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ORIGINAL RESEARCH ARTICLE

Morphology and characteristics of polyethersulfone membrane modified with polyethylene glycol hexadecyl ether and nanocarbon

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ABSTRACT

The development of polyethersulfone (PES) membranes to improve membrane performance can be done in various ways; one is by combining two types of additives (organic and inorganic) in one dope polymer solution. In this study, researchers analyzed the effect of combining polyethylene glycol hexadecyl ether (PEG-HE) as an organic additive and nanocarbon as an inorganic additive on the characteristics and performance of PES membranes. The membrane performance test includes analysis of pure water flux and rejection of synthetic fertilizer factory wastewater (Mg^{2+}) with a concentration of 300 ppm. The membrane characterization was carried out by analyzing the morphology of the membrane structure using Scanning Electron Microscopy (SEM), water contact angle (WCA), porosity, and membrane pore size. Ultrafiltration experiment showed that the modified PES membrane with PEG-HE and Nanocarbon had the highest pure water flux. The most significant rejection coefficient of 96.88% was found in an ultrafiltration experiment using pure PES membranes. The characteristic of other membranes will be described in detail in this article.

Keywords: hybrid membrane; nano carbon; polyether sulfone; polyethylene glycol hexadecyl ether

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1. Introduction

Research developments related to membrane technology today are increasingly leading to modifications of fillers or additives[1]. Modifying this additive is intended to improve the performance of the resulting membrane. Improved membrane performance can be evaluated from the results of the performance and characteristics of the membrane, especially after undergoing modification of the membrane with pure polymer. The pure polymer used as a raw material for making membranes can be used in the filtration process to purify water, but problems will arise as long as the membrane is used. The main problem that often occurs in membrane materials is fouling[2]. There are many ways that researchers have done to overcome this fouling problem, one of which is by modifying the polymer used[3], adding additives[4], modifying additives by

hybridization[5], and incorporating additives by blending in dope membrane solutions[6]. Fouling on membranes with polyethersulfone (PES) polymers can quickly occur due to the highly hydrophobic nature of the membrane surface. This high hydrophobic nature is a challenge for researchers to conduct research in the ways mentioned above and in new ways that might be developed in the future. This development aims to reduce the membrane's hydrophobic properties so that the PES membrane material becomes more hydrophilic than the pure PES membrane.

The addition of additives to improve the hydrophilicity of the membrane has been proven effective in increasing the performance of the membrane. The additive used aims to influence the structure of the PES membrane by increasing hydrophilic hydroxyl (OH) groups so that the membrane material can more easily interact with water molecules[7]. The use of additives not only improves membrane performance but also increases the service life of the membrane by reducing fouling. Fouling events and increased hydrophilic properties of the membrane are closely related because the more hydrophilic the membrane material, the more difficult it is for inorganic particles to form bonds with the membrane surface. Inorganic particles tend to efficiently bind to the surface of the membrane because the PES membrane is a material that is very hydrophobic but has good mechanical properties when used during the filtration process[8]. The hydrophobicity of PES membranes is highly undesirable but poses a dilemma because of its good mechanical properties. Therefore, looking for the best conditions in modifying PES membranes by adding additives is more focused so as not to affect other membrane properties.

Polyethylene glycol hexadecyl ether (PEG-HE) and nanocarbon are often added when making PES membranes. Both additives are often used to increase the hydrophilicity of the membrane because of their hydroxyl groups. Many research reports examining the effect of PEG-HE and nanocarbon have reported satisfactory results in improving membrane performance from permeability and selectivity analysis. Lusiana et al. researched by adding PEG in a PES membrane dope solution. Parameters evaluated included porosity, water contact angle (WCA), water permeation, and membrane morphology. The results of porosity and WCA are inversely proportional to the increasing amount of PEG used, where the WCA value is getting smaller (75°–15°), and the porosity value is getting bigger (8.22%–76.59%). This result shows that the influence of PEG is considerable in changing the hydrophobicity of the PES membrane, which is hydrophobic. The morphological results can confirm this result, showing that more pores are formed with increasing PEG. The increasing hydrophilicity of the PES-PEG membrane increases the pure water flux yield due to the increasing number of membrane pores. However, in this study, it was not reported how the results of selectivity in filtering a substance could not be compared with the increase in the yield of pure water flux[9]. Another study by Chan et al. reported the results of using PEG additives at concentrations of 3, 6, and 9 wt%, with PES and dimethylformamide (DMF) concentrations of 20 wt% (fixed concentration) and 77, 74, and 71 wt%, respectively. This study aimed to determine the effect of PEG additives on Cu(II) metal rejection. WCA results show a decrease from 76.61° to 56.14° with increased PEGs. This decrease in WCA value also affects the value of pure water flux, where there is an increase in the results obtained. Besides that, the value is inversely proportional to the rejection of Cu(II) metal because the rejection value obtained gets smaller with the increasing amount of PEG used[10]. As previously mentioned, it is essential to find optimum conditions where high pure water flux values can be obtained, but there is no significant reduction in rejection. Many other studies have modified PEG additives intending to find the optimum conditions for the permeability and rejection relationship, including those by Ma et al., who grafted PEG on PES for gas separation[11], Mokarizadeh and Raisi who compared PES/PEG and PES/PVP (polyvinylpyrrolidone) blending methods for industrial wastewater purification[12], and Prince et al. perform thermal grafting on PEG and Ag (silver nanoparticle) which are then mixed in the PES dope solution[13].

Nanocarbon also can increase the hydrophilicity of the membrane. Khan et al. researched using oxidized

nanocarbon black (ONC) to make ultrafiltration membranes. Using nanocarbon is expected to improve the performance of the membrane by suppressing fouling and increasing hydrophilicity. ONC was applied to polysulfone (PS) membranes with concentrations of 0.5, 1, 1.5, and 2 wt%. The membrane characteristics for hydrophilicity were evaluated by analyzing the WCA value, which showed that the results were insignificant. However, it was confirmed that the WCA value decreased from 84.5° to 77.5° as the ONC was used. Water permeation flux showed an optimum yield of 307 $Lm^{-2}h^{-1}$ at 1.0 wt% ONC which was 63% greater than pure PS membrane. However, the addition of ONC greater than 1.0 wt% to the dope solution increased the viscosity of the solution and affected the pore size so that the flux decreased[14]. In addition to the water separation process, there are also many applications of nanocarbon on non-reflective substrates for nanophotonic materials, one of which was in a study conducted by Zhigunov et al. using single-walled carbon nanotubes (SWCNT). This study reported that using SWCNT produced a membrane with better optical contrast than a pristine membrane. The high transparency yield, low reflection rate, and small thickness make SWCNT membranes potential for applications in nanophotonics, bioimaging, and the science of synchrotron radiation[15].

From the various potentials for developing PES membrane materials with the addition of organic (PEG-HE) and inorganic (nanocarbon) additives, these two materials can be used as performance-supporting materials for PES membranes. The application of these additives is still limited to using each additive, such as PEG-HE in a PES membrane matrix and nanocarbon in a PES membrane matrix. Regarding developing PES membrane modification research, the results of blending organic and inorganic additives in one PES membrane matrix have yet to be reported. Therefore, in this study, a combination of the two types of additives was carried out using the blending method in a PES membrane dope solution to determine the characteristics and performance of the resulting membrane. Several membrane characteristics and performance parameters were evaluated in this study, including morphology, hydrophilicity, porosity and pore size, permeability, and rejection of magnesium (Mg^{2+}). As a matter of convenience, the nomenclature list has shown in **Table 1**.

Table 1. Nomenclature list.

Nomenclature			
PES	Polyethersulfone	A	Membrane surface area
DMF	Dimethylformamide	L	Membrane thickness
PEG-HE	Polyethylene Glycol Hexadecyl Ether	rm	Pore size
Nc	Nanocarbon	η	Water viscosity
SEM	Scanning Electron Microscopy	Q	Permeate flow rate
WCA	Water Contact Angle	J	Pure water flux
ε	Porosity	C_P	Permeate concentration
ρ	Water density	C_F	Feed concentration

2. Experimental method

2.1. Materials

The primary materials in this study, such as polyethersulfone (PES Mw: 65 kDa, Ultrason E6020 P, BASF, Ludwigshafen, Germany) as the polymer, Dimethylformamide (DMF, Merck, Taufkirchen, Germany) as the solvent, polyethylene glycol hexadecyl ether (PEG-HE Mw: 4 kDa, Merck, Darmstadt, Germany) as the organic additive and Nanocarbon (Sw-Cnt, 0.78 nm, Sigma Aldrich, St. Louis, MI, USA) as the inorganic additive.

2.2. Membrane fabrication

The dope solution was made from 16 wt% of PES, then 5 wt% PEG-HE and 0.05 wt% Nanocarbon were added, and the last added was the solvent (DMF) with a total weight of the mixture is 20 g. The detailed composition of the dope solution can be seen in **Table 2**. After the solution was homogenous, it should keep for one night to remove the bubble. The dope solution was ready to cast on a flat glass using a membrane applicator (thickness of 300 μm), followed by immersion into a bath containing distilled water after all the bubbles were removed.

Table 2. Composition of the dope solution by weight.

PES (%)	PEG-HE (%)	Nanocarbon (%)	DMF (%)	Membrane
16	0	0	84	P
16	5	0	79	PB
16	0	0.05	83.95	PNc
16	5	0.05	78.95	PBNc

2.3. Membrane characterization and performance

2.3.1. Membrane morphology

The morphology of the membranes was evaluated using scanning electron microscopy (SEM, FEI Quanta Feg 250 model). The samples were immersed in liquid nitrogen to create the cross-section specimen, which was freeze-dried at a temperature of −55 °C before being fractured to create a clean cut. The surface analysis was prepared by cutting the membranes into small sizes (0.5 cm × 0.5 cm) and putting them on the metal holder.

2.3.2. Water contact angle (WCA)

The membrane's hydrophilicity was evaluated using water contact angle analysis. Measurement was conducted ten times in different ten points on the membrane's surface, and calculated the average value and standard deviation. The equipment type of this analysis was the KSV Attension Theta model, Turkey.

2.3.3. Porosity and pore size

Porosity and pore size tests were carried out by comparing the initial and final weights of the membranes after being dried in an oven at 60 °C for 3 h, with 3 repetitions for each membrane. The porosity of the membrane can be calculated using the following Equation (1), where W_1 is membrane wet weight (g), W_2 membrane is dry weight (g), ρ is water density (g/cm^2), A is membrane surface area (cm^2) and l is membrane thickness (cm).

$$\varepsilon\ (\%) = \frac{W_1 - W_2}{\rho . A . l} \tag{1}$$

The pore size of the membrane can be calculated using Equation (2), where ε is membrane porosity, η is water viscosity, Q is permeate flow rate (cm^3/s), and ΔP is pressure difference (Pa)[16].

$$rm\ (\mu m) = \sqrt{\frac{(2.9 - 1.75\varepsilon) \times 8\eta l Q}{\varepsilon \times A \times \Delta P}} \tag{2}$$

2.3.4. Membrane permeation performance

An effective membrane performance can be observed through the filtration process based on the results of pure water flux. The series of nanofiltration module units used in this work is shown in **Figure 1**. The procedure was conducted by applying a peristaltic pump with a flow rate of 0.1 L/min and a pressure of 1.0

bar. Filtration time recording begins when the water exits through the membrane material, and measurements are taken every 10 min as the filtration occurs. The valve is opened to allow feed water to flow through the pipe to the membrane. Water that penetrates the membrane wall (permeate) is accommodated in a 100 mL measuring cup, and the permeate flow rate is measured by recording the volume accommodated every 10 min until it reaches a constant volume. Water that cannot penetrate the membrane wall is continuously returned to the feed tank.

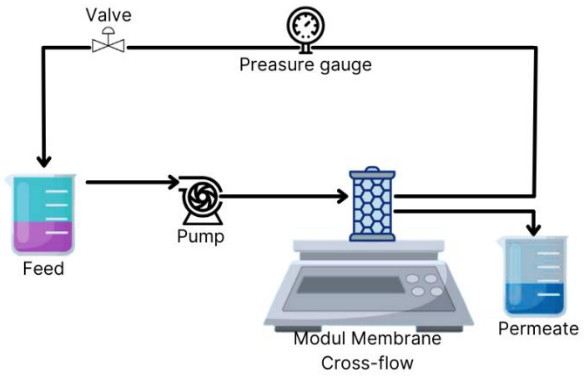


Figure 1. Schematic of nanofiltration equipment.

The membrane flux was obtained from the change in the permeate volume per unit of time and the membrane surface area. The equation used to calculate the flux is shown in Equation (3), where J is flux (L/m^2.h), V is the volume of permeate (L), A is the surface area of the membrane (m^2), and t is the filtration time (h)[17].

$$J = \frac{V}{A.t} \tag{3}$$

The membrane's selectivity was obtained by measuring the rejection of 300 ppm Mg^{2+} solution. The concentration of the Mg^{2+} solution was analyzed using a UV-Vis Spectrometer. The rejection of the Mg^{2+} solution was calculated using Equation (4), where R is the rejection coefficient (%), C_f is the Mg^{2+} concentration in feed (ppm), and C_p is the Mg^{2+} concentration in permeate (ppm).

$$R = 1 - \frac{C_P}{C_F} \times 100\% \tag{4}$$

3. Result and discussion

3.1. Membrane morphological analysis

Based on the results of SEM observation in **Figure 2**, all membranes generally appear to have similar surface morphology. However, adding PEG-HE and nanocarbon creates more macro voids than the original PES membranes. Based on **Figure 2**, it can be seen that the macro voids PES membrane cross-section have finger-shaped with the same size. The membrane cross-section with the addition of PEG-HE and nanocarbon also showed an increase in the membrane pores in the form of sponge macro cavities on all sides of the membrane cross-section. This increase is because adding the PEG-HE additive, which is amphiphilic with a hydrophilic head and hydrophobic tail, can reduce the surface tension at the points where surfactant molecules are present, thereby facilitating the diffusion of water molecules. This phenomenon causes a delay in demixing in the coagulation bath and increases pores in the cross-section membrane. The combination of hydrophilic PEG-HE and nanocarbon additives positively affects the membrane characteristics. The membrane produces using both additives shown has good porosity, flux values, and optimal rejection[18].

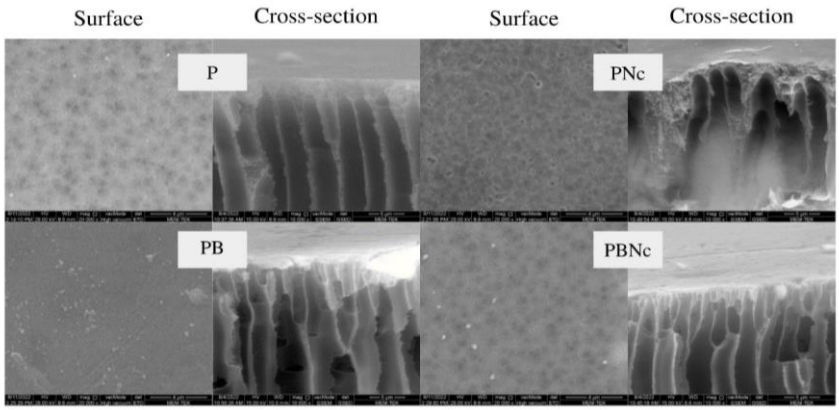


Figure 2. Surface and cross-section SEM images of the membrane samples.

3.2. Membrane hydrophilicity

The size of the angle formed between the membrane's surface and the air is represented by the water contact angle (WCA). A smaller WCA value indicates more hydrophilic properties of the membranes. The WCA analysis was conducted to determine the hydrophilicity of the membrane by dripping air onto its surface. The results of the WCA analysis can be seen in **Figure 3**. Based on **Figure 3**, it can be seen that the addition of the PEG-HE additive resulted in the WCA value decreasing from 69.5° on the PES membrane without additives to 58° on the PBNc membrane. The addition of the PEG-HE additive surfactant is proven to increase the hydrophilicity of the membrane, which is characterized by a decrease in the WCA value of the membrane. The hydrophilic properties of the membrane also affect the formation of pores in the membrane, this is because the addition of additives mixed in the membrane solution plays a role in increasing water interactions thereby accelerating the exchange of solvents and non-solvents during the phase inversion process[19]. The better hydrophilicity of the membrane has the potential to be an excellent anti-fouling agent. Impurities in the waste will not easily stick to the surface of the hydrophilic membrane, so the increase in hydrophilicity can prevent the membrane from fouling effect[20].

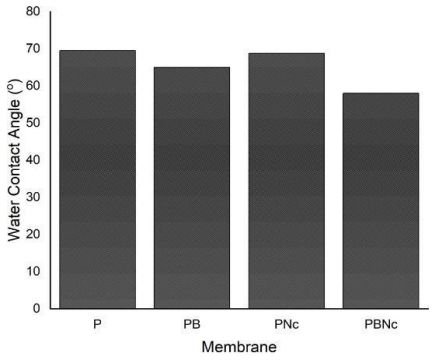
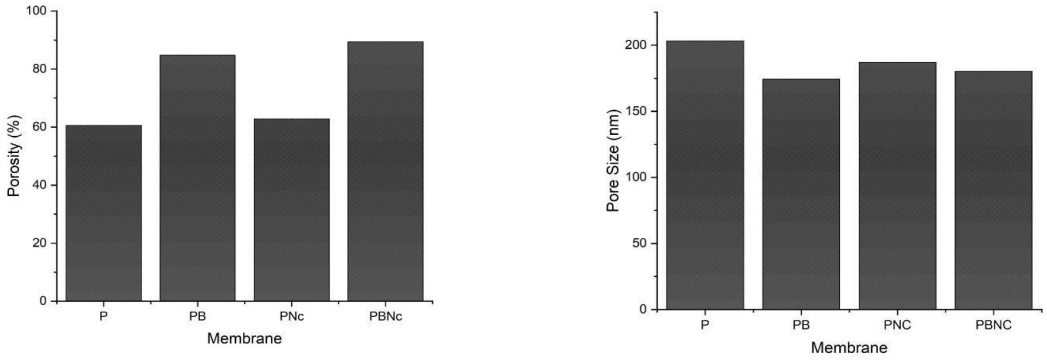


Figure 3. Hydrophilicity profile of the membranes.

3.3. Porosity and pore size

Porosity is the ratio between the pore volume and the total membrane volume. The porosity test is carried out to determine space (cavity) in the membrane. The membrane pore size significantly affects the membrane's performance in determining the water flux's value. The results of the porosity and pore size test can be seen in **Figure 4**. Based on **Figure 4**, it can be seen that the modified PBNC membrane has the most significant porosity value, which is 89.44%. Adding hydrophilic additives causes a slowdown of solvent-non-solvent exchange in the solidification process. This slowdown results in the formation of cavities in the membrane structure. According to Kusworo et al.[21], adding additives can increase the porosity of the membrane because

the additives can diffuse evenly to form a larger pore size.



(a) **(b)**

Figure 4. (a) porosity; and **(b)** membrane pore size value.

3.4. Membrane pure water flux

Flux is one of the parameters tested to describe membrane filtration performance, in general pure water flux has a direct relationship with the porosity and pore size of the membrane. The results of the membrane pure water flux test can be seen in **Figure 5**. The pure PES membrane had a flux value of 39.18 L/m^2.h, the membrane flux increased as it was modified with PEG-HE and nano carbon, the highest flux was obtained by the PBNc membrane with a flux value of 63.17 L/m^2 .hr. This is because the addition of additives provides various changes in the membrane such as hydrophilicity and pore size, based on **Figure 4**. It can be seen that the porosity of the PBNc membrane has a higher value compared to other membranes. Besides the hydrophilicity value of the membrane shown in **Figure 3** it also affects the pure water flux value, the PBNc membrane has the lowest WCA value of 58° which can be interpreted as the membrane with the best hydrophilicity. However, the addition of nano-carbon without the PEG-HE mixture had a reverse effect on the pure water flux value, this phenomenon can happen because the added nanocarbon only changes the cross-sectional properties but does not change the pore structure of the membrane surface, and affecting the pure water flux value[22].

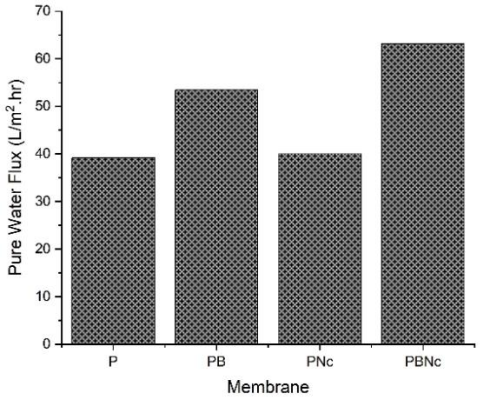


Figure 5. Pure water flux profile of the membranes.

3.5. Effect of additives on Mg^{2+} rejection

Determination of membrane rejection was accomplished by feeding synthetic fertilizer factory wastewater with a concentration of Mg^{2+} 300 ppm. The ability of membrane rejection can be seen from the amount of Mg^{2+} retained when passing through the membrane; the results of the membrane rejection test can

be seen in **Figure 6**. Based on **Figure 6**, it can be seen that the addition of additives to the membrane can affect the mg ions pass through the membrane pores; the higher of Mg^{2+} ions that are retained, the greater the rejection coefficient on the membrane, and the most significant rejection coefficient is pure PES membranes, which is 96.88%, then followed by a PBNc membrane with a rejection coefficient of 96.45%. Mg^{2+} rejection at the membrane is due to the narrow and tortuous structure formed during the phase inversion process. This structure has a smaller passage compared to Mg^{2+}, so it will be rejected if it wants to pass through the membrane. These channel complexes are present on the surface of the P and PBNc membranes (**Figure 2**) which will result in turtuousity on the membrane surface, because of this the rate of ion diffusion will decrease with increasing turtousity[23].

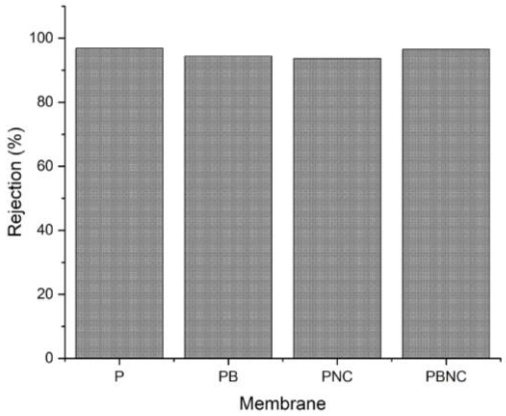


Figure 6. Mg^{2+} rejection ability of the membranes.

4. Conclusion

This study reviews the differences between pure PES membranes and modified PES membranes (PEG-HE and nanocarbon) on the performance and characteristics of each membrane. The PES membrane showed the best rejection of Mg^{2+} with a value of 96.88% but further modifications are needed to increase the water flux and hydrophilicity of the membrane. PBNc is the membrane with the best modification obtained, this is due to the higher flux value of 63.17 $L/m^2.h$, compared to the other three membranes and Mg^{2+} rejection of 96.45%, which is almost close to the rejection of pure PES membranes. This shows that the PBNc membrane has fairly good stability against water flux and Mg^{2+} rejection.

Author contributions

Conceptualization, NA; data curation, MEP and AF; formal analysis, RDH, AZM and ACA; investigation, RDH and AZM; project administration, SM; resources, IK; supervision, NA, A, MRB and SM; writing—original draft, RDH and AZM; writing—review and editing, NA, MRB and MPA. All authors have read and agreed to published version of the manuscript.

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Conflict of interest

The authors declare no conflict of interest.

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