

Removal of Natural Organic Matter from Water by Ultrafiltration Using Modified Polyethersulfone-Polyethylene Glycol Hexadecyl Ether Membrane

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ABSTRACT

The accumulation of organic pollutants in Indonesian waters is challenging for water treatment, because high pollutant levels increase the burden on the water technology used. Membrane technology can be used to optimize the filtration of organic pollutants. However, this technique is still limited by the fouling phenomenon on the membrane surface. The aim of this study was to investigate the effect of humidity on the structure formation and antifouling properties of the fabricated polyethersulfone (PES)-polyethylene glycol hexadecyl ether (PEG-HE) membranes. The PES membrane was modified by adding the additive PEG-HE and printed using the vapor and non-solvent induced phase separation (VNIPS) technique with air humidity of 70% and 90%. Changes in membrane properties and performance were analyzed before and after modification. Overall, the results show that adding 3% PEG-HE additive and using the VNIPS technique have a favorable impact on the membrane, namely, increasing the hydrophilicity, as well as the number and size of pores. The PES membrane modified with PEGHE additives and 90% air humidity exhibited the highest stability in various aspects of analysis, such as a water contact angle of 55.11°, a pure water flux of 69.86 L/m².h, a flux recovery ratio (FRR) of 91.91%, and humus acid rejection of 86.84%. In conclusion, the present research provides valuable insights into developing PES membranes with antifouling properties for filtering organic pollutants.

Keywords: PES, VNIPS, antifouling membrane, natural organic matter.

INTRODUCTION

Recently, the water crisis has been the most severe problem in the world. The main causes are high industrial expansion and an increase in the global population [Dias and Ghisi, 2024]. Although 70% of the Earth is covered with water, only 1% of it can be used by humans [Elhamamah et al., 2024]. This number continues to decrease due to continuous water pollution. The water pollution in Indonesia has reached a critical stage, with 70% of water sources being polluted [Suryani, 2020]. The cause of this pollution is waste from the household,

industrial, agricultural, livestock, and mining sectors. This waste contains organic pollutants, heavy metals, and other toxic substances [Amin et al., 2023].

The decomposition of plants and aquatic organisms produces a complex matrix of organic compounds known as organic pollutants. This matrix contains various substances, including humus, fulvic, and transphilic [Jarukas et al., 2021]. Organic pollutants can persist for a long time in the environment and are toxic to organisms. In addition, organic pollutants are bioaccumulated and bioconcentrated, meaning that the higher the position of an organism in the food chain, the

higher the pollutant level. This can endanger human health, leading to cancer, obesity, hormonal disorders, and nervous system disorders [Alharabi et al., 2018]. Organic pollutants can also contribute to global warming, climate change, and ecological change [Anderson et al., 2023]. Therefore, it is necessary to reduce the level of organic pollutants in water before humans consume it. However, the conventional water treatment technologies that are widely applied in Indonesia, such as coagulation, flocculation, and sedimentation, have not been able to completely remove organic pollutants and have many shortcomings, including the use of chemical compounds, requiring large areas of land, and taking a long time [Iyare et al., 2020]. Therefore, it is necessary to develop the technology that can overcome this problem.

Optimizing membrane technology is one way to reduce the number of pollutants in water, especially organic pollutants. Compared to conventional water treatment methods, this technique has several advantages, such as easy operating conditions, low energy consumption, the ability to be combined with various other processes, and does not require a large area of land [Kallem et al., 2020; Mulyati et al., 2024]. Several studies have reported the influence of membrane type and filtration methods in reducing the amount of organic pollutants in water. [Mulyati et al., 2024] researched PVDF membrane surface modification using vanillin for humus acid removal, which provided good results regarding membrane properties and performance. [Ambarita et al., 2021] used a polyethersulfone (PES) membrane with a combination of dragon blood resin (DBR) and AgNP, which showed increased membrane hydrophilicity, high permeability, and good rejection of humus acid. [Fatanah et al., 2024] examined the synergistic impact of the combination of $Mg(OH)_2$ and silica to fabricate PES membranes, which improved flux performance, selectivity, and resistance to fouling.

On the basis of several reports on using membranes as filtration media and other reference studies, the membrane process can remove organic pollutants in water. Unfortunately, this technology still faces problems in the form of fouling, namely the accumulation of particles on the membrane surface, which can interfere with the filtration process [Yu et al., 2021]. One approach that can be used to restore membrane performance is the vapor- and non-solvent-induced phase separation (VNIPS) method, which combines two membrane manufacturing techniques, namely vapor-induced phase separation

(VIPS) and Non-solvent-induced phase separation (NIPS), which can prevent the dissolution of additives into non-solvents to increase the antifouling properties of the membrane. To date, few studies have attempted to produce membranes with antifouling properties using membrane surface VNIPS for filtering organic pollutants.

Therefore, this study aimed to examine the potential of membrane fabrication using the VNIPS surface technique to produce membranes with antifouling properties through a combination of PES and polyethylene glycol hexadecyl ether (Brij 58). PES was used because it has various advantages, such as thermal stability high pressure resistance, hydrolytic stability, excellent oxidative activity, and resistance to organic solvents [Fahrina et al., 2022]. Furthermore, PEGHE or Brij 58 was chosen because it can increase the hydrophilicity, high compability with various polymers, helps in pore control, and good thermal stability [Mukramah et al., 2017]. Antifouling PES membranes were made using the VNIPS technique, where a partial phase inversion process occurs by exposing air to the membrane before solidification in a coagulation bath. The solvent used was dimethylacetamide (DMAc). Variations in air humidity change the morphology of the resulting membrane. Thus, this combination technique has the opportunity to improve the performance of the membranes produced in this research. The prepared membranes were then evaluated for their characteristics by observing their morphology using a scanning electron microscope (SEM), functional group analysis using a Fourier transform infrared (FTIR) instrument, and measuring porosity and water contact angle (WCA). Next, the filtration performance, such as antifouling and rejection properties, were tested. Humic acid was used as an organic pollutant in this study.

MATERIALS AND METHODS

Materials

The main material used is polyether sulfone (PES, Ultrason E6020P). The solvent was dimethylacetamide (DMAc, Wako Pure Chemical Industries, Japan). Polyethylene glycol hexadecyl ether (Brij 58, Mw 1.124) (Sigma Aldrich Co., LLC, Germany) was used as an additive, distilled water as a non-solvent, and a humic acid solution (Sigma Aldrich Co., LLC, Germany) was used as a model organic pollutant.

Membrane preparation

Membrane preparation began by dissolving PES and the Brij 58 polymers in DMAc. The solution was stirred until homogeneous (24 h) using a magnetic stirrer. The homogenized membrane solution was left for 15 min to remove air bubbles. Next, the VNIPS technique was used to fabricate the membrane. The membrane solution was poured onto a glass plate and printed using a membrane applicator with a thickness of 300 μm . After that, the membrane was exposed to air for 0 and 15 s with humidity variations in 70% and 90% at room temperature (30 °C). The compositions of the membrane solutions and treatments of the membranes are listed in Table 1. The membrane and glass plate were then dipped in a coagulant bath containing water as a non-solvent. The formed membrane sheets are then stored in a container containing distilled water to remove any remaining solvent from the membrane. The detailed membrane preparation procedure is shown in Figure 1.

Analysis of membrane properties and characterization

Scanning electron microscopy (SEM)

Membrane morphology analysis of membrane pore size, pore distribution, and overall pore geometry will be performed using scanning electron microscopy (SEM, Joel JSM 6360, LA). The SEM test was performed by placing the sample in a sample

holder, which was attached using carbon tape, and the sample was coated with a conductive material. The sample was then scanned with an SEM instrument, and the scanned image was observed.

Fourier transform infrared (FTIR)

Fourier transform infrared (FTIR, Shimadzu 8400) spectroscopy was used to analyze the functional groups contained in the membrane. Analysis was performed by cutting a membrane with an area of 4 cm^2 . The sample is then placed in a sample holder and analyzed at a wavelength between 500 and 4000 cm^{-1} . The results of the instrument readings were then analyzed to determine the functional groups of the membrane.

Hydrophilicity test

A water contact angle meter (Drop Master 300) was used to determine the water contact angle on each membrane. The test was carried out by placing the membrane on a glass panel and placing a drop of water on its surface. The angle between the water droplet and the flat surface was automatically recorded on the monitor.

Filtration performance test

Flux test

The flux test was carried out using a crossflow filtration module. Before the test, the membrane was first compressed for 1 h using distilled water

Table 1. Membrane code and polymer composition

Membrane code	PES (%)	PEG-HE (%)	DMAc (%)	Air exposure (seconds)	Humidity (%)
P	16	–	84	–	–
PP		3	81	–	–
P-70%		3	81	15	70
P-90%		3	81	15	90
PP-70%		3	81	15	70
PP-90%		3	81	15	90

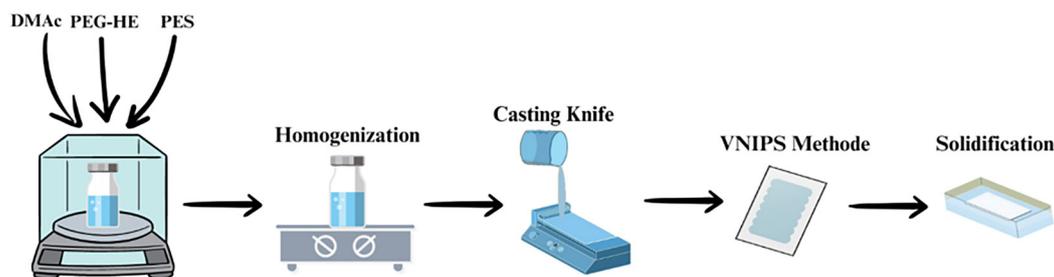


Figure 1. Membrane preparation scheme

at room temperature and a transmembrane pressure of 1 bar. Next, pure water filtration as a feed solution was carried out with the same treatment; the permeate was weighed every 10 min until a constant weight was obtained. Flux calculations can be performed using Equation 1.

$$J = \frac{V_p}{A \times \Delta t} \quad (1)$$

where: J is the flux (L/m^2h), V_p is the permeate volume (L), A is the effective membrane area (m^2), and Δt is the permeation time (h).

Rejection test

The rejection test used the same equipment as the flux test. Humic acid with a concentration of 50 ppm was used as a model for the detection of organic pollutants in water samples. The humic acid concentration in the feed solution before and after filtration was measured using a UV-Vis spectrophotometer. The humus acid rejection is calculated using Equation 2.

$$R_m = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \quad (2)$$

where: R_m is the humus acid rejection value (%), C_p is the feed concentration in the feed (mg/L), and C_f is the feed concentration in the permeate (mg/L) [Raja et al., 2024].

Fouling resistance

The antifouling performance of the membrane during the filtration process was analyzed

by evaluating the following fouling indices: flux recovery ratio (FRR), total fouling ratio (R_t), reversible fouling (R_r), and irreversible fouling (R_{ir}). The antifouling performance of the membrane was calculated using Equations 3–6:

$$FRR = \left(\frac{J_R}{J_0}\right) \times 100\% \quad (3)$$

$$R_t = \left(1 - \frac{J_p}{J_0}\right) \times 100\% \quad (4)$$

$$R_r = \left(\frac{J_R - J_p}{J_0}\right) \times 100\% \quad (5)$$

$$R_{ir} = \left(\frac{J_0 - J_R}{J_0}\right) \times 100\% \quad (6)$$

where: J_0 is the pure water flux ($L/m^2 \cdot h$), J_p is the flux from a water sample containing pollutants ($L/m^2 \cdot h$), and J_R is the flux from pure water after pollutant filtration ($L/m^2 \cdot h$) [Raja et al., 2024].

RESULTS AND DISCUSSION

Membrane structure and morphology

SEM analysis is a microscopic technique that provides information about the morphology of the prepared membrane. The SEM results are shown in Figure 2, which shows that all membranes generally have a similar surface structure with different pore distributions. The PP-90% membrane exhibited a greater pore distribution than other membranes. This increase occurred due to a delay

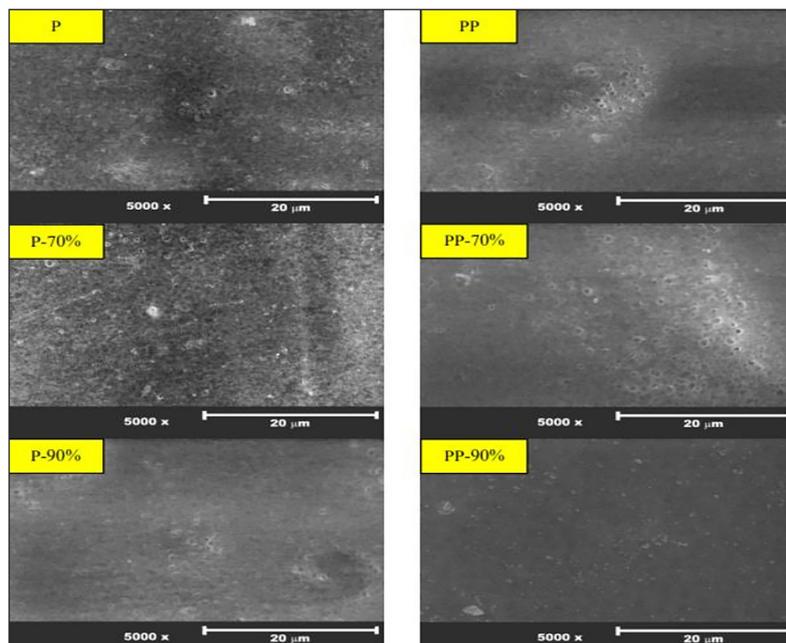


Figure 2. SEM image of the membrane surface

in demixing in the VNIPS process, which resulted in more additives being retained in the membrane matrix [Moghadassi et al., 2018]. In addition, exposure to air at a certain relative humidity also increases the number of pores on the membrane surface. When the relative humidity of the air was increased, the density of water vapor in the air also increased. Therefore, more water vapor enters the membrane surface, increasing the number of pores in the membrane [Chen et al., 2022].

Functional groups

Fourier transform infrared (FTIR) spectroscopy is used to identify functional groups in membrane samples. The FTIR spectra of all membrane samples are shown in Figure 3. The wave numbers detected include 1484 cm^{-1} (Benzene), 1238 cm^{-1} (C-O-C), 1148 cm^{-1} (O=S=O), 834 cm^{-1} (C-H), 717 cm^{-1} (C-S), and 554 cm^{-1} (SO_2). Some of these wave numbers were functional PES groups reported in previous research (Mulyati et al., 2024). The spectrum did not differ significantly, because the membrane material was the same (PES). However, membranes added with PEGHE and printed using the VNIPS technique showed changes in peak intensity at wave numbers 2929 cm^{-1} (-CH₃ and -CH₂-) and 3630 cm^{-1} (-OH). This is caused by a delay in demixing in the VNIPS process, which leads to the retention of more additives in the membrane matrix [Moghadassi et al., 2018]

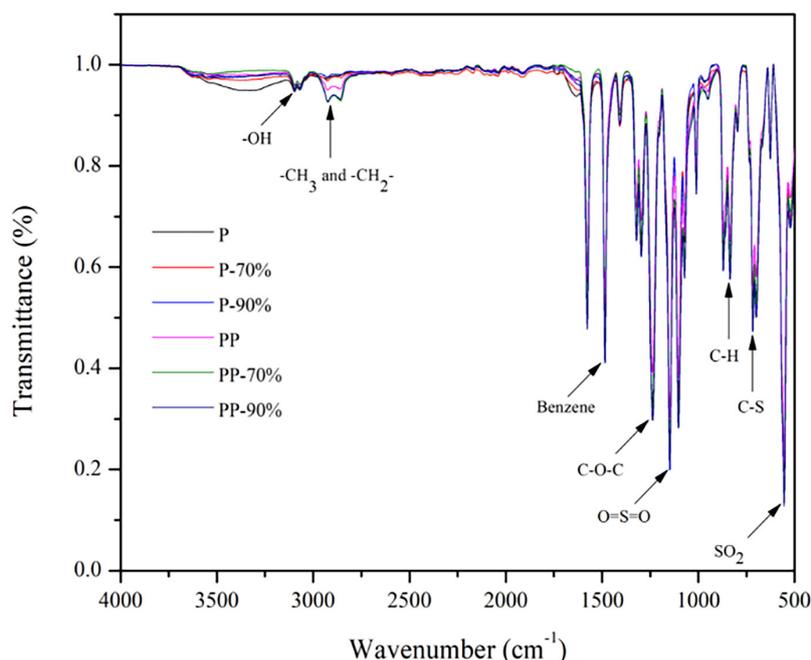


Figure 3. IR spectra of original and modified membrane

Membrane hydrophilicity properties

The water contact angle (WCA) is an important indicator of the hydrophilicity of a membrane. Membranes with low WCA values show high hydrophilicity. The results of the WCA analysis are presented in Figure 4. The P membrane prepared using the NIPS method exhibited a WCA of 75.31° . The use of PEGHE additives in the fabrication of PP membranes increases their hydrophilicity, which is evident from the decrease in the WCA value to 60.05° . When the P-70%, P-90%, PP-70%, and PP-90% membranes were prepared using the VNIPS technique at air humidity values

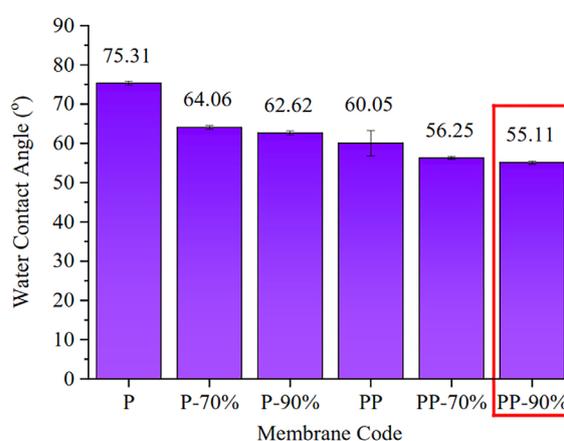


Figure 4. Water contact angle of the original and modified membrane

of 70% and 90%, a further decrease in the WCA value was observed. This is caused by the formation of partial structures during exposure to air. The structure formed on the membrane surface during exposure to air is an obstacle for the PEGHE additive during demixing in a coagulation bath [Naw et al., 2020]. The hydrophilicity of the membrane also influences the formation of pores in the membrane because using a mixture of additives in the dope solution can increase the speed of phase inversion between the solvent and the non-solvent. Membranes with high hydrophilicity can act as good antifouling agents [Haikal et., 2023].

Permeation profile and membrane rejection ability

The volume of clean water that can pass through the membrane layer and the ability of the membrane to reject humic acid particles are shown in Figures 5 and 6, respectively. Membrane P has a flux of 25.53 L/m².h. When the PP membrane was added with PEGHE, the flux increased to 36.97 L/m².h. Adding PEGHE additives improves membrane performance [Mukramah et al., 2017]. Furthermore, the membranes exposed to air with a humidity of 70% or 90% also showed a significant increase in flux. This increase is caused by stronger interactions between the membrane surface and water [Barambu et al., 2020].

Humic acid is an organic compound formed from the degradation of plants and molecules, and it increases the hydration layer of living creatures through the action of microorganisms, both chemically and biologically [Gul and Yalcinkaya, 2021]. The presence of humic acid can affect the quality, color, and taste of water [Mulyati et al., 2024]. In this study, humic acid was used as a contaminant model to test the performance of the membrane in rejecting organic pollutants (Figure 6). The test results showed that the average percentage of humus acid rejection exceeded 50%. Although the PP-90% membrane exhibited the highest pure-water flux (Figure 5), it exhibited the lowest rejection percentage. This is caused by the formation of membrane pores, which are influenced by additives and exposure to air, which affects the ease of water and humic acid flow through the membrane surface.

The application of various modified PES membranes in treating different types of polluted raw water has been explored in other studies. A PES membrane modified with CuO-functionalized

Fe₃O₄ nanoparticles was employed to treat water contaminated with sodium chloride and methylene blue. Using a straightforward filtration process with a trans-membrane pressure of 0.5 bar, the contaminant concentration could be reduced by up to 87.98% [El-Sawaf et al., 2024]. The PES membrane has also been applied to produce clean water from raw water contaminated with sodium alginate as well as calcium and magnesium ions. Modification of the PES membrane with 2-(methacryloyloxy) ethyl phosphorylcholine can reduce the formation of a cake layer on the membrane surface, thereby extending the filtration time [Arahman et al., 2018]. Furthermore, the application of the PES membrane has also been tested for the treatment of clean water from raw water containing dextran. In this case, the modification of the PES membrane can enhance the antifouling performance of a membrane and reduce dextran content in the water by up to 87% [Rahman et al., 2008].

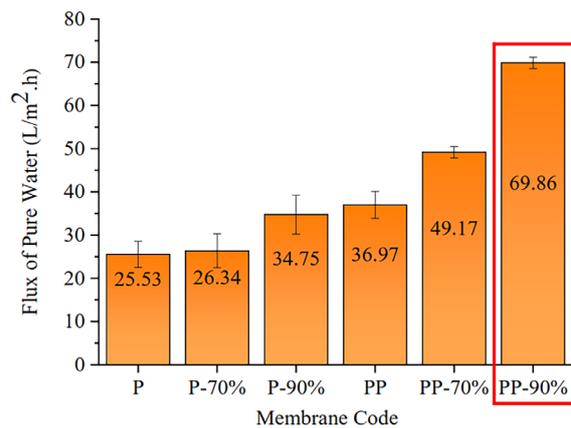


Figure 5. Filtration profile of original and modified membrane

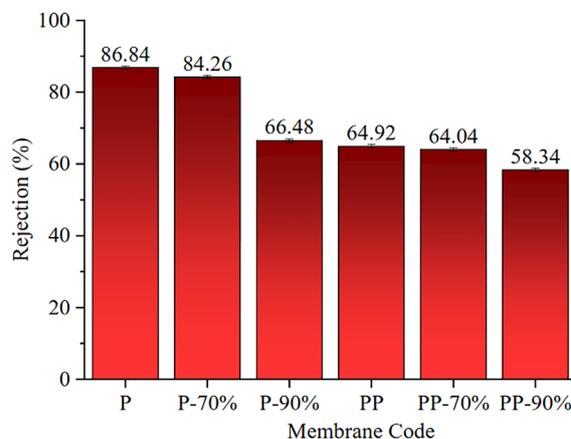


Figure 6. Rejection of humic acid solution filtrated by original and modified membrane

Antifouling properties

Fouling is the deposition of organic material on the surface or in the membrane pores, which can reduce the performance of the membrane [Ambarita et al., 2024]. The PP-90% membrane exhibited the highest FRR of 91.91%, indicating that the pure water flux after the humus acid filtration process was close to the pure water flux before the filtration process (Figure 7). The PP-90% membrane is highly hydrophilic [Mulyati et al., 2024]. In addition to FRR, fouling behavior can also be studied from the loss of flux due to total, reversible, and irreversible fouling (Figure 8). The irreversible fouling of the PP-90% membrane was 8.08%. The test results prove that the VNIPS technique can produce membranes with excellent fouling resistance.

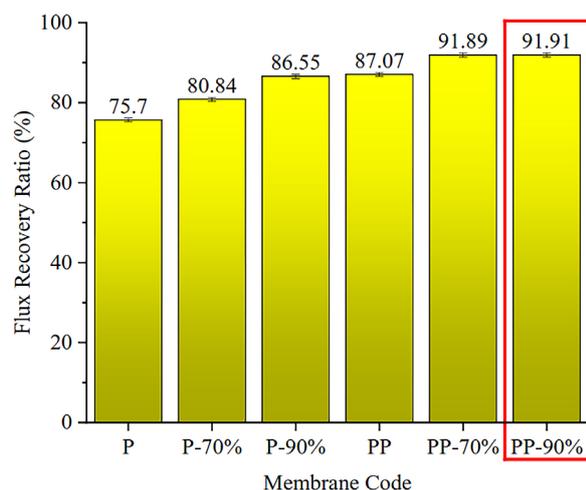


Figure 7. Flux recovery ratio (FRR)

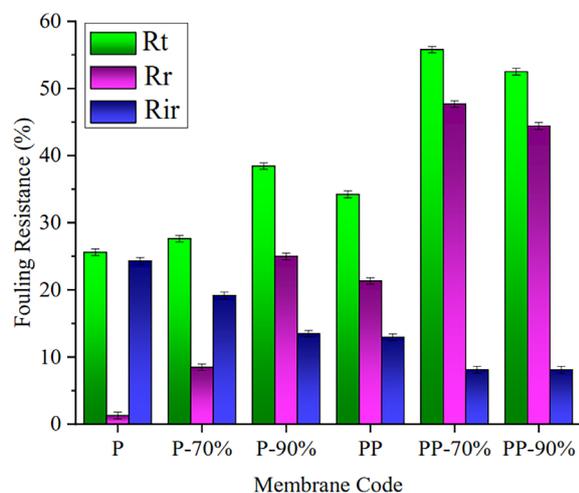


Figure 8. Fouling resistance

CONCLUSIONS

The use of VNIPS has a positive impact on membrane characteristics and performance. The characteristic test results of the PP-90% membrane were superior due to the greater distribution of membrane pores, -OH, -CH₃, and -CH₂-functional groups, and the highest reduction in water contact angle, reaching 73.18% of that of the pure PES membrane, indicating its greater hydrophilicity. Evaluation of the pure water flux and humic acid filtration process proved that the PP-90% membrane was superior to the other membranes. Furthermore, the PP-90% membrane exhibited the highest flux and antifouling properties of 69.86 L/m².h and 91.91%. However, the membrane rejection rate in the PP-90% membrane rejection process was the lowest at 58.34%. This occurred because the size of the membrane pores formed by the additives and exposure to air affected the ease of water and humic acid flow across the membrane surface.

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