIMs were cut into strips of approximately 30 × 10 mm and immersed in deionized water at 25 °C for 48 hours. After immersion, the samples were gently blotted with absorbent paper to remove surface water. Water uptake was then calculated using Equation (1):

$$WU = \frac{(m_{hydrated} - m_{dry})}{(m_{dry})} \times 100\% \quad (1)$$

where m_{dry} is the membrane mass before immersion and $m_{hydrated}$ is the membrane mass after immersion.

The swelling degree was measured by comparing membrane thickness before and after immersion, as shown in Equation (2):

$$SD = \frac{(x_{hydrated} - x_{dry})}{(x_{dry})} \times 100\% \quad (2)$$

where x_{dry} represents the thickness of the dry membrane and $x_{hydrated}$ is the thickness after immersion.

RESULTS AND DISCUSSION

Swelling index and water uptake measurement

The swelling index indicates how much a membrane swells when absorbing a liquid, often water. The membrane's thickness or volume changes because of this absorption. Water uptake, however, indicates the total amount of water absorbed by the membrane without showing any physical expansion (Zheng et al., 2018).

According to Table 1, the membrane fabricated using DMAc and subjected to air quenching exhibits the lowest swelling index (40.68%) and the highest water uptake (18.94%) compared to those

prepared with NMP and DMF. During absorption, hydrogen bonds form between water molecules and the active sites of the carrier. More carrier content means more active sites, allowing for greater water retention in the membrane (Elozeiri et al., 2024). These findings suggest that DMAc is a preferable solvent for producing highly porous membranes for REE extraction.

Table 1: Swelling index and water uptake results

Type of Quenching	Solvent	Water Uptake (%)	Swelling Index (%)
Air	DMAc	18.94	40.68
	NMP	9.28	79.63
	DMF	11.32	61.54
Water	DMAc	18.75	42.11
	NMP	13.69	78.85
	DMF	13.34	81.84

Thickness of PVDF-PIMs measurement

PVDF-PIMs prepared with DMAc and DMF became noticeably thicker after immersion in water compared to those exposed to air (Figure 2). This is most likely caused by pore enlargement from water absorption when soaking. Additionally, solvents that attract water, like DMAc and DMF, often cause the polymer to precipitate quickly. Rapid precipitation can cause pore expansion and an increase in membrane thickness (Bashiri et al., 2022). Quenching in water accelerates phase separation, resulting in membranes with more porosity and a higher tendency to expand while absorbing water. The observed changes in membrane thickness after immersion reflect the degree of swelling, which is closely associated with the membrane's internal structure, particularly its porosity and hydrophilic character (Venault et al., 2014).

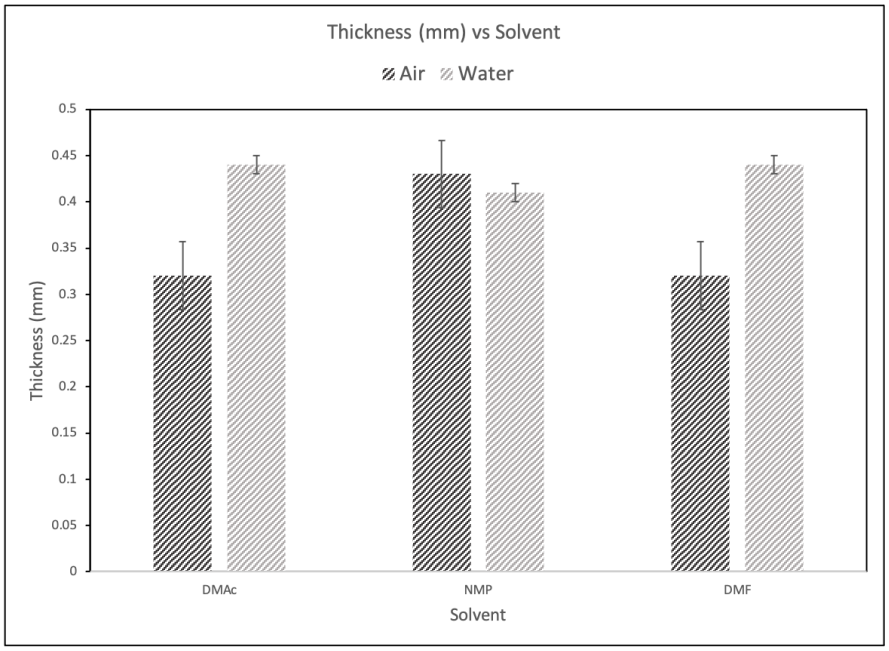

Figure 2: Thickness of PVDF-PIMs dissolved in different solvents

Tensile strength analysis

The tensile strength results obtained from the Universal Tensile Machine (UTM) for membranes prepared under different solvent systems and quenching conditions are presented in Figure 3. Of the samples tested, the membrane made with DMAc as the solvent and air quenched has a moderate tensile strength of 4.07 MPa. Typically, mechanical strength values between 2 and 4 MPa are considered sufficient for REE extraction, where membranes work under low-pressure conditions for long periods. Applying higher pressure may compress the polymer structure, which in turn could interfere with the membrane's ability to maintain stable REE transport performance (Anim-Mensah et al., 2025).

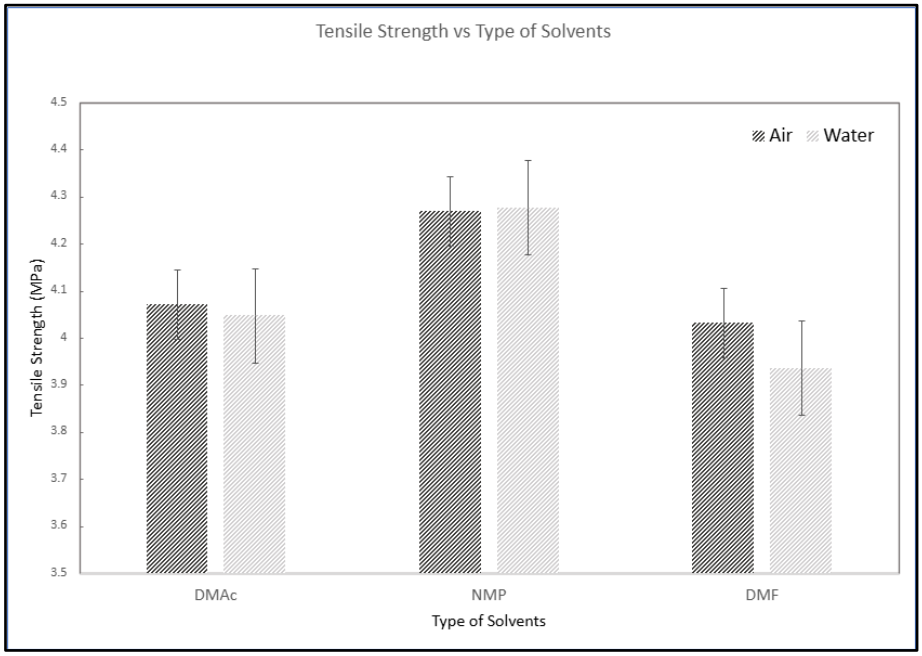

Figure 3: Tensile strength of PVDF-PIMs dissolved in varying solvents and quenching conditions

Morphology analysis

The SEM micrographs displayed in Figure 4 demonstrate the cross-sectional structure of PVDF-based PIMs created with various solvents at a 3000× magnification. The choice of solvent affects the membrane's structure due to differences in properties, like volatility, viscosity, and compatibility with the nonsolvent. The membrane made with DMAc shows a highly porous structure with many interconnected macrovoids, indicating fast phase inversion due to quick solvent-nonsolvent exchange. This observation matches the findings of AlMarzooqi et al. (2016), who concluded that membranes made solely with DMAc solvent develop a two-part structure consisting of finger-like and sponge-like sections.

As seen in Figure 5, the membrane fabricated with a PVDF concentration of 1.0 g/L exhibits a highly porous and interconnected structure, marked by large, round, and evenly distributed voids. This clear morphology is more defined than membranes made with 0.5 g/L and 0.75 g/L PVDF. Fundamentally, the interconnected pore network in the membrane prepared at a concentration of 1.0 g/L facilitates the movement and transport of REE ions, thereby enhancing both permeability and selectivity. This

enhancement is due to the direct and efficient pathways it provides for REE ion migration through the mesoporous surface layer and the macrovoids-rich bulk layer (Cheng & Bae, 2024).

In comparison, the membrane containing 0.5 g/L PVDF exhibits a very open yet uneven pore morphology, characterized by thin polymer walls and macrovoids. Hence, the overall network from 0.5 g/L PVDF concentration appears disordered and less structured.

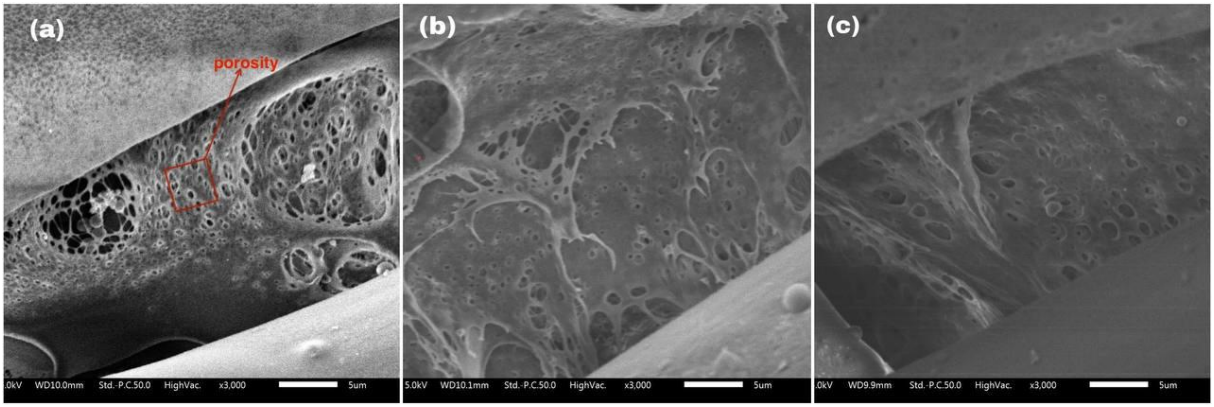

Figure 4: SEM results of PVDF-PIMs dissolved in different solvents (a) DMAc, (b) DMF, (c) NMP at 3000 times magnification

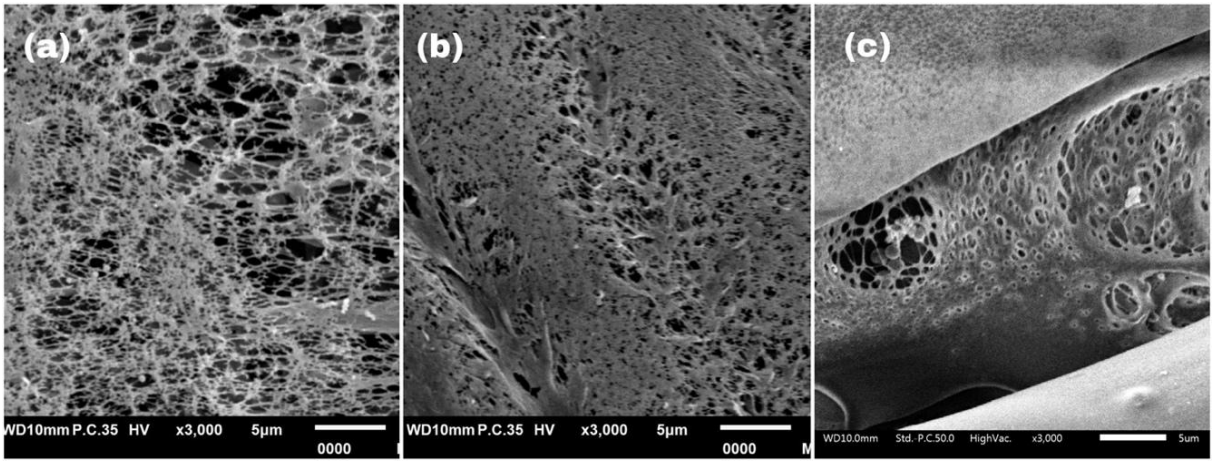

Figure 5: SEM results of PVDF-PIMs dissolved in different PVDF concentrations using DMAc solvent (a) 0.5 g/L, (b) 0.75 g/L, (c) 1.0 g/L at 3000 times magnification.

Functional group analysis

FTIR spectroscopy was used to analyze the chemical structure and phase composition of PVDF membranes produced with 3 different types of solvents (DMAc, DMF, NMP) and varied PVDF concentrations (0.5, 0.75, and 1.0 g/mL). Figure 6 illustrates the comparison of FTIR spectra for PVDF membranes cast with varied solvents. The characteristic peaks of the β-phase of PVDF were observed at 841 and 1240 cm^{-1}, which are linked to CF_2 stretching and skeletal vibrations characteristic of the polar crystalline arrangement (Li et al., 2013). All membranes exhibited characteristic absorption bands of PVDF, which are peaks at 840 cm^{-1} (CF_2 stretching), 872 and 720 cm^{-1} (CH_2 rocking), 1240 cm^{-1} (CF_2 asymmetric stretching), 1406 cm^{-1} (CH_2 bending), and the region between 1016 and 1092 cm^{-1} (C–F stretching) (Barrau et al., 2018).

Several of these peaks, specifically at 490, 720, 872, 1016, and 1406 cm^{-1} are linked to the α-phase of PVDF which correspond to CF_2 wagging, CH_2 rocking, and CF_2 wagging, and non-polar backbone

vibrations. In addition, the presence of a C=O peak around 1710 cm^{-1} indicates residual solvent traces, most likely from DMAc, DMF, or NMP (Cai et al., 2017).

Figure 7 shows the comparison when using different PVDF concentrations. The FTIR spectra indicated that as the concentration of PVDF increased, the baseline noise decreased, suggesting enhanced membrane uniformity and stronger polymer matrix formation. While all concentrations displayed both α- and β-phase signals, the α-phase peaks became increasingly dominant at higher PVDF content, especially at 1.0 g/mL.

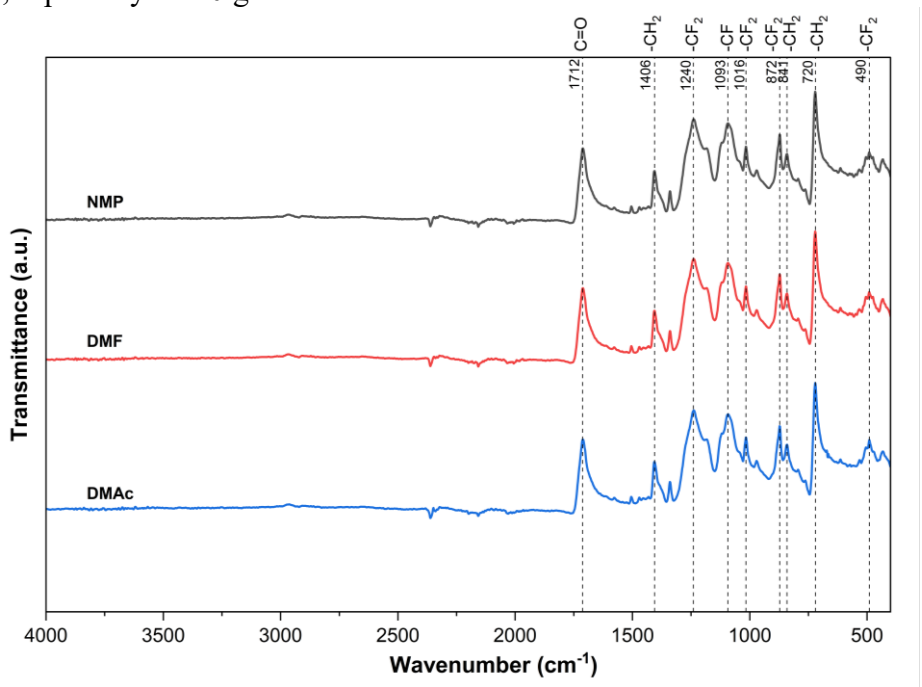

Figure 6: FTIR Spectra of PVDF-PIMs dissolved in different solvents (NMP, DMF, DMAc)

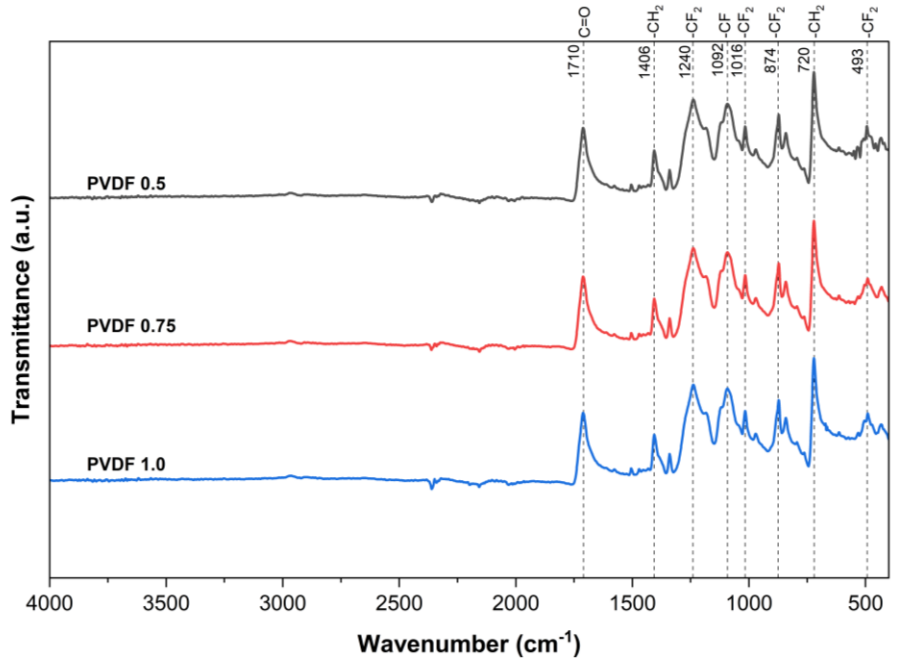

Figure 7: FTIR Spectra of PVDF-PIMs with different PVDF concentrations (0.5, 0.75, 1.0 g/L)

CONCLUSION

In conclusion, the comprehensive evaluation of the synthesized PVDF-PIM membranes has elucidated the critical parameters influencing membrane efficiency in REE extraction. This study effectively identified that the optimal combination of solvent and quenching method is DMAc with air quenching, which resulted in a remarkable swelling index of 40.68% and the highest water uptake at 18.94%. The membranes demonstrated moderate tensile strength of 4.07 MPa and a highly porous network, characterized by interconnected macrovoids that significantly enhance their performance.

A detailed examination of the PVDF concentration revealed that membranes fabricated with 1.0 g/L PVDF exhibited the best overall properties, balancing higher water uptake (4.71%) with relatively low swelling index (6.67%), suggesting a balance between hydrophilicity and dimensional stability, and developing a more defined porous structure, which is closely related to improved permeability and mechanical strength. Functional group characterization further validated these results by indicating the molecular organization of the 1.0 g/L membrane, which can be correlated with its chemical stability and suitability for long-term performance in separation applications.

To advance the applicability of PVDF-PIMs in more challenging environments, future research should expand the range of characterization tests carried out. For instance, water contact angle measurements would be useful to better understand how the membrane surface interacts with liquids, which in turn affects wetting behavior and ion transfer efficiency. In addition, studying how variations in water bath temperature during the quenching process influence pore development and membrane morphology could reveal important insights. These assessments would allow a clearer link to be established between fabrication conditions, surface properties, and overall membrane stability, providing a stronger basis for tailoring membranes to specific applications.

In addition to surface and quenching analyses, another critical direction for future work is to examine how physical structure and mechanical performance influence membrane functionality. Furthermore, the correlation among membrane thickness, ion transport rates, and tensile strength will be essential for maximizing the extraction efficiency of rare earth elements. A thicker membrane may offer mechanical durability but may slow ion transfer, whereas thinner membranes may offer higher transport rates at the cost of strength. A systematic study of these trade-offs is necessary. The structural and chemical characteristics of PVDF-PIMs can be carefully tuned to optimize performance for laboratory testing as well as for long-term industrial operation where durability, selectivity, and consistent performance are all necessary.

ACKNOWLEDGEMENTS

The work in this manuscript was funded by the Ministry of Higher Education Malaysia (FRGS/1/2024/TK09/UIAM/02/1).

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