**Effect of Acid Treatment in Zeolite-Y as Filler in Polysulfone based Membrane for Water Desalination**

Berlian Imazdalifa1, Henky Aden Saputra1, Lukman Atmatdja1, R.Y. Perry Burhan1, Ardi Nugroho Yulianto2, Tutuk D Kusworo3, Irvan Irvan4, Triyanda Gunawan1,a)

Author Affiliations

1Department of Chemistry, Faculty of Science and Data Analytics, Institut Teknologi Sepuluh Nopember, Sukolilo, Surabaya 60111, Indonesia

2Department of Naval Architecture, Faculty of Marine Technology, Institut Teknologi Sepuluh Nopember, Sukolilo, Surabaya 60111, Indonesia

3Department of Chemical Engineering, Faculty of Engineering, Unversitas Diponegoro, Semarang 50275, Indoensia

4Department of Chemical Engineering, Faculty of Engineering, Universitas Sumatera Utara, Medan, 20155, Indonesia

Author Emails

a) Corresponding author: triyanda@its.ac.id

**Abstract.** Desalination using membranes is an important option to address the shortage of clean water, as it offers lower energy use, reduced operating cost, and flexibility in scale. In this study, polysulfone (PSf) membranes were prepared by phase inversion with the addition of acid-activated Zeolite-Y (0.5–1.5 wt%) as filler. Acid treatment was applied to improve the pore structure and surface properties of Zeolite-Y. The filler was characterized by XRD, and the membranes were tested for water contact angle (WCA), pure water flux, and NaCl rejection. The PSf/ACZ 1.0 membrane gave the best performance, with a WCA of 74.8°, a water flux of 153.1 ± 3.6 L·m⁻²·h⁻¹, and a salt rejection of 95.96 ± 2.66%. For comparison, the neat PSf membrane had a flux of 108.4 ± 0.35 L·m⁻²·h⁻¹ and a rejection of 92.88 ± 4.56%, while the PSf/NaY 1.0 membrane (unmodified zeolite-Y) achieved 61.3 ± 0.9 L·m⁻²·h⁻¹ flux and 95.60 ± 0.87% rejection. These results confirm that acid activation of zeolite-Y enhances its role as a filler, giving higher water permeability while maintaining high salt rejection. This study demonstrates that acid-activated zeolite-Y is a promising additive for mixed-matrix PSf membranes and provides a useful approach for further improving desalination performance.

**Keywords** : Polysulfone membrane, Acid-activated zeolite-Y, Mixed-matrix membrane, desalination, zeolite-Y

# introduction

Water scarcity is becoming one of the biggest global problems. Reports from the UN warn that by 2025 about two-thirds of the world population will live under water stress, and around 1.8 billion people will face serious shortages of clean water [1,2]. Since most of the water on earth, about 97%, is seawater, desalination is seen as one of the main ways to provide fresh water. At the moment membrane desalination is used more than thermal processes. Reverse osmosis alone contributes to more than 65% of desalination plants in operation worldwide [3].

Polysulfone (PSf) is a polymer that is often chosen for membrane preparation[4–10]. It is strong, stable at high temperature, and not expensive. The main drawback is that it is hydrophobic, so the water flux is low and fouling is usually high. Because of this, many studies try to add fillers to PSf to improve its hydrophilicity. Zeolite is one of the fillers that is often used because it has pores and surface hydroxyl groups that can interact with water molecules [11–15].

Several works already reported the benefits of zeolite in PSf. Anbealagan et al. (2021) showed that adding zeolite RHO at 0.5–1 wt% could increase water flux by about 45% compared to neat PSf [16]. But untreated zeolite-Y usually contains impurities in its pores. These impurities limit ion transport and reduce the effect of zeolite. Acid treatment is one of the ways to clean and open the pores. Velichkina et al. (2021) reported that acid activation of zeolite-Y increased its surface area by more than 30% and also improved adsorption [17].

There are still very few studies that combine acid-activated zeolite-Y with PSf membranes for desalination. Most studies only used untreated zeolite or other types of fillers. In this work, PSf membranes were prepared with the incorporation of low loading (0.5–1.5 wt%) acid-activated zeolite-Y. The fillers and membranes were characterized, and tested the water contact angle, flux, and salt rejection. The purpose is to see how acid treatment of zeolite-Y affects the performance of PSf membranes and whether it can be useful for desalination applications.

# materials and methods

**Materials**

The materials used in this study were commercial Zeolite-Y (Jiangsu Xfnano Materials Tech Co., Ltd.) and hydrochloric acid (HCl, 37%, SAP) for the acid activation process. Polysulfone (PSf, Udel-P3500, Amoco Chemicals, USA) and *N-methyl-2-pyrrolidone* (NMP, Merck) were used for membrane preparation. Sodium chloride (NaCl, 99.9%, Sigma-Aldrich) and demineralized water (aqua-DM) were used in the desalination experiments.

# Synthesis Acid Activation of Zeolit-Y

The acid activation of Zeolite-Y was carried out employing the method of Side et al. (2023), albeit with some modifications [18]. First, 100 g of commercial Zeolite-Y was rinsed with 1000 mL of distilled water and agitated with a magnetic stirrer for 3 to 4 hours. The suspension was then heated to 50 °C until its weight stayed the same. After drying, 1 g of the zeolite powder was mixed with 40 mL of 2 M HCl and agitated for 15 hours at room temperature. To get the acid-activated Zeolite-Y powder, the solid was filtered, rinsed well with distilled water, and then dried in an oven to obtain a fine white powder.

# Polysulfone/Acid Activated Zeolite-Y for Membrane Preparation

The fabrication of membranes refers to the previous method with some adjustment [19,20]. Dope solutions are prepared from solvent polysulfone and NMP polymers with a 15:85 w/w ratio. First, the already prepared activated zeolite (0; 0,5; 1; 1,5 wt% relative to Psf mass) were dispersed into NMP through ultrasonication for 30 minutes and name as Neat, PSf/ACZ0.5, PSf/ACZ1.0, and PSf/ACZ1.5, respectively. The MMMs with 1.0wt% of Zeolite-Y was also fabricated using the same protocol as PSf/ACZ1.0 to be used as comparison. Then followed by the addition of polysulfone pellets are added little by little into the mixture and stirred at 420 rpm for 12 hours. The dope solutions were then sonicated for 2 mins followed by stored inside the cabinet to remove the excess bubble. The flat sheet membranes were fabricated through the phase inversion method into a coagulant bath containing aqua DI. The post treatment process by immersing the membrane in ethanol to eliminate any residual solvent and followed by drying for 24 hours.

# Characterization and Performance Test of Membrane

The Acid-activated zeolite-Y filler is characterized using X-ray diffraction (XRD-Philips PW1140/90) to determine the crystal phase formed with a specific phase that refers to the main peak of the diffractogram pattern through the Debye Scherrer equation approach. During the characterization process, the X-rays used are generally Cu-Kα radiation with a wavelength of 1.54 Å.

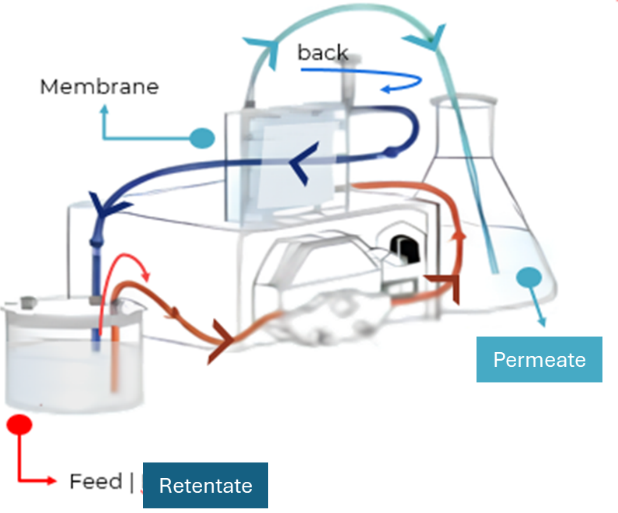
WCA (Water Contact Angle) measurement is carried out to determine the hydrophility of the resulting contact angle. WCA testing is measured by dripping water over the surface of the membrane, then the angle of contact formed is measured. Membrane samples are prepared at a square size of 5 cm x 5 cm. Hydrophility is detected with a contact angle of less than 90 degrees. Basically, hydrophility is defined as a condition in which water is easily absorbed on the surface of the membrane. Thus, the opposite condition when the contact angle value is more than 90 degrees is categorized as hydrophobicity.

In addition, the flux test is determined based on the amount of permeate passing through the membrane and the rejection test (Figure 1). The concentration of salt used was (NaCl 3.5wt%). The membrane to be tested is cut into a square shape with a size of 50 cm2 and inserted into a filtration module. Next. The filtration test was carried out with a time variation of 10 minutes and 15 minutes. Flux can be calculated using the formula:

where, J is the flux (l/m2.h), V is the permeate volume (ml), A is the surface area of the membrane (m2), and t is the time (hours).

As for finding out the concentration of salt after passing through the membrane, the value of the rejection coefficient is measured. The rejection coefficient can be calculated by using the formula

where, R is the rejection coefficient (%), Cp is the concentration of solute in the permeate, and Cf is the concentration of solute in the feed.



**FIGURE 1**. schematic tools for membrane performance test

# RESULT AND DISCUSSION

# Results of X-Ray Diffraction (XRD) on Acid Activated Zeolite-Y

Figure 2 shows diffractogram of zeolite-Y with peaks of 2θ = 6.3°; 10,2°; 11,9°; 20,3°; 23,5°; 26,9°; 30,6°; 31.2°. The result of the diffractogram results in the highest peak at 2θ = 6.3°, which indicates the zeolite-Y crystal plane (111). The results of the zeolite-Y diffractogram are in accordance with the previous reported research, where zeolite-Y has a typical peak at 2θ ~6° [21–25]. The results of the XRD diffractogram from zeolite-Y are also in accordance with the Powder Diffraction File (PDF) obtained from the Joint Committee on Powder Diffraction Standards (JCPDS) Data Base number 39-1380. Thus, it can be confirmed that the commercial zeolite used is indeed zeolite-Y. Furthermore, it shows that the diffractogram pattern of activated zeolite-Y is similar to the pristine zeolite, indicating that the structure of the zeolite remains intact after the HCl treatment, indicating a very strong acid resistance properties. However, there were slight difference in the intensity of the activated zeolite, which is stronger than pristine zeolite. This is due to the slight increment in the Si/Al ratio in zeolite after acid treatment. Resulting in higher crystallinity and a more perfect zeolite crystal structure [18].

A graph of a red line

Description automatically generated with medium confidence

**FIGURE 2**. XRD results of zeolite- Y and Acid-Activated Zeolite-Y 2M

# Results of Characterization and Performance Test of Membrane

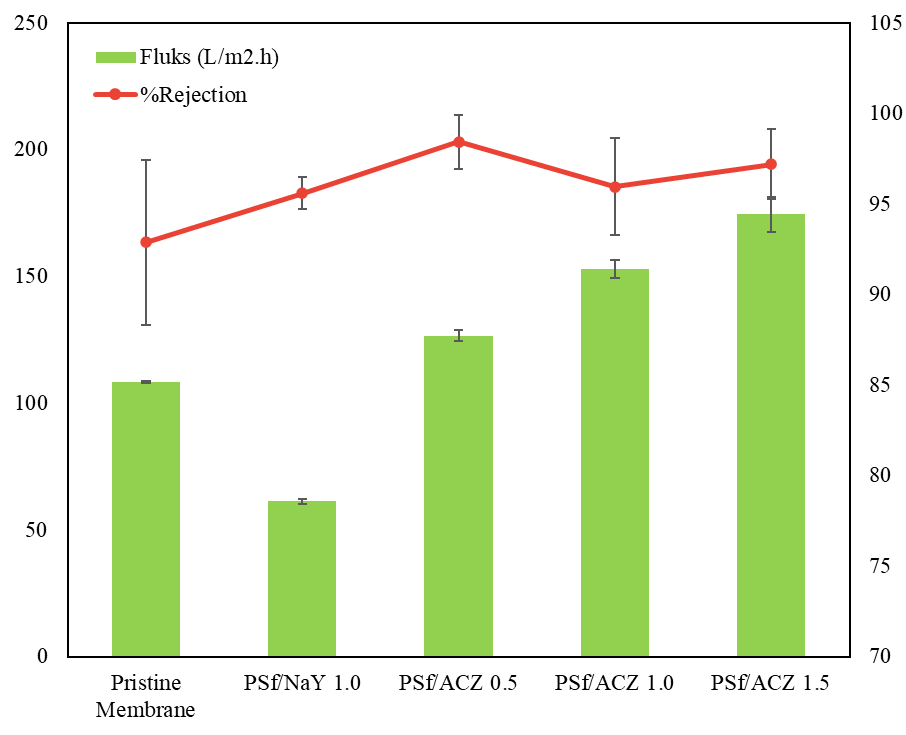
Figure 3 shows the water contact angle of the membranes. The neat PSf membrane had the highest value, 113.35 ± 3.98°, which shows it is hydrophobic [26]. When acid-activated zeolite-Y was added, the value decreased. At 0.5 wt% the contact angle was 79.54 ± 0.80°, and at 1 wt% it reached the lowest value of 74.76 ± 0.07°. At 1.5 wt% the contact angle increased again to 84.88 ± 1.81°. The contact angle drop at 1 wt% filler incorporation suggests that the zeolite was well distributed in the polymer, giving more hydrophilic sites. The increase at 1.5 wt% is likely due to agglomeration of the filler, which reduced the effect. This trend is like other mixed-matrix membranes reported in the literature, where higher filler loading often leads to aggregation. Overall, the addition of acid-activated zeolite-Y made the PSf membrane more hydrophilic, with the best result at 1 wt% [27].

A graph of blue rectangular bars

AI-generated content may be incorrect.

**FIGURE 3**. Membrane water contact angel analsys results

The results of flux and rejection are shown in Table 1 and Figure 4. The neat PSf membrane gave a flux of 108.4 ± 0.35 L·m⁻²·h⁻¹ with a rejection of 92.88 ± 4.56%. When unmodified zeolite-Y was added (PSf/NaY 1.0), the flux dropped to 61.3 ± 0.89 L·m⁻²·h⁻¹, while the rejection went up slightly to 95.60 ± 0.87%. With acid-activated zeolite-Y, the performance was better. At 0.5 wt% loading, the flux increased to 126.7 ± 2.17 L·m⁻²·h⁻¹ and the rejection reached 98.44 ± 1.49%. At 1 wt% loading, the flux was 153.1 ± 3.56 L·m⁻²·h⁻¹ and the rejection was 95.96 ± 2.66%. The highest flux was obtained at 1.5 wt% loading (174.5 ± 6.96 L·m⁻²·h⁻¹) with a rejection of 97.20 ± 1.91% [28]. These results show that acid activation improved the role of zeolite-Y as a filler. The membranes with ACZ had higher flux and still maintained high rejection. At higher loadings the flux kept increasing, while the rejection stayed above 95%. Compared to neat PSf and PSf/NaY, the ACZ-filled membranes gave a better balance between flux and rejection.



**FIGURE 4**. Membrane Separation Performance

**Table 1** Membrane performance test results

|  |  |  |
| --- | --- | --- |
| **Polysulfone Membrane** | **Water Flux (L/m­2h)** | **Rejection (%)** |
| Neat | 108.38 ± 0.35 | 92.88 ± 4.56 |
| Psf/NaY 1.0 | 61.26 ± 0.89 | 95.60 ± 0.87 |
| PSf/ACZ 0.5 | 126.66 ± 2.17 | 98.44 ± 1.49 |
| PSf/ACZ 1.0 | 153.07 ± 3.56 | 95.96 ± 2.66 |
| PSf/ACZ 1.5 | 174.51 ± 6.96 | 97.20 ± 1.91 |

# CONCLUSION

This study showed that incorporating acid-activated zeolite-Y into polysulfone membranes improved desalination performance. The best balance was observed at 1 wt% loading, where the membrane had a water contact angle of 74.8°, a water flux of 153.1 ± 3.6 L·m⁻²·h⁻¹, and a salt rejection of 95.96 ± 2.66%. Compared to the neat PSf membrane (flux 108.4 ± 0.35 L·m⁻²·h⁻¹, rejection 92.88 ± 4.56%) and the PSf/NaY membrane (flux 61.3 ± 0.89 L·m⁻²·h⁻¹, rejection 95.60 ± 0.87%), the acid-activated zeolite-Y membranes showed both higher water permeability and consistently high rejection. These results confirm that acid activation enhances the role of zeolite-Y as a filler, providing membranes with better hydrophilicity, higher flux, and reliable salt rejection. Further work should focus on optimizing filler distribution, exploring different operating pressures, and testing long-term performance under real seawater conditions.

# ACKNOWLEDGEMENTS

The author greatly appreciates the financial support given by Institut Teknologi Sepuluh Nopember, through Penelitian Prima, scheme Riset Kolaborasi Indonesia under contract no: 950/PKS/ITS/2025.

# reference

[1] A. Abushawish, I. Bouaziz, I.W. Almanassra, M.M. AL-Rajabi, L. Jaber, A.K.A. Khalil, M.S. Takriff, T. Laoui, A. Shanableh, M.A. Atieh, A. Chatla, Desalination Pretreatment Technologies: Current Status and Future Developments, Water (Switzerland) 15 (2023). https://doi.org/10.3390/w15081572.

[2] R. Mahdavi Far, B. Van der Bruggen, A. Verliefde, E. Cornelissen, A review of zeolite materials used in membranes for water purification: history, applications, challenges and future trends, Journal of Chemical Technology and Biotechnology 97 (2022) 575–596. https://doi.org/10.1002/jctb.6963.

[3] M. Tawalbeh, L. Qalyoubi, A. Al-Othman, M. Qasim, M. Shirazi, Insights on the development of enhanced antifouling reverse osmosis membranes: Industrial applications and challenges, Desalination 553 (2023) 116460. https://doi.org/10.1016/j.desal.2023.116460.

[4] R. Wijiyanti, A.N. Ubaidillah, T. Gunawan, Z.A. Karim, A.F. Ismail, S. Smart, R. Lin, N. Widiastuti, Polysulfone mixed matrix hollow fiber membranes using zeolite templated carbon as a performance enhancement filler for gas separation, Chemical Engineering Research and Design 150 (2019). https://doi.org/10.1016/j.cherd.2019.08.004.

[5] T. Gunawan, S.R. Lusman, S.N.P. Wahyudhie, A.R. Widyanto, N. Widiastuti, H. Fansuri, S.D. Nurherdiana, D. Prasetyoko, Introducing PDMS layer on PSF membrane to enhance CO2/CH4 separation, in: The 5th International Seminar on Chemistry (ISOC) 2022, 2024: p. 020007. https://doi.org/10.1063/5.0206252.

[6] N. Widiastuti, I.S. Caralin, A.R. Widyanto, R. Wijiyanti, T. Gunawan, Z.A. Karim, M. Nomura, Y. Yoshida, Annealing and TMOS coating on PSF/ZTC mixed matrix membrane for enhanced CO 2 /CH 4 and H 2 /CH 4 separation, R Soc Open Sci 9 (2022) 322–334. https://doi.org/10.1098/rsos.211371.

[7] A. Junaidi, N. Sazali, T. Gunawan, N. Widiastuti, A Mini Review on Advancements in Membrane Technologies for CO2 Separation: The Role of Polyphenylene Sulfide Fillers, Journal of Applied Membrane Science & Technology 29 (2025) 1–17. https://doi.org/10.11113/jamst.v29n1.305.

[8] W.H. Ng, N.N.R. Ahmad, C.P. Leo, A.L. Ahmad, Polysulfone/SAPO-34 zeolite membrane impregnated with 1-ethyl-3-methyl imidazolium bis(tri-fluoromethylsulfonyl)imide ionic liquid for CO2 removal, in: 2019: p. 030004. https://doi.org/10.1063/1.5117126.

[9] H.S. Fahmy, R. Abouzeid, M.S.A. El-sadek, G.T. Abdel-Jaber, W.Y. Ali, H.M. Mousa, Fabrication of polysulfone membranes by blending with polyaniline and cellulose nanocrystals: towards the effective separation of oil-in-water emulsions, Cellulose 30 (2023) 5871–5893. https://doi.org/10.1007/s10570-023-05237-1.

[10] L. Badrinezhad, S. Ghasemi, Y. Azizian-Kalandaragh, A. Nematollahzadeh, Preparation and characterization of polysulfone/graphene oxide nanocomposite membranes for the separation of methylene blue from water, Polymer Bulletin 75 (2018) 469–484. https://doi.org/10.1007/s00289-017-2046-7.

[11] M. Sakai, Y. Nomura, M. Matsukata, Strategy of Development on Zeolite Membrane for Forward Osmosis Operation; Effects of Structural Parameter of Support Layer and Si/Al Ratio of ZSM-5 Layer on Water Flux, Journal of Chemical Engineering of Japan 56 (2023). https://doi.org/10.1080/00219592.2023.2251529.

[12] H.L. Choi, Y. Jeong, H. Lee, T.H. Bae, High-Performance Mixed-Matrix Membranes Using a Zeolite@MOF Core-Shell Structure Synthesized via Ion-Exchange-Induced Crystallization and Post-Synthetic Conversion, JACS Au 4 (2024) 253–262. https://doi.org/10.1021/jacsau.3c00680.

[13] L. Li, J. Dong, T.M. Nenoff, R. Lee, Desalination by reverse osmosis using MFI zeolite membranes, J Memb Sci 243 (2004) 401–404. https://doi.org/10.1016/j.memsci.2004.06.045.

[14] D. Astira, R. Abdullah, U. Zulfiani, D.O. Sulistiono, Z. Rahmawati, T. Gunawan, M.H. Dzarfan Othman, M. Hasan, R. Ediati, H. Fansuri, Comparing two synthesis methods: Exploring unique characteristics and catalytic activity of fenton catalyst Fe3O4/zeolite-NaY in methylene blue degradation, Case Studies in Chemical and Environmental Engineering 10 (2024) 100963. https://doi.org/10.1016/j.cscee.2024.100963.

[15] D. Astira, R. Abdullah, A.R. Widyanto, H.N. Cipta Dharma, L. Santoso, D.O. Sulistiono, Z. Rahmawati, T. Gunawan, J. Jaafar, M.H. Dzarfan Othman, R. Ediati, M. Hasan, H. Fansuri, A recent development on core-shell-based material and their application in membranes for water and wastewater treatment, Inorg Chem Commun 160 (2024) 111678. https://doi.org/10.1016/j.inoche.2023.111678.

[16] L.D. Anbealagan, T.Y.S. Ng, T.L. Chew, Y.F. Yeong, S.C. Low, Y.T. Ong, C. Ho, Z.A. Jawad, Modified Zeolite/Polysulfone Mixed Matrix Membrane for Enhanced CO2/CH4 Separation, Membranes (Basel) 11 (2021) 630. https://doi.org/10.3390/membranes11080630.

[17] L. Velichkina, Y. Barbashin, A. Vosmerikov, Effect of Acid Treatment on the Properties of Zeolite Catalyst for Straight-Run Gasoline Upgrading, Catalysis Research 1 (2021). https://doi.org/http://dx.doi.org/10.21926/cr.2104004.

[18] S. Side, S.E. Putri, D.E. Pratiwi, A. Rahma, Abd. Rahman, The Effect Of Acid Treatment On The Characteristics Of Modernite Zeolite, Sainsmat : Jurnal Ilmiah Ilmu Pengetahuan Alam 12 (2023) 114. https://doi.org/10.35580/sainsmat122511932023.

[19] S.N.P. Wahyudhie, S.R. Lusman, A.R. Widyanto, N. Widiastuti, T. Gunawan, H. Fansuri, Y. Kusumawati, S. Akhlus, A. Hamzah, A.D. Pramata, Performance of polysulfone membrane for biogas upgrading, in: The 5th International Seminar on Chemistry (ISOC) 2022, 2024: p. 020010. https://doi.org/10.1063/5.0206253.

[20] A. Junaidi, U. Zulfiani, S. Khomariyah, T. Gunawan, N. Widiastuti, N. Sazali, W.N.W. Salleh, Utilization of polyphenylene sulfide as an organic additive to enhance gas separation performance in polysulfone membranes, RSC Adv 14 (2024) 2311–2319. https://doi.org/10.1039/d3ra06136a.

[21] T. Gunawan, R. Wijiyanti, N. Widiastuti, Adsorption–desorption of CO 2 on zeolite-Y-templated carbon at various temperatures, RSC Adv 8 (2018) 41594–41602. https://doi.org/10.1039/C8RA09200A.

[22] A.R. Widyanto, I.S. Caralin, N. Widiastuti, T. Gunawan, R. Wijiyanti, W.N.W. Salleh, A.F. Ismail, M. Nomura, K. Suzuki, Improvement N2/SF6 separation performance on P84 derived carbon membrane by incorporating of zeolite-carbon composite, AIP Conf Proc 2349 (2021) 020008. https://doi.org/10.1063/5.0052171.

[23] T. Gunawan, T.Q. Romadiansyah, R. Wijiyanti, W.N. Wan Salleh, N. Widiastuti, Zeolite templated carbon: Preparation, characterization and performance as filler material in co-polyimide membranes for CO2/CH4 separation, Malaysian Journal of Fundamental and Applied Sciences 15 (2019) 407–413. https://doi.org/10.11113/mjfas.v15n3.1461.

[24] T. Gunawan, N. Widiastuti, A.R. Widyanto, H. Fansuri, S. Akhlus, W.N.W. Salleh, A.F. Ismail, N. Sazali, R. Lin, J. Motuzas, S. Smart, Fine-tuning zeolite pore structures with carbon coating for enhanced gas separation in polyimide-based mixed matrix membrane, Chemical Engineering Research and Design 204 (2024) 556–571. https://doi.org/10.1016/j.cherd.2024.02.042.

[25] R. Wijiyanti, I.S. Caralin, A.R. Widyanto, T. Gunawan, Z.A. Karim, A.F. Ismail, M. Nomura, N. Widiastuti, Evaluation of different carbon-modified zeolite derivatives preparation methods as a filler in mixed matrix membrane on their gas separation performance, Microporous and Mesoporous Materials 359 (2023) 112650. https://doi.org/10.1016/j.micromeso.2023.112650.

[26] U. Zulfiani, A.W. Pratama, R. Abdullah, D. Astira, T.Q. Romadiansyah, A.R. Widyanto, Saiful, J. Jafaar, T. Gunawan, N. Widiastuti, Improving the performance of HDPE plastic membranes for heavy metal removal by incorporating EVA/PDMS, Results in Engineering 24 (2024) 103643. https://doi.org/10.1016/j.rineng.2024.103643.

[27] T. Gunawan, N. Widiastuti, A.R. Widyanto, H. Fansuri, S. Akhlus, W.N.W. Salleh, A.F. Ismail, N. Sazali, R. Lin, J. Motuzas, S. Smart, Fine-tuning zeolite pore structures with carbon coating for enhanced gas separation in polyimide-based mixed matrix membrane, Chemical Engineering Research and Design 204 (2024) 556–571. https://doi.org/10.1016/j.cherd.2024.02.042.

[28] A. Elrasheedy, N. Nady, M. Bassyouni, Matrix Membranes : Review on Applications in Water Purification, Membranes (Basel) 9 (2019) 1–31. https://doi.org/10.3390/membranes9070088.