Reaktor

p-ISSN 0852 – 0798
e-ISSN 2407 – 5973

Website: http://ejournal.undip.ac.id/index.php/reaktor/

Reaktor, Vol. 24 No. 1, April Year 2024, pp. 28-35

Gravity-Based PVC/PVP/SiO_2 Membrane for River Water Treatment

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(Received: 12 August 2024; Accepted: 1 October 2024; Published: 10 October 2024)

Abstract

In this study, ultrafiltration gravity-driven membranes (UF-GDM) were fabricated by blending PVC (12-14% wt.%), PVP (0-5% wt.%), and SiO_2 (1-3% wt.%) in dimethyl acetamide (DMAc) as a solvent. The formulation with a PVC/PVP/SiO_2 ratio of 12/5/1 achieved the lowest water contact angle of 55° and a permeate flux of 34 $L.m^{-2}.h^{-1}$. However, due to its high porosity (84%), this membrane exhibited a higher susceptibility to fouling, resulting in a 21% flux decline after 5 hours of operation. In contrast, the formulation with a PVC/PVP/SiO_2 ratio of 14/2.5/1 demonstrated higher contaminant removal, reducing turbidity by over 99.9%, TDS by 47-51%, conductivity by 96%, and organic matter by 97.5%. Despite its lower permeate flux of 8 $L.m^{-2}.h^{-1}$, attributed to its tighter pore structure, this formulation showed a 20% flux decline over 5 hours. The optimal performance was observed in the PVC/PVP/SiO_2 composition of 12/2.5/1, which sustained a stable permeate flux of 37 $L.m^{-2}.h^{-1}$ with only a 9% decline over 5 hours operation. This membrane formulation effectively reduced TDS, turbidity, conductivity and organic matter by 38%, 99.9%, 96%, and 98%, respectively. These results highlight the potential of the 12/2.5/1 formulation for developing environmentally friendly and energy-efficient membrane for clean water supply applications.

Keywords: Energy efficiency; clean water; gravity-driven membrane; PVC membrane; ultrafiltration.

How to Cite This Article: Aryanti, P.T.P, Herlambang, N.A., Putri, D.H., Apriyani, S., and Putri, T.A, (2024), Gravity-Based PVC/PVP/SiO2 Membrane for River Water Treatment, Reaktor, 24 (1), 28 - 35, https://doi.org/10.14710/reaktor. 24. 1. 28 - 35

INTRODUCTION

Membrane technology, particularly ultrafiltration (UF) membranes, has proven to be highly effective in converting raw water into potable water. The latter can be achieved upon removing its containing particulates, colloids, microorganisms, bacteria, and viruses due to their small pore size, which ranges from 0.01 to 0.1 µm (Khan et al., 2022). However, the energy consumption required to operate electric pumps in UF processes is critical, especially in large-scale applications where significant energy input is necessary to maintain pump-driven pressure. An alternative approach involves utilizing elevation differences to generate pressure within the membrane system, a method known as Gravity Driven Membrane (GDM) (Pronk et al., 2019). Due to their low energy requirements, UF-GDM membranes have been successfully applied for community water supply from diverse sources such as river water, wells, and rainwater. They are also used as a pre-treatment stage in seawater desalination processes (Wu et al., 2024). This approach demonstrates substantial potential for sustainable and energy-efficient water treatment applications.

One of the limitations of GDM systems is their low permeate flux, ranging from 2 to 10 L/m².h, particularly in large-scale applications. Enhancing membrane hydrophilicity and effective fouling control are crucial for advancing the performance of GDM membranes. Jiang et al. (2019) developed an amphiphilic multi-arm polymer, propylene glycol-silane-poly(ethylene glycol) (PPG-Si-PEG), which was incorporated into polyvinylidene fluoride (PVDF) using a phase separation method. The water flux of the resulting PVDF/PPG-Si-PEG membrane increased significantly from 4.6 to 12 $L.m^{-2}.h^{-1}$, with a flux recovery of 99.4% after membrane cleaning. Lin et al. (2022) compared the performance of GDM-PVDF and polyvinyl chloride (PVC) membranes, finding that although the PVDF membrane initially exhibited a permeate flux 10 (ten) times higher than the PVC membrane, the latter demonstrated more excellent long-term stability due to its inherently higher hydrophilicity.

Aryanti et al. (2023) further modified a UF PVC GDM membrane by incorporating polyethylene glycol (PEG400) and ZnO particles, achieving turbidity removal of 99.9% and a permeate flux of 11 $L.m^2.h^{-1}$ at 12% PVC, 5% PEG400, and 1% ZnO. These findings suggested that PVC membranes show considerable potential for further development in UF-GDM applications to achieve higher flux and improved performance. Further research is needed to address challenges, such as low permeate flux and fouling resistance to optimize the functionality of these membranes.

This study focused on enhancing PVC-based ultrafiltration gravity-driven membranes (UF-GDM) by incorporating polyvinylpyrrolidone (PVP) and silica nanoparticles (SiO_2-NP) to improve membrane hydrophilicity and flux performance. The research employed a dual modification strategy to combine the porosity-enhancing properties of PVP with the hydrophilicity enhancement provided by SiO_2 nanoparticles, aiming to improve the performance of PVC membranes in GDM applications. This strategy aimed to enhance both water permeability and resistance to fouling, which are crucial for advancing the efficiency of UF-GDM systems. The study investigated the combined effects of PVP and SiO_2-NP concentrations at various PVC levels on membrane morphology, characteristics, and performance under different operating pressures. Additionally, the research integrated an activated carbon layer onto the UF-GDM membrane to improve product water quality without compromising flux stability. The objectives of this research were to systematically evaluate the impact of PVP and SiO_2-NP concentrations on membrane properties and performance, assess the effectiveness of the activated carbon layer in enhancing water quality, and develop an energy-efficient and environmentally friendly UF-GDM membrane suitable for sustainable clean water production.

METHOD

Research Materials

This research used PVC resin (FJ-60R), produced by PT Asahimas Chemical, while dimethylacetamide (DMAc) of 99.9% purity was supplied from Shandong FanTai Fine Chemical Biotechnology Co. Ltd. Meanwhile, PVP K-90 (molecular weight 111.14 $g.mol^{-1}$) and SiO_2-NP were obtained from local suppliers. The river water was taken from the area in the vicinity of Universitas Jenderal Achmad Yani Cimahi.

Membrane Preparation

The UF-GDM membranes were prepared by dissolving PVC (12 and 14 wt.%), PVP (0-5 wt.%), and SiO2-NP (1-3 wt.%) in dimethylacetamide (DMAc) solvent. The solution was stirred for 48 hours until it was homogeneous and left to stand until the air bubbles disappeared. The membrane solution was coated onto flat glass with a 100-150 µm thickness and then submerged in a coagulation bath filled with water for 24 hours (refer to Figure 1a). Subsequently, the membrane was cut into 10 cm diameter rounds and placed in the GDM module for permeate flux and rejection testing.

SEM and FTIR Test Analysis

Prior to analysis, the membrane samples were dried in a desiccator for at least 24 hours and then cut into 1x2 cm pieces for testing. The membrane morphology was observed using SEM photographs (JEOL JSM-6510A). An FTIR spectrophotometer (model DRS-8000, Shimadzu, Japan) was utilized to identify the functional groups present in the resulting UF membrane structure.

Porosity and Water Contact Angle Test Procedure

The flat-sheet membranes were weighed in wet (m_1) and dry (m_2) states. The porosity of the membrane was determined using equation (1), as follows:

$$\varepsilon = \frac{(m_1 - m_2)/\rho_w}{\frac{m_1 - m_2}{\rho_w} + m_2/\rho_m} x 100\% \qquad (1)$$

where, ρ_m ($g{\cdot}cm^{-3}$) and ρ_w ($g{\cdot}cm^{-3}$) represent the polymer density (PVC) and water, respectively. Whereas m_1 and m_2 represent the weight of wet membrane and dry membrane, respectively.

The contact angle determines the hydrophilicity of the membrane surface. Membrane samples were cut with a size of 2×2 cm, and then dripped with water, Pictures were taken using a contact angle tool. The contact angle test results were obtained in the form of angle magnitude data from each sample (**Figure 1b**).

Flux, Permeability, Rejection and Contaminant Reduction Testing

The round-cut membranes (10 cm diameter) were placed in a module connected to a 1 m vertical pipe. Feed water was filled into the pipe tube to a height of 80 cm. The permeate flux was measured over 5 hours of operating time, with product water sampling every 1 hour (Figure 1c). The permeate flux was calculated using the following equation:

$$J = \frac{V}{A \times t} \qquad (2)$$

where *J* is the permeate flux (L.m^2.h^{-1}) described as the volume of permeate (*V*, Liter) passing through a membrane having an area *A* (m^2) at a given time t (h). Meanwhile, the selectivity of the membrane is expressed through a value of the coefficient of rejection (%*R*), which is calculated by equation (3) below:

$$R = 1 - \frac{Cp}{Cf} \times 100\% \quad (3)$$

The variables c_p and c_f are the contaminant concentrations in permeate and feed, respectively. The organic compound content was measured using UV-Vis at a wavelength of 254 nm. Equation (3) was also used for color, turbidity, and conductivity rejection measurements.

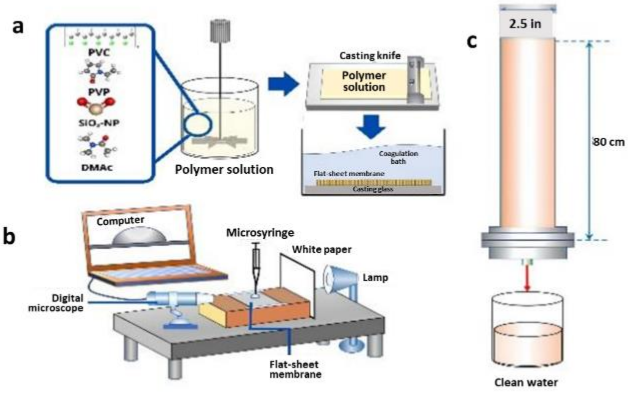


Figure 1. Experimental set-up for (a) UF membrane preparation, (b) water contact angle measurement, and (c) permeate flux testing of UF-GDM.

RESULTS AND DISCUSSION

Effect of Polymer Solution Composition on Membrane Morphology

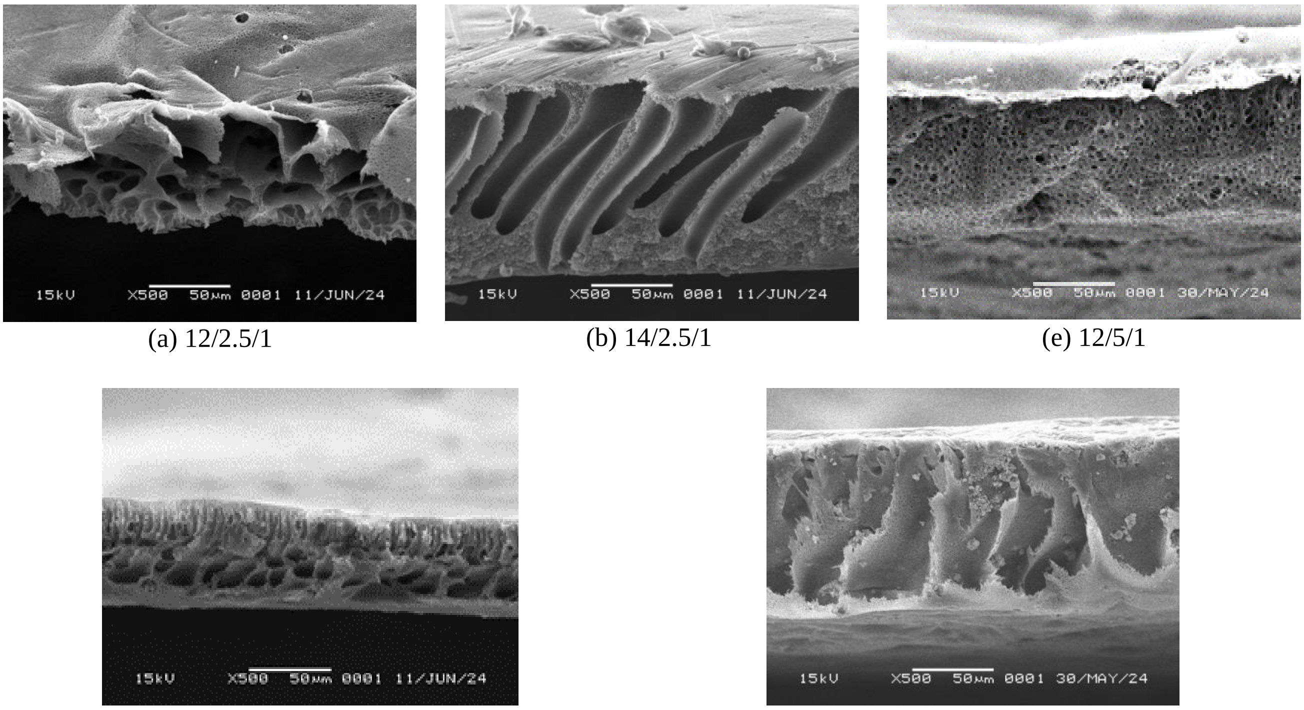
The effect of polymer solution concentration on UF membrane structure was investigated using Scanning Electron Microscopy (SEM) at 500× magnification (Figure 2). The analysis revealed that variations in PVC concentration (12% and 14% wt.%) led to notable differences in pore morphology. At 14 wt.% PVC, the membrane exhibited a finger-like structure, whereas at 12 wt.%, the pores were predominantly sponge-like with a larger size (Figures 2a and 2b). This structural variation was attributed to the higher viscosity of the 14 wt.% PVC solution, which significantly influenced the membrane formation process. The increase of resistance to pore formation at 14 wt.% suggests that polymer concentration plays a critical role in determining membrane morphology. Furthermore, the increased porosity observed in membranes with 14 wt.% PVC was attributed to the elevated viscosity, which restricted the expansion of pore nuclei during the phase inversion process. These findings highlight the importance of precise regulation of polymer concentration in the development of PVC-PVP-SiO$_2$-NP-based UF-GDM membranes to optimize membrane structure and enhance performance.

The effect of PVP concentration on membrane morphology is illustrated in Figures 2a, 2d, and 2e. An increase in PVP concentration resulted in the formation of smaller pores, which can be attributed to the elevated solution viscosity that inhibits pore growth and facilitates the development of a sponge-like structure. Figure 2e indicates that at a 5% PVP concentration, surface defects were observed, likely due to the excessive accumulation of the polymer solution on the membrane surface. This phenomenon arises due to the elevated PVP concentration, which decreases the solution penetration rate into the membrane matrix during the phase inversion process, thereby impacting the overall membrane formation and structure (Ho & Su, 2022). Therefore, controlling the concentration of PVP also plays an important role in producing optimal membrane morphology and avoiding surface defects that can affect the overall membrane performance.

Figures 2a and 2c display the effect of SiO$_2$-NP concentration on the membrane pore structure. At 1 wt.% SiO$_2$-NP concentration, the membrane exhibits larger and more evenly distributed pores compared to the 3 wt.% concentration. This finding highlights the significance of addressing particle agglomeration and the phase separation process in the solution containing 3 wt.% SiO$_2$-NP during pore formation. Furthermore, understanding the pore-forming properties of SiO$_2$-NP is crucial, as it contributes to the creation of pores of varying sizes, with implications for the overall membrane Performance (Khan et al., 2020)

Chemical Characteristics of Membranes

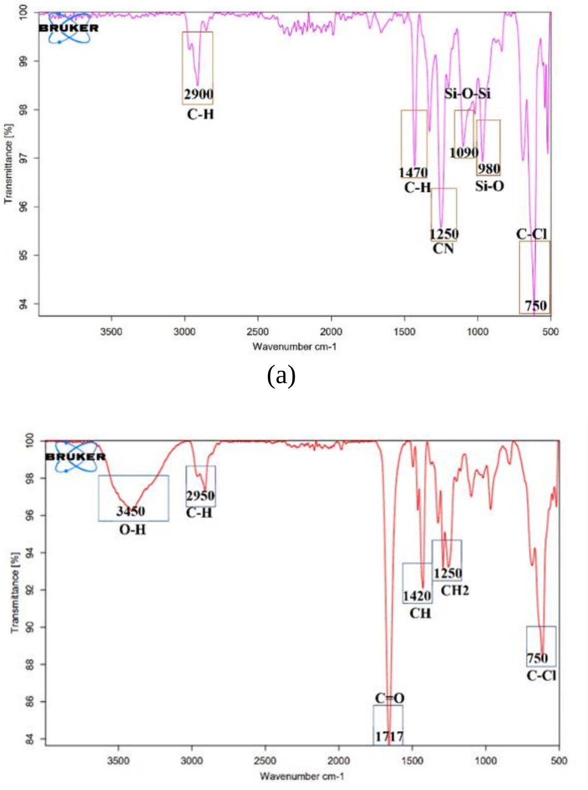
Figure 3 represents the chemical properties of the membrane observed through FTIR (Fourier Transform Infra-Red) to identify the compounds and functional groups present in the membrane structure. The FTIR spectrum of PVC is shown at peaks with wavelengths of 1470 cm^{-1} (Figure 3a) and 1420 cm^{-1} (Figure 3b) which are the result of vibrational transitions in the C-H bond. Meanwhile, the peak at 750 cm^{-1} wavelength appears due to the stretching of the C-Cl bond in CH-Cl in PVC. The peaks occurring at 2900-2950 cm^{-1} are the result of C-H bond stretching in DMAc (Figure 3a) and the broad peak at 3450 cm^{-1} designates



(a) 12/2.5/1 (b) 14/2.5/1 (e) 12/5/1

(c) 12/2.5/3 (d) 12/0/1

Figure 2. SEM of the cross-section of UF membrane at various concentration ratio of PVC/PVP/SiO_2-NP: (a) 12/2.5/1, (b) 14/2.5/1, (c) 12/2.5/3, (d) 12/0/1, and (e) 12/5/1



(a)

(b)

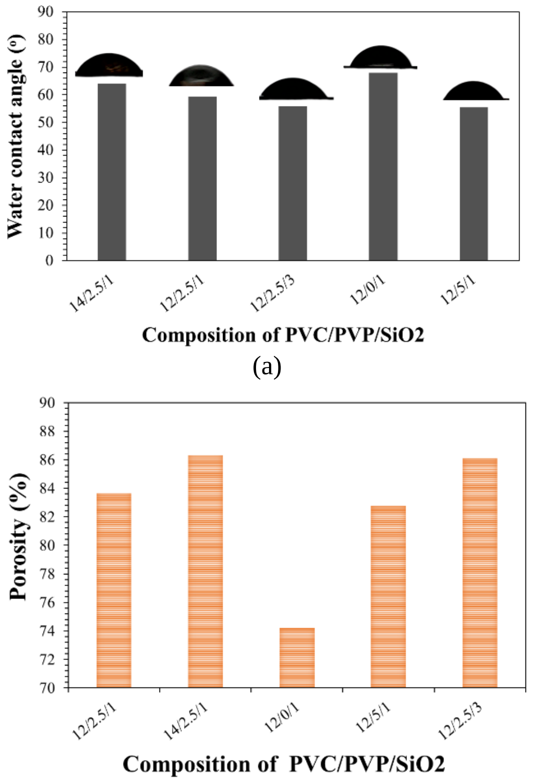
Figure 3. FTIR analysis results on membranes with concentration: (a) PVC/PVP/SiO_2-NP 12/0/1 and (b) PVC/PVP/SiO_2-NP 12/2.5/0.

the presence of hydroxyl groups followed by a frequency peak at 1420 cm^{-1} (Figure 3b).The peak at 1090 cm^{-1} indicates the presence of Si-O-Si bonds, the peak at 900 cm^{-1} conforms the presence of silicate ions (SiO_3^{-2}) derived from SiO_2-NP (Figure 3a), and the peak at 1250 cm^{-1} is identified as a result of the C-H_2 bond and the peak at 1717 cm^{-1} indicates the C=O bond in PVP.

Effect of Polymer Solution Composition on Porosity and Hydrophilicity of Membrane

Figure 4a presents the changes in membrane contact angle, which serve as an indicator of hydrophilicity, following the incorporation of PVC, PVP, and SiO_2-NP. An increase in PVC concentration from 12 wt.% to 14 wt.% resulted in a rise in the contact angle from 59° to 64°, reflecting the hydrophobic nature of PVC, which reduces membrane hydrophilicity.

In contrast, the addition of PVP and SiO_2-NP enhanced membrane hydrophilicity. While increasing the SiO_2-NP concentration from 1 to 3 wt.% led to a negligible reduction in contact angle. This may be attributed to the positioning of SiO_2-NP particles beneath the polymer surface, limiting their direct interaction with water. Notably, the incorporation of PVP at concentrations ranging from 0 wt.% to 2.5 wt.% significantly reduced the contact angle from 68° to 59°, underscoring the prominent role of PVP in enhancing membrane hydrophilicity.



(a)

(b)

Figure 4. Effect of membrane composition on (a) hydrophilicity (contact angle) and (b) porosity

The composition of the membrane solution was found to significantly affect membrane porosity when varying concentrations of PVC, PVP, and SiO_2-NP were used (Figure 4b). Across all membrane variations, porosity ranged from 74% to 86%. At lower PVC concentrations (12 wt.%), the membrane exhibited larger pores, driven by increased membrane thickness due to lower solution viscosity, which promoted pore growth. Increasing the PVC concentration to 14 wt.% further enhanced membrane porosity, reaching 86%. This increase in viscosity hindered the growth of pore nuclei, allowing water to permeate deeper into the membrane solution (Ashfaq et al., 2022). Consequently, pore growth extended to the base of the membrane, contributing to a significant increase in porosity.

The addition of PVP to the membrane solution had a meaningful impact on membrane porosity. Notably, at a PVP concentration of 2.5 wt.%, there was a significant increase in porosity compared to the membrane without PVP. This increase can be attributed to the higher viscosity of the solution, which promotes the formation of smaller, more numerous pores, ultimately enhancing overall membrane porosity. However, further increasing the PVP concentration from 2.5 wt.% to 5 wt.% resulted in a decrease in porosity. This decrease is a result of the higher viscosity at this concentration, which hinders water penetration into the polymer solution during the phase inversion process, thus restricting pore formation and reducing membrane porosity.

Furthermore, the increase in SiO_2-NP concentration resulted in higher surface porosity. The incorporation of SiO_2-NP disrupts the uniformity of the solution, acting as nucleation sites for pore formation (Khan et al., 2020). Consequently, a greater concentration of SiO_2-NPs contributes to the development of more pores, significantly enhancing membrane porosity.

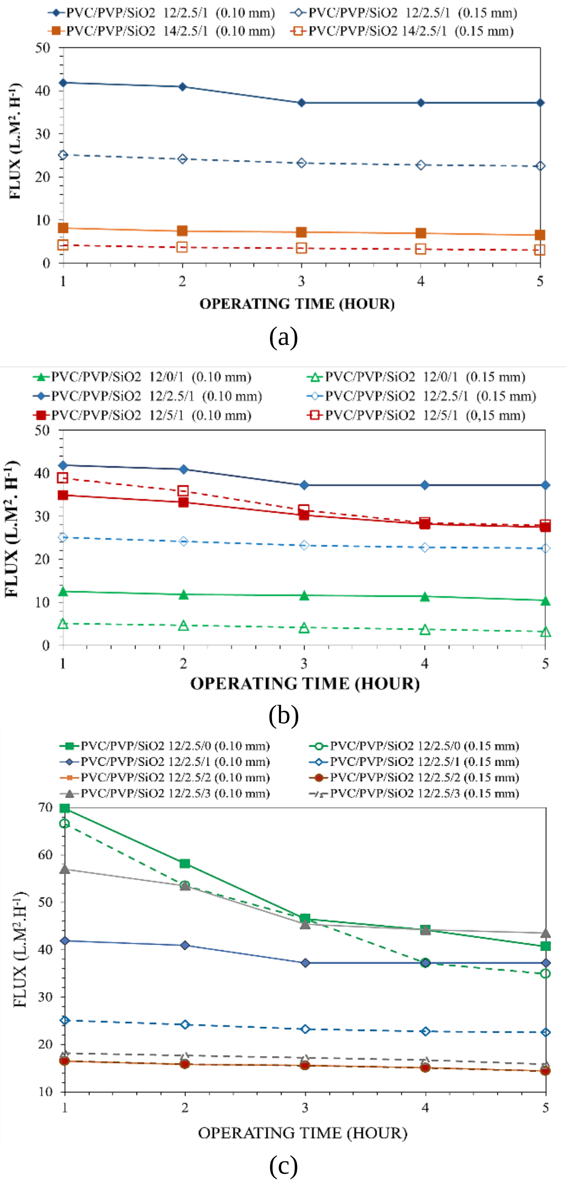
Effect of Polymer Solution Composition on Permeate Flux

Figure 5a illustrates the permeate flux of membranes with varying concentrations of PVC, PVP, and SiO_2-NP. The composition of 12 wt.% PVC, 2.5 wt.% PVP, and 1 wt.% SiO2-NP achieved the highest stable flux of 37 L/m^2·h. This is attributed to the more uniformly distributed pore structure across the membrane thickness, which reduces hydraulic resistance during water permeation. A uniform pore distribution along the membrane thickness minimizes the resistance encountered by water (Van et al., 2010) and also helps prevent non-uniform fouling on the membrane surface, maintaining stable operational conditions and reducing pressure and flux fluctuations (Hung et al., 2022).

Figure 5a also shows that increasing the PVC concentration from 12 wt.% to 14 wt.% resulted in a significant 80.5% reduction in flux (from 41.9 to 8.1 L.m^2·h^{-1}). This decline is due to the hydrophobic nature of PVC, which impedes water penetration through the membrane. Furthermore, the increased membrane thickness exacerbates this effect, as the added thickness raises the resistance to water flow. For example, increasing membrane thickness from 100 µm to 150 µm led to a flux decrease from 41 L.m^2·h^{-1} to 25 L.m^2·h^{-1}, representing a 39% reduction.

The effect of PVP concentration on permeate flux is shown in Figure 5b. The addition of 2.5 wt.% PVP resulted in a membrane with a higher permeate flux (41 L.m^2·h^{-1}) compared to a 5 wt.% PVP concentration (34 L.m^2·h^{-1}). The greater porosity in the membrane with 2.5 wt.% PVP contributed to the higher flux. Interestingly, increasing membrane thickness had a minimal effect on flux, as an increase from 100 µm to 150 µm only led to a slight rise in flux from 34 L.m^2·h^{-1} to 38 L.m^2·h^{-1} at 5 wt.% PVP. This suggests that PVP concentration plays a more dominant role in enhancing membrane hydrophilicity than membrane thickness.

The effect of SiO_2-NP concentration on the permeate flux of the membrane is shown in Figure 5c. Membranes without the addition of SiO_2-NP produced a higher permeate flux (69 L.m^2.h^{-1}) compared to membranes containing 1 wt% of SiO_2-NP (41 L.m^2.h^{-1}). The addition of SiO_2-NP may fill the membrane pores which has an impact on reducing the effective pore size of the membrane, thus providing water resistance through the membrane pores (Lim, 2024) Therefore, the membrane flux without SiO_2-NP becomes greater. However, the resulting flux is unstable and tends to decrease over time. Therefore, although the flux without SiO_2-NP is initially higher, its use is less efficient for long-term applications.



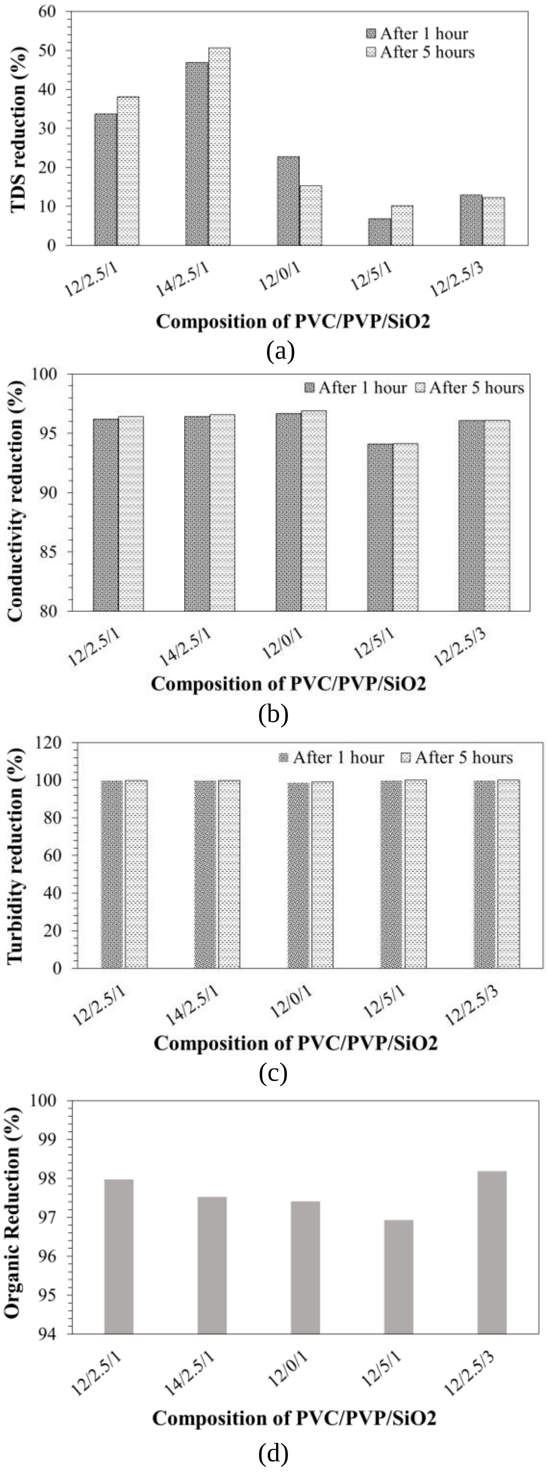
(a)

(b)

(c)

Figure 5. The influence of (a) PVC, (b) PVP, and (c) SiO_2-NP concentrations on permeate flux during 5 hours of gravity-driven ultrafiltration

The incorporation of SiO_2-NP at a concentration of 2 wt.% led to agglomeration of the additives, which negatively impacted membrane porosity and led to non-uniform pore distribution. At a concentration of 3% by weight, an increase in water flux was observed, attributed to the dominance of the hydrophilic properties of the SiO_2-NPs. However, when the concentration exceeded 1% by weight, a significant decline in membrane performance was recorded, with a 29% reduction in flux due to particle agglomeration.



(a)

(b)

(c)

(d)

Figure 6. effect of membrane solution composition on rejection performance in different tests: (a) total dissolved solids (TDS), (b) conductivity, (c) turbidity, and (d) organic matter

This agglomeration formed larger agglomeration of particles within the membrane matrix may block the water channels and reducing the efficiency in water transport through the membrane pore.

Effect of Polymer Solution Composition on Contaminant Reduction Efficiency

The impact of membrane solution composition on the reduction efficiency of inorganic substances is illustrated in Figure 6. The membrane composed of 14 wt.% PVC, 2.5 wt.% PVP, and 1 wt.% SiO_2-NP exhibited the highest rejection rate. The concentration of TDS decreased significantly from 294 $mg \cdot L^{-1}$ to 156 $mg \cdot L^{-1}$ after 1 hour of operation (or by 47% reduction), and further to 145 $mg \cdot L^{-1}$ after 5 hours (or by 50% reduction). This decrease was attributed to the higher PVC concentration, which enhanced the hydrophobicity of the membrane, resulting in reduced porosity and pore size. These structural changes facilitated the formation of fouling on the membrane surface, which functions as a secondary filtration layer, thereby improving the contaminant rejection efficiency.

In contrast, the PVC/PVP/SiO_2 composition of 12/5/1 exhibited the lowest rejection efficiency, with only a 10% reduction in TDS (from 294 $mg \cdot L^{-1}$ to 264 $mg \cdot L^{-1}$). In comparison to other membrane compositions, this composition exhibits a relatively lower percentage of conductivity reduction at 94% and a 96% reduction in organic substances. The decrease in reduction is attributed to defects on the membrane surface resulting from the high concentration of PVP in the PVC/PVP/SiO_2 12/5/1 composition. This condition significantly impacts the membrane's ability to reject contaminants.

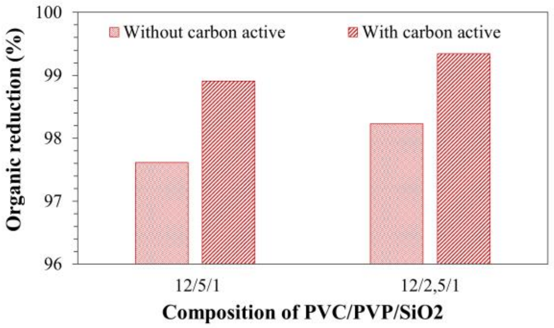


Figure 7. Effect of activated carbon on organic substance rejection by the UF-GDM surface.

To enhance contaminant reduction efficiency, applying activated carbon as a pre-treatment before the water passes through the membrane can be beneficial. This coating has minimal impact on the permeate flux of the membrane but can improve the reduction of organic substances. For instance, it can increase the reduction from 97% to 98% for PVC/PVP/SiO_2 12/5/1 composition, and from 98% to 99% for PVC/PVP/SiO2 12/2.5/1 composition (refer to Figure 7).

CONCLUSION

The development of composite membranes using PVC 60-R, combined with PVP K-90 and SiO_2 nanoparticles, offered valuable insights into enhancing membrane performance. The study revealed that the ratios of PVC, PVP, and SiO2-NP significantly influenced membrane porosity, with the highest porosity of 86% achieved at a composition of 14/2.5/1, and the lowest at 74% for 12/0/1. Furthermore, it was also observed that the PVP concentration affected the contact angle as a measure of surface hydrophilicity, with a minimum contact angle of 55° noted at a 12/5/1 composition, attributed to the hydrophilic properties of PVP.

In terms of permeate flux, the composition of 14/2.5/1 exhibited the lowest flux at 8 $L/m^2 \cdot h$, due to PVC's hydrophobic nature, while a composition of 12/2.5/1 demonstrated a stable flux of 37 $L/m^2 \cdot h$, with only a 9% decline, thanks to uniform pore distribution reducing resistance and fouling. Moreover, the highest contaminant rejection efficiency was achieved with the 14/2.5/1 composition, despite the lower permeate flux. Application of activated carbon on the membrane's surface further improved rejection rates, notably for compositions 12/5/1 and 12/2.5/1, achieving up to 99% efficiency. These findings underscore the potential for developing energy-efficient ultrafiltration (UF) membranes suitable for diverse applications, including emergency situation and rural areas, highlighting the critical role of precise polymer composition tuning.

ACKNOWLEGEMENT

This research was supported by the Student Creativity Program (PKM) from the Directorate of Learning and Student Affairs of Indonesia [Decree No. 2546/E2/DT.01.00/2024, dated April 19, 2024].

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