Journal of Physical Science
Vol. 37(1), 77–93, 2026

Comparison Study on Separation of Diethanolamine, Monoethanolamine and Triethanolamine from Wastewater using Thin Film Composite Membrane

Qurratu Ain Uzma Saifuddin, Sunarti Abd Rahman,* Syed Mohd Saufi Tuan Chik
and Ellora Priscille Ndia Ntone

Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang
Al-Sultan Abdullah, 26300 Gambang, Pahang, Malaysia

*Corresponding author: sunarti@umpsa.edu.my

To cite this article: Saifuddin, Q. A. U. et al. (2026). Comparison study on separation of diethanolamine, monoethanolamine and triethanolamine from wastewater using thin film composite membrane. *J. Phys. Sci.*, 37(1), 77–93. https://doi.org/10.21315/jps2026.37.1.4

To link this article: https://doi.org/10.21315/jps2026.37.1.4

ABSTRACT: This study presents a comparative evaluation of thin film composite (TFC) membranes for the separation of monoethanolamine (MEA), diethanolamine (DEA) and triethanolamine (TEA) from wastewater. Amines, commonly released from gas treatment, pharmaceutical and chemical industries, must be removed due to their toxicity, contribution to eutrophication and interference with biological treatment processes. The performance of TFC membranes was assessed in terms of water permeability, pure water flux and amine rejection under varying operational conditions, including amine concentration, type, inlet pressure and membrane type. Results showed distinct separation efficiencies, with TEA (53.28%) achieving the highest rejection, followed by DEA (41.15%) and MEA (33.79%) at 150 ppm. Membrane surface properties, particularly contact angle (75.38°) and surface charge (–10 mV), significantly influenced separation behaviour. The findings highlight the potential of TFC membranes as an effective solution for amine-rich wastewater treatment and provide useful insights for optimising membrane-based separation processes in industrial applications.

Keywords: monoethanolamine, diethanolamine, triethanolamine, graphene oxide, thin film composite membrane

1. INTRODUCTION

Amines have long been released into the environment and aquatic systems, posing both benefits and drawbacks. Structurally, amines are functional groups and chemical compounds containing a basic nitrogen atom with a lone pair of electrons. They may include organic substituents such as aryl or alkyl groups or be derived from ammonia through the replacement of one or more hydrogen atoms.[1] Amides, in contrast, are characterised by the presence of a nitrogen atom adjacent to a carbonyl group ($R–C(=O)–NR_2$), and display diverse chemical properties. Examples of naturally occurring and synthetic amines include biogenic amines, aniline and trimethylamine (commonly associated with the odour of fish). Many amines possess strong, unpleasant odours and can be harmful. They contribute to the characteristic smell of substances such as urine, rotten meat, ammonia and decomposing seafood. The maximum permissible amine concentration in water samples is around 30 g/mL.[2] Therefore, accurate measurement of amine concentrations is essential for effective removal from water.

Membrane technology is widely recognised as an efficient approach for removing amines from aqueous solutions. Membranes play a crucial role in numerous separation processes and have become increasingly significant in modern chemical technology. Polyethersulfone (PES), owing to its excellent chemical and mechanical stability, is one of the most commonly used polymers for fabricating ultrafiltration and nanofiltration membranes in both industrial and research settings.[3] Graphene oxide (GO), a nanostructured material with high porosity and surface area, has gained attention as a nanofiller for membrane development. Incorporating GO into polymeric substrates or polyamide (PA) top layers can significantly improve water flux, hydrophilicity and fouling resistance, while also influencing the mechanical strength and roughness of the host polymer.[4–8]

Thin film composite (TFC) membranes, composed of a microporous support layer coated with a thin selective PA layer, are widely utilised in water treatment due to their excellent selectivity and chemical resistance. However, conventional TFC membranes often suffer from relatively low water flux and susceptibility to fouling. To overcome these limitations, researchers have explored various surface modification strategies aimed at enhancing hydrophilicity and antifouling performance.[9] For instance, the incorporation of porous zeolite particles into TFC membranes has been shown to increase water permeability with minimal loss of salt rejection.[10] Yin et al. developed a GO-modified hydrophilic PA nanofiltration membrane via interfacial polymerisation (IP) using m-phenylenediamine (MPD) and trimesoyl chloride (TMC), achieving effective removal of salts such as NaCl and Na_2SO_4.[11]

Amines such as monoethanolamine (MEA), diethanolamine (DEA) and triethanolamine (TEA) are extensively used in industries including gas treatment, pharmaceuticals and cosmetics.[12] Their discharge into wastewater poses environmental risks, as these compounds can disrupt aquatic ecosystems and contaminate water resources. Consequently, there is a growing need for efficient and sustainable treatment technologies for the separation and recovery of amines from industrial effluents.[13] Membrane filtration, particularly TFC membranes, has demonstrated strong potentials for the removal of organic contaminants such as amines owing to their high permeability, selectivity and chemical stability. The separation performance of TFC membranes, however, depends on several factors including the type and concentration of the amine, operating pressure and membrane material properties.

The most advanced membranes, including those used for gas separation, reverse osmosis, nanofiltration and pervaporation, are typically fabricated using organic polymers via phase separation techniques. Both wet and dry preparation methods exist for TFC and other composite membranes. Examples of dry methods include radiation polymerisation, photo-initiated polymerisation, plasma polymerisation and vapour deposition, while wet methods include polymer coating, spreading on water surfaces and interfacial polymerisation.[14,15] While membrane filtration for contaminant removal has been extensively studied, limited research exists on the comparative separation of MEA, DEA and TEA using TFC membranes. Despite their structural similarities, these amines differ in molecular size, polarity and other physicochemical properties that affect their interaction with membranes. Understanding these differences is critical for optimising wastewater treatment processes and improving the sustainability of industrial practices.

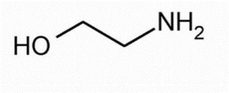
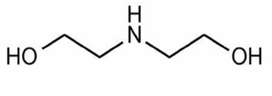
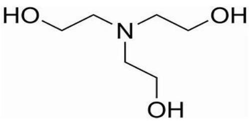
The aim of this study is therefore to systematically compare the separation performance of TFC membranes for MEA, DEA and TEA removal from wastewater. Specifically, the investigation evaluates pure water permeance, flux and amine rejection under varying operational conditions. By providing a comparative analysis, this research seeks to deliver insights that can guide the development of more efficient, cost-effective and sustainable membrane-based treatment technologies for amine-rich wastewater.

2. METHODOLOGY

2.1 Materials

To fabricate the PES substrate membrane, polyethersulfone resin (PES, Mw = 58,000 g/L), N,N-dimethylacetamide (DMAc, 99.9%) and polyvinylpyrrolidone (PVP, 99%, Mw = 25,000 g/mol) were supplied by KHL Global SDN BHD, Kuantan, Malaysia. The active monomers used to form the polyamide (PA) layer were piperazine anhydrous (PIP, 99%) and trimesoyl chloride (TMC, 99%), both purchased from Macklin, China. PIP and TMC were dissolved in deionised (DI) water and n-hexane, respectively, to prepare the aqueous and organic monomer solutions. Graphene oxide (GO, > 99%) nanofiller was obtained from Platonic Nanotech Private Limited. Finally, MEA, DEA and TEA solutions (150–300 ppm) were provided by KHL Global SDN BHD, Kuantan, Malaysia. The properties of MEA, DEA and TEA are summarised in Table 1.

Table 1: Characteristics of MEA, DEA and TEA

Properties	MEA	DEA	TEA
Solubility Differences	Highly soluble in water	Moderately soluble in water	Slightly soluble in water
Boiling Points	Boiling point around 170°C	Boiling point around 270°C	Boiling point around 360°C
Density	1.017 g/cm³ at 25°C	1.095 g/cm³ at 25°C	1.124 g/cm³ at 25°C
Acid-Base Properties	MEA, DEA and TEA are all alkanolamines, meaning they have both amine (–NH_2) and alcohol (–OH) functional groups. They can act as weak bases, forming salts with acids		
	Primary amine	Secondary amine	Tertiary amine
Molecular structure			

2.2 Preparation of PES Support Layer

Asymmetric flat-sheet PES dense membranes were fabricated by phase inversion using the immersion precipitation technique. Membranes were prepared with GO nanoplate incorporation for the mixed matrix membrane (MMM) approach, and without GO for the IP method. The casting solutions consisted of PES (15 wt.%), PVP (1 wt.%) and predetermined amounts of GO nanoplates dispersed in DMAc as the solvent. The detailed compositions of the casting solutions are listed in Table 2. To ensure uniform dispersion, GO nanoplates were first dispersed in DMAc and sonicated using DT 102H Bandelin ultrasonic equipment (Germany) for 30 min. PES and PVP were then added to the dope solution and stirred continuously for 24 h to achieve complete dissolution. Residual air bubbles were removed by an additional 10 min sonication step. The homogeneous solution was cast onto a clean glass plate at a thickness of 100 µm using a makeshift casting knife. The cast film was immediately immersed into a non-solvent bath of distilled water maintained at 15°C to induce phase separation. Following initial phase separation and membrane formation, the membranes were stored in fresh distilled water for 24 h to ensure complete phase inversion.

Table 2: The compositions of casting solutions

Membrane	PES (wt.%)	PVP (wt.%)	GO nanoplates (wt.%)
PES/GO-0	15	1.0	–
PES/GO-0.1	15	1.0	0.1
PES/GO-0.2	15	1.0	0.2
PES/GO-0.3	15	1.0	0.3
PES/GO-0.4	15	1.0	0.4

2.3 PES/GO Composite Nanofiltration Membrane Preparation

2.3.1 Interfacial polymerisation

The PES membranes were immersed in DI water for 24 h prior to use. After gentle drying in air for 2 min, the wet PES membranes were placed in a custom-built setup with the membrane surface facing upward. A mixture of PIP and dispersed GO was then applied onto the PES surface and allowed to remain for 120 s. Excess solution was removed from the membrane surface using a soft rubber blade and the aqueous phase solution. Subsequently, an IP reaction was initiated by contacting the membrane surface with an n-hexane organic solution containing 0.1 (w/v) % TMC. After the reaction, the organic solution was drained from the surface. To ensure complete polymerisation, the PPA/GO nanofiltration membranes were heat-treated at 80°C for 5 min. The membranes were then thoroughly rinsed and stored in DI water until use. These membranes were evaluated in aqueous solutions to determine the optimal GO content. The different PPA/GO composite nanofiltration membranes prepared by the IP method, along with their fabrication details and compositions, are presented in Table 3.

Table 3: The PPA/GO composite membrane prepared in several circumstances

Membrane ID	GO content (wt.%)	PIP content (wt.%)	TMC content (w/v%)	IP time (s)
PES/GO-0	0	1.5	0.1	30
PES/GO-0.1	0.1	1.4	0.1	30
PES/GO-0.2	0.3	1.2	0.1	30
PES/GO-0.3	0.5	1.0	0.1	30
PES/GO-0.4	1.0	0.5	0.1	30

2.4 Membrane Analysis

2.4.1 Membrane morphology analysis

The morphological structure of the fabricated nanofiltration (NF) membranes was examined using a field emission scanning electron microscope (FE-SEM, Hitachi S-5500, Tokyo, Japan).

2.4.2 Water contact angle analysis

The membrane contact angle was measured using a contact angle goniometer (JY-82, Chengde Testing Machine Co. Ltd., China). Each membrane sample was placed on a glass slide, and a droplet of 5 μL ultrapure water was carefully deposited onto the membrane surface using an 'I'-shaped needle. A static image of the water droplet was captured, and the contact angle was determined using image analysis software. Measurements were taken at five different positions on each membrane, and the average value was recorded.

2.4.3 Membrane porosity and mean pore radius measurement

Membrane porosity was determined using the gravimetric method, which measures the amount of water retained in the membrane pores. First, the membranes were immersed in distilled water at room temperature for 24 h. After soaking, excess surface water was gently removed with filter paper and the wet weight of the membrane was recorded. The membranes were then dried in an oven at 60°C for 12 h, and the dry weight was measured using an analytical balance. The membrane porosity was calculated using Equation 1.

$$\varepsilon = \frac{M_w - M_d}{Al\rho} \tag{1}$$

where,

ε = Porosity

m_w = Wet mass of the membrane (kg)

m_d = Dry mass of the membrane (kg)

A = Sample area (m^2)

L = Specimen thickness (m)

ρ = Density of pure water (kg/m^2)

The filtering procedure was used to estimate the membrane's mean pore size, which can be computed using Equation 2.

$$r_m = \sqrt{\frac{(2.9 - 1.75\varepsilon) \times 8\eta l Q}{\varepsilon \alpha \Delta P}} \tag{2}$$

where,

r_m = mean pore radius (m)

η = pure water viscosity (Pa.s)

L = specimen thickness (m)

Q = permeated flow (m3/s)

ΔP = applied pressure (Pa)

2.4.4 Surface chemistry

Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR, Japan) was used to analyse the surface functional groups of the PPA/GO and PPA membranes.

2.5 Performance of the Membrane

2.5.1 Amine rejection

A TFC/GO-based membrane with enhanced sustainability characteristics was fabricated using IP. Amine removal performance was evaluated through ultrafiltration (UF) adsorption experiments conducted in UF stirred cells. A feed solution containing 150 ppm of amine was used to test the membranes. Prior to testing, membranes were cut into circular samples with a diameter of 5.2 cm, rinsed with DI water, and mounted in the UF cell. The experiments were carried out at a constant pressure of 5 bar. Permeate samples were collected every 30 min over a period of 1 h, and the average of three replicate measurements was reported for each condition. Amine concentrations in the feed and permeate were analysed using gas chromatography (GC), and the rejection efficiency of the membranes was calculated using Equation 3.

$$Rejection = \left(1 - \frac{Cp}{Cf}\right) \times 100\% \tag{3}$$

where,

C_f = concentration of the amine solution in the feed

C_p = the concentration of the permeate.

2.5.2 Pure water permeability

Pure water permeability (PWP) is an important parameter in evaluating membrane performance. Accordingly, the fabricated membranes were tested for pure water filtration, and PWP was calculated using Equation 4.

$$WP = \left(\frac{\Delta V}{A \times \Delta t \times \Delta P} \right) \tag{4}$$

where,

V = the permeation volume (L)

A = the membrane surface area (m^2)

P = the applied pressure (bar)

t = the filtration time (h)

3. RESULTS AND DISCUSSION

3.1 Characterisation of GO-TFC Membrane

The SEM cross-section of the PES/GO-0, PES/GO-0.1, PES/GO-0.3, PES/GO-0.5 and PES/GO-1.0 membranes at ×1,000 magnification is shown in Figure 1. The images displayed the active layer on top of each of the five membranes at ×1,000 magnification. According to the study, the accumulating condition emerged as the GO ratio increased. The best results will be achieved when GO is evenly distributed throughout the membrane's surface.[16] The SEM images reveals a porous finger-like sub-layer on the PES substrate caused during the phase inversion process of the membrane. However, at the top layer, a thin layer is observed which is the presence of the PA layer of the GO. The combination of these structures is advantageous for pure water flux and amine removal during filtration.

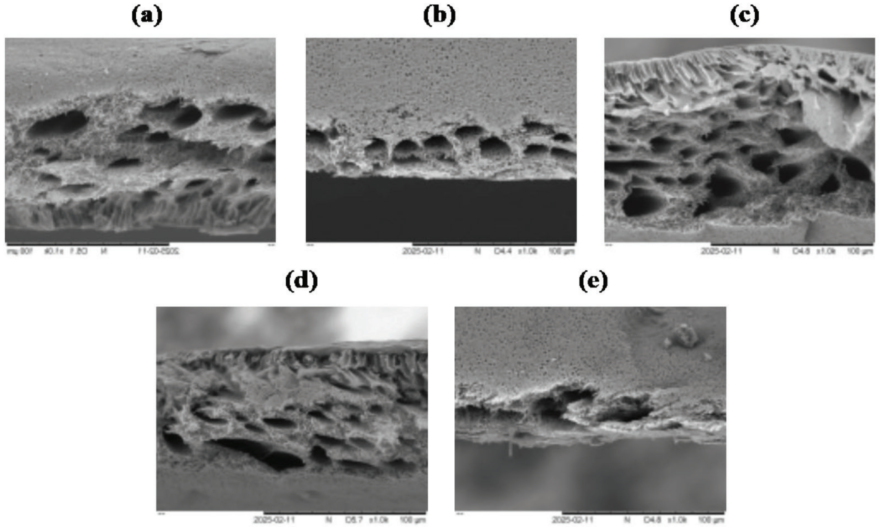


Figure 1: The SEM cross-sectional image of the GO-TFC membrane at ×1,000 magnification shows the ratio of GO, (a) 1.5:0.1:0.0, (b) 1.4:0.1:0.1, (c)1.2:0.1:0.3, (d) 1.0:0.1:0.5 and (e) 0.5:0.1:1.0, with a polyether sulfone (PES) membrane serving as a support membrane and n-hexane organic solution containing 0.1 (w/v) % of trimesoyl chloride (TMC).

The functional groups of the GO-TFC membranes were analysed using FTIR spectra, as presented in Figure 2. Characteristic peaks were observed at 1,450 cm^{-1} (C=O stretching), 1,547 cm^{-1} (amide II: C–N stretching vibration and N–H bending), 1,610 cm^{-1} (N–H stretching vibration) and 1,660 cm^{-1} (amide I: C=O stretching vibration of amide), confirming the formation of amide functional groups and the polyamide (PA) layer.[17] The broad peak at 3,367 cm^{-1} corresponds to the O–H group of GO, with intensity increasing at higher GO loadings.[18,19] The incorporation of these functional groups enhances membrane hydrophilicity, thereby improving water permeability and mitigating fouling. Furthermore, the improved surface chemistry facilitates stronger interactions between the membrane and amine molecules (MEA, DEA, TEA), resulting in higher rejection rates, particularly for larger and more hydrophilic amines such as TEA.[20] Figure 2 confirms successful incorporation of GO and formation of amide functional groups in the polyamide selective layer.

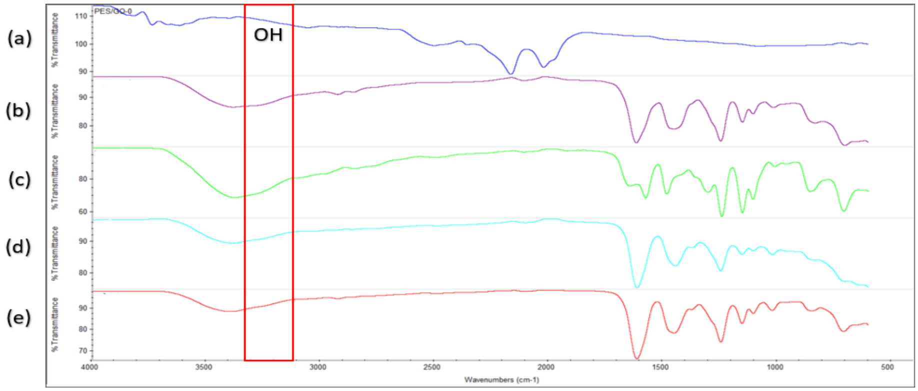


Figure 2 Polyether sulfone (PES) membrane as a support membrane and n-hexane organic solution with 0.1 (w/v) % TMC as functional groups for PPA/GO membranes in the following ratios: (a) 1.5:0.1:0.0, (b) 1.4:0.1:0.1, (c) 1.2:0.1:0.3, (d) 1.0:0.1:0.5 and (e) 0.5:0.1:1.0.

3.2 Membrane Porosity and Mean Pore Radius Measurement

Porosity and mean pore radius of the fabricated membranes are presented in Figures 3 and 4. As shown in Figure 3, membranes with ratios of 1.4:0.1:0.1, 1.2:0.1:0.3 and 1.0:0.1:0.5 exhibited higher porosity compared to the control membrane (1.5:0.1:0.0). With increasing GO concentration from 0.1–0.5 wt.%, porosity increased markedly from 21.21%–84.85%. However, further increasing GO to 1 wt.% led to a slight decrease in porosity to 72.12%. The highest porosity (84.85%) was observed in the membrane with a ratio of 1.0:0.1:0.5, representing a 63.63% increase compared to the bare membrane.

The rise in porosity with GO incorporation can be attributed to the phase separation process. Due to its hydrophilicity, GO enhances thermodynamic instability during demixing, accelerating solvent–non-solvent exchange and thereby increasing porosity.[21,22] However, dope solution viscosity is another critical factor: higher viscosity slows down demixing, resulting in reduced porosity.[21,22] In this study, increasing GO content raised solution viscosity, and at 1 wt.% the viscosity effect outweighed the hydrophilicity effect, limiting porosity enhancement.[23]

As shown in Figure 4, the mean pore radius increased from 0.0375 μm–0.0853 μm as GO content rose from 0 wt.% to 0.3 wt.%. The membrane with a ratio of 1.2:0.1:0.3 exhibited the largest mean pore radius, 2.27 times that of the bare membrane. At 0.5 wt.% GO, the mean pore size decreased to 0.0484 μm but rose again with further addition up to 1 wt.%. This trend differs from earlier findings by Vatanpour et al., where pore size initially increased but later decreased with higher GO due to viscosity effects.[21]

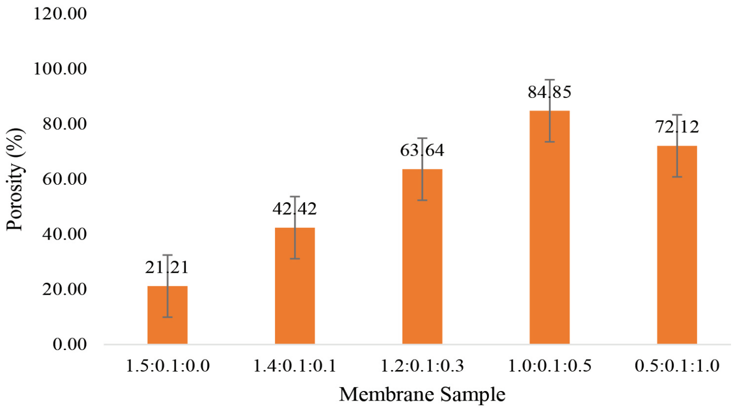


Figure 3: Porosity of membranes.

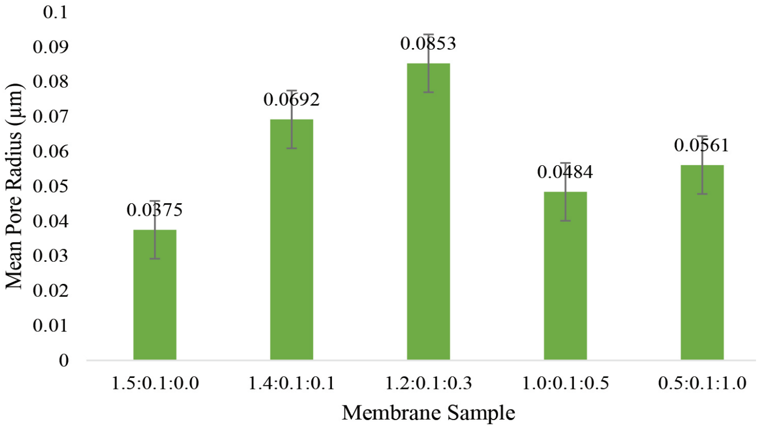


Figure 4: Mean pore radius of membranes.

Table 4 shows the summary of the membrane sample's mean pore radius and porosity values.

Table 4: The fabricated membranes' porosity and pore diameter

Membrane sample	GO (wt.%)	Porosity (%)	Mean pore radius, (μm)
1.5:0.1:0.0	0.0	21.21	0.0375
1.4:0.1:0.1	0.1	42.42	0.0692
1.2:0.1:0.3	0.3	63.64	0.0853
1.0:0.1:0.5	0.5	84.85	0.0484
0.5:0.1:1.0	1.0	72.12	0.0561

3.3 Performances of GO-TFC membrane

3.3.1 PWP

Figure 5 shows the effect of GO on PWP. As GO concentration increased, the PWP rose significantly. This improvement can be attributed to the enhanced hydrophilicity, increased free volume and larger effective mean pore size of the membrane induced by GO incorporation.[24,25]

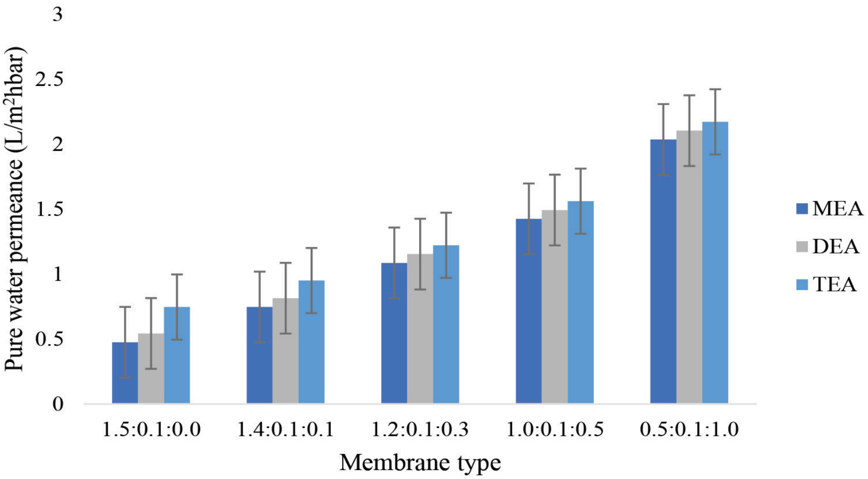


Figure 5: Pure water permeance for varies of GO vs time.

3.3.2 Water Flux

Figure 6 shows that water flux increased from membrane PES/GO-0.1 to PES/GO-0.5. This improvement is primarily attributed to the enhanced hydrophilicity imparted by GO, which allows more water molecules to be attracted and transported across the membrane.[26,27] However, increasing GO concentration also raised the viscosity of the dope solution, which reduced porosity and mean pore size but still resulted in improved water flux.[28–30] Moreover, operating time was observed to influence water flux: although a slight decline occurred over time due to particle accumulation on the membrane surface, the overall reduction remained minimal.

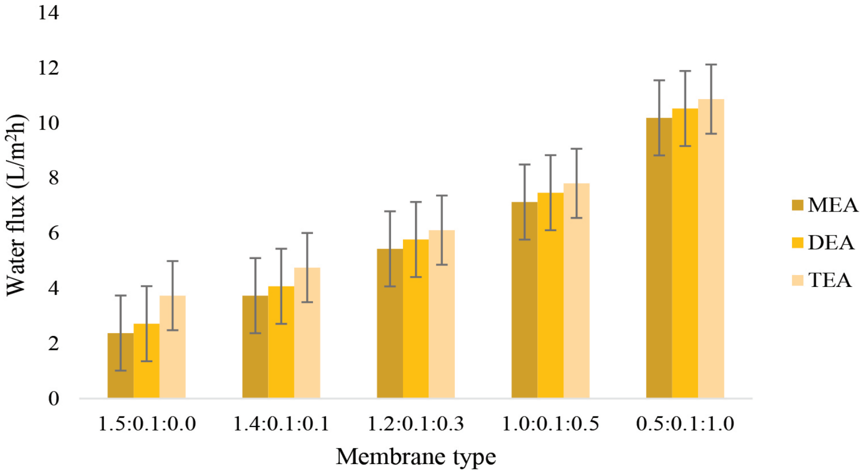


Figure 6: Pure water flux of different GO-TFC membrane at different time.

3.3.3 AMINE REJECTION

Figure 7 presents the effect of GO concentration on the rejection of MEA, DEA and TEA. Clear differences were observed among the three amines, with the PES/GO-0.1 membrane showing the highest rejection performance. The rejection rates were 33.80% for MEA, 41.15% for DEA and 53.28% for TEA. This indicates that the enhanced hydrophilicity of the PES/GO-0.1 membrane contributed to higher rejection efficiency. Furthermore, the presence of GO is suggested to have influenced membrane pore structure, promoting more effective separation of the amines.

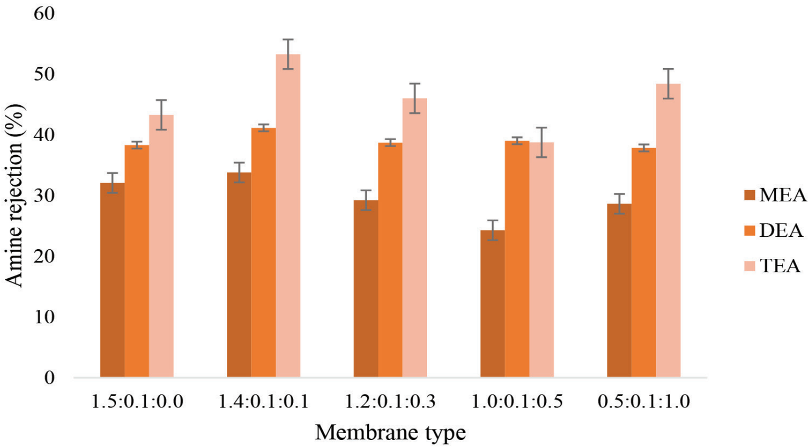


Figure 7: Amine rejection for each ratio of GO-TFC membrane.

Table 5 presents a comparative study of various GO-based TFC membranes for pollutant removal alongside the current work. The analysis indicates that incorporating GO into the PA layer significantly enhances pure water permeance while simultaneously improving pollutant rejection efficiency.

Table 5: Comparative study of GO-based TFC membranes for pollutant removal

Membrane	GO concentration	Pure water permeance ($Lm^2h^{-1}bar$)	Rejection efficiency (%)	Ref.
PSf/MGO	0.225 wt.%	31.80	Congo red = 98.4%	31
PES/GO	0.2 wt.%	7.40	Acetaminophen = 97.7% Diclofenac = 99.13% Ibuprofen = 95.78%	17
PES/GO	0.008 wt.%	34.30	Benzene = 97% Phenol = 84% Toluene = 91%	32
PES/GO-TFC	0.5 wt.%	10.86	MEA = 33.79% DEA = 41.15% TEA = 53.28%	This study

4. CONCLUSION

The comparative study on the separation of MEA, DEA, and TEA from wastewater using TFC membranes demonstrated their effectiveness for selective separation, with performance varying according to amine type. TEA showed the highest rejection rate with 53.28%, followed by DEA with 41.15% and MEA with 33.79%, owing to its larger molecular size and greater hydrophilicity, which enhanced its interaction with the membrane. Operational factors, including amine concentration, amine type, inlet pressure and membrane type, significantly influenced separation efficiency and permeate flux. While higher pressure and temperature improved flux and rejection, the benefits levelled off at elevated values. Conversely, higher feed concentrations reduced flux, particularly for MEA. These findings suggest that although all three amines can be separated by TFC membranes, fouling intensity and performance stability are closely linked to amine properties. In conclusion, TFC membranes present a promising and efficient approach for removing amines from wastewater, with TEA demonstrating the most favourable outcomes. This study provides valuable insights for optimising membrane-based treatment of amine-rich industrial effluents and underscores the importance of tailoring operational conditions to enhance both efficiency and membrane lifespan. Further research should focus on membrane modifications and operational strategies to reduce fouling and improve treatment of more challenging wastewater streams.

5. ACKNOWLEDGEMENTS

The authors would like to express their gratitude to Universiti Malaysia Pahang Al- Sultan Abdullah for supporting this work under University Matching Grant (RDU223204).

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