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Foulant-specific transformations of polysulfone membrane: A mechanistic study of shrimp feed and humic acid fouling with implications for Recirculating Aquaculture Systems (RAS)

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The integration of ultrafiltration (UF) into recirculating aquaculture systems (RAS) is a promising strategy for managing organic wastewater contaminants, yet its efficacy is hindered by severe membrane fouling and physico-chemical transformation. This study investigates these challenges by evaluating polysulfone (PS) membrane under dead-end and cross-flow hydrodynamics. Performance was systematically assessed using shrimp feed (SF; 1.5–2.5 g L^{-1}) and humic acid (HA; 0.2–0.7 g L^{-1}) as aquaculture-relevant model foulants under varied operating pressures. The single-foulant systems were employed to isolate foulant-specific membrane responses under controlled conditions rather than to reproduce the complete physicochemical composition of actual RAS water. Pristine and fouled membranes were characterized (MWCO, SEM, FTIR, contact angle) to elucidate fouling mechanisms. Post-operation analyses revealed distinct, foulant-specific surface transformations. Higher operating pressures were associated with more pronounced time-dependent flux deterioration, particularly during SF filtration, whereas HA maintained consistently high rejection across the investigated pressure range. Furthermore, Cross-flow operation exhibited higher mean flux than dead-end filtration under the investigated conditions, with the configuration-associated enhancement being particularly pronounced for SF.

1. Introduction

The growth of the aquaculture industry continues to increase which simultaneously raises the demand for sustainable aquaculture wastewater management [1,2]. The Recirculating Aquaculture System (RAS) is an attractive option for contaminant removal through a series of integrated processes, which results in a reduction in the amount of water consumption [3–5]. The primary objective of this process is to eliminate any unwanted substances and pathogens for water recycling purposes [6–8]. The uneaten feed and the animal excretions need to be minimized as both are ammonia contributors in aquaculture systems and are harmful to the animals living in the system [9,10].

It is commonly known that organic contaminant in aquaculture systems have diverse characteristics and sizes and are distributed randomly. Thus, conventional processes such as classification or centrifugation may not be effective in handling them [11]. In addition, the presence of protein in the system can lead to the formation of colloids, thus making the coagulation-flocculation process as one of the potential techniques to solve this problem [12]. However, it is indispensable to minimize additional chemicals in the system. Therefore, the membrane filtration process which has no additional chemicals arises as an alternative to removing contaminants. Among the variety of pore size available, microfiltration (0.1 – 10 μm) and ultrafiltration (0.001 – 0.01 μm) have the potential to replace the coagulation – flocculation process [13]. In addition, nanofiltration and reverse osmosis can also be used in aquaculture systems, although both processes generally require more energy than microfiltration or ultrafiltration [10,14,15]. Besides, Yong et al. have applied membrane distillation in aquaculture systems, which showed success in recovering ammonia up to 465 mg L^{-1} (~23 times the initial concentration at around 19.83 mg L^{-1}) [16]. Simultaneously, almost all of the ammonia is rejected, resulting in a high separation factor of 148 (only 0.04 mg L^{-1} of ammonia is able to pass through the membrane).

Despite the advantages, the implementation of membrane processes in aquaculture remains limited by several challenges, including (1) low membrane selectivity, (2) high operating costs, (3) membrane fouling,

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and (4) membrane degradation [17]. Among those obstacles, membrane fouling is the most difficult barrier to overcome. In addition, various factors also affect the occurrence of membrane fouling namely: (1) wastewater characteristics, (2) operating conditions, (3) membrane properties [18,19].

The membrane properties, such as porosity, tortuosity, hydrophilicity, surface charge, pore size, and other physical properties, are crucial in separation processes. In general, membranes with hydrophilic properties are always preferred in aqueous-based separations, which cannot be separated from their high surface wettability and anti-fouling ability [20,21]. In addition, membranes with high surface wettability produce high flux without reducing their selectivity [22]. Furthermore, with certainty, the size ratio between contaminants and pore size can determine the existing fouling mechanism [18]. Overall, the relationship between membrane surface charge and isoelectric points of contaminants is affected by pH. Up to now, the electrostatic repulsion between the membrane and the contaminants has resulted in lower fouling and increased flux [18].

Several membrane fouling studies are summarized in Table S1, indicating that fouling behavior is strongly dependent on membrane properties, foulant characteristics, solution chemistry, and operating conditions. Actual RAS water contains a complex mixture of feed- and fecal-derived organic matter, fine suspended solids and colloids, microbial-derived compounds, dissolved organic matter, nitrogen species, and inorganic constituents [23–25]. Feed input and feed-derived particles have been directly associated with increased membrane fouling in marine RAS, while the continuous recirculation of water promotes the accumulation and transformation of dissolved organic matter, including humic- and fulvic-type compounds [23,24]. Consequently, a single synthetic solution cannot reproduce the complete fouling environment of an operating RAS. Nevertheless, controlled model-foulant systems are useful for isolating membrane responses to chemically distinct foulant classes, and previous aquaculture membrane studies have employed shrimp-feed-derived organic matter and humic acid for this purpose [26]. Therefore, in this study, shrimp feed (SF) and humic acid (HA) were employed as aquaculture-relevant model foulants representing feed-derived and humic-type organic matter, respectively. The objective was to investigate their individual effects on the filtration performance and physicochemical properties of a commercial polysulfone ultrafiltration membrane under controlled laboratory conditions, rather than to reproduce the complete composition of actual RAS wastewater. The effects of operating pressure, foulant concentration, and filtration configuration were evaluated, while MWCO, SEM, FTIR, and water contact angle analyses were used to characterize the corresponding membrane transformations.

2. Material and methods

2.1. Materials

Commercial polysulfone (PS) ultrafiltration membranes with a nominal molecular weight cut-off (MWCO) of 20,000 Da were supplied by RisingSun Membrane Technology. Shrimp feed (SF; IRAWAN 681 V, Central Proteina Prima Indonesia) and humic acid (HA; Sigma-Aldrich) were used as aquaculture-relevant model foulants. The shrimp feed contained protein (>30%), crude fiber (<4%), fat (>5%), moisture (<12%), and ash (<11%). Sodium chloride (NaCl), hydrochloric acid (HCl), sulfuric acid (H_2SO_4), phenol (C_6H_5OH), glucose ($C_6H_{12}O_6$), sucrose ($C_{12}H_{22}O_{11}$), and dextran standards with molecular weights of 1–40 kDa were used for feed preparation and membrane characterization. Deionized water produced in the laboratory, with a conductivity of approximately 15 μS cm^{-1} and total dissolved solids of approximately 11 mg L^{-1}, was used throughout the experiments.

2.2. Methods

2.2.1. Filtration feed preparation

Shrimp feed was ground and sieved to obtain particles smaller than 200 mesh. The sieved particles were then dispersed in deionized water at concentrations of 1.5, 2.0, and 2.5 g L^{-1}. The suspensions were homogenized using an IKA T25 digital ULTRA-TURRAX® at 20,000 rpm for 5 min at 25 ± 2 °C, followed by filtration to remove residual large particles. The resulting filtrate was exposed to UV-C irradiation to minimize microbial growth before use in the filtration experiments. Humic acid solutions were prepared separately at concentrations of 0.2, 0.4, and 0.7 g L^{-1}. NaCl was added to each feed solution at 10 g L^{-1} to provide a simplified saline background relevant to low-salinity shrimp aquaculture conditions [27]. The NaCl-based medium was used to control ionic background and was not intended to reproduce the complete ionic composition of actual RAS water.

SF and HA were investigated separately as single-foulant challenge solutions to enable controlled comparison of their individual membrane-fouling behavior. Previous aquaculture membrane studies have employed residual shrimp feed and HA as model organic foulants [26]. However, the SF concentrations employed in the present study (1.5–2.5 g L^{-1}) are higher than the 0.3–0.8 g L^{-1} shrimp-feed range previously investigated by Widiasa et al. [26]. Similarly, the HA concentrations used here (0.2–0.7 g L^{-1}) are within or above the upper range of high-concentration model-fouling studies; for example, HA concentrations of 50–150 mg L^{-1} and 500 mg L^{-1} have been employed in accelerated membrane-fouling evaluations [28,29]. Accordingly, the present concentrations are interpreted as accelerated fouling conditions designed for comparative mechanistic evaluation rather than as environmentally representative concentrations of actual RAS water. Therefore, the resulting fouling rates should not be quantitatively extrapolated to full-scale RAS operation.

2.2.2. Filtration procedure

The filtration experiments were organized into three experimental sets (See Fig. 1). The effect of operating pressure was evaluated under stirred dead-end filtration at 3, 5, 7, and 10 bar using SF at 2.0 g L^{-1} or HA at 0.4 g L^{-1}. The effect of foulant concentration was evaluated under dead-end filtration at a fixed pressure of 5 bar using SF concentrations of 1.5, 2.0, and 2.5 g L^{-1} and HA concentrations of 0.2, 0.4, and 0.7 g L^{-1}. Filtration-configuration effects were evaluated separately at 5 bar by comparing dead-end and cross-flow operation using SF at 2.0 g L^{-1} and HA at 0.4 g L^{-1}. All experiments were conducted at 25 ± 2 °C.

Prior to filtration, PS membrane coupons were immersed in deionized water for 24 h. Pure-water filtration was subsequently performed to establish the baseline membrane response. Dead-end filtration was conducted using a stirred cell with an effective membrane area of 15.9 cm^2, with N_2 gas providing the transmembrane pressure and continuous stirring at 500 rpm. Cross-flow experiments were conducted using a Sterlitech Explorer CF042P cell with an effective membrane area of approximately 42 cm^2 and a hold-up volume of 17 mL. The feed was recirculated at approximately 1.0 L min^{-1}, corresponding to an average cross-flow velocity of approximately 0.18 m s^{-1}, calculated from the volumetric recirculation rate and the cross-sectional area of the flow channel. Prior to foulant filtration, pure-water flux was measured using the pristine PS membrane under the corresponding operating pressures. For dead-end filtration, pure-water measurements were conducted at 3, 5, 7, and 10 bar to establish the pressure-dependent baseline membrane response. These measurements were used as clean-water controls for distinguishing pressure-related changes in membrane permeability from additional resistance arising during foulant filtration. The corresponding pure-water flux profiles are provided in Fig. S1. The upper pressure of 10 bar was included as a high-pressure challenge condition to evaluate the membrane response over an extended operating range and should not be interpreted as a recommended operating pressure for RAS ultrafiltration.

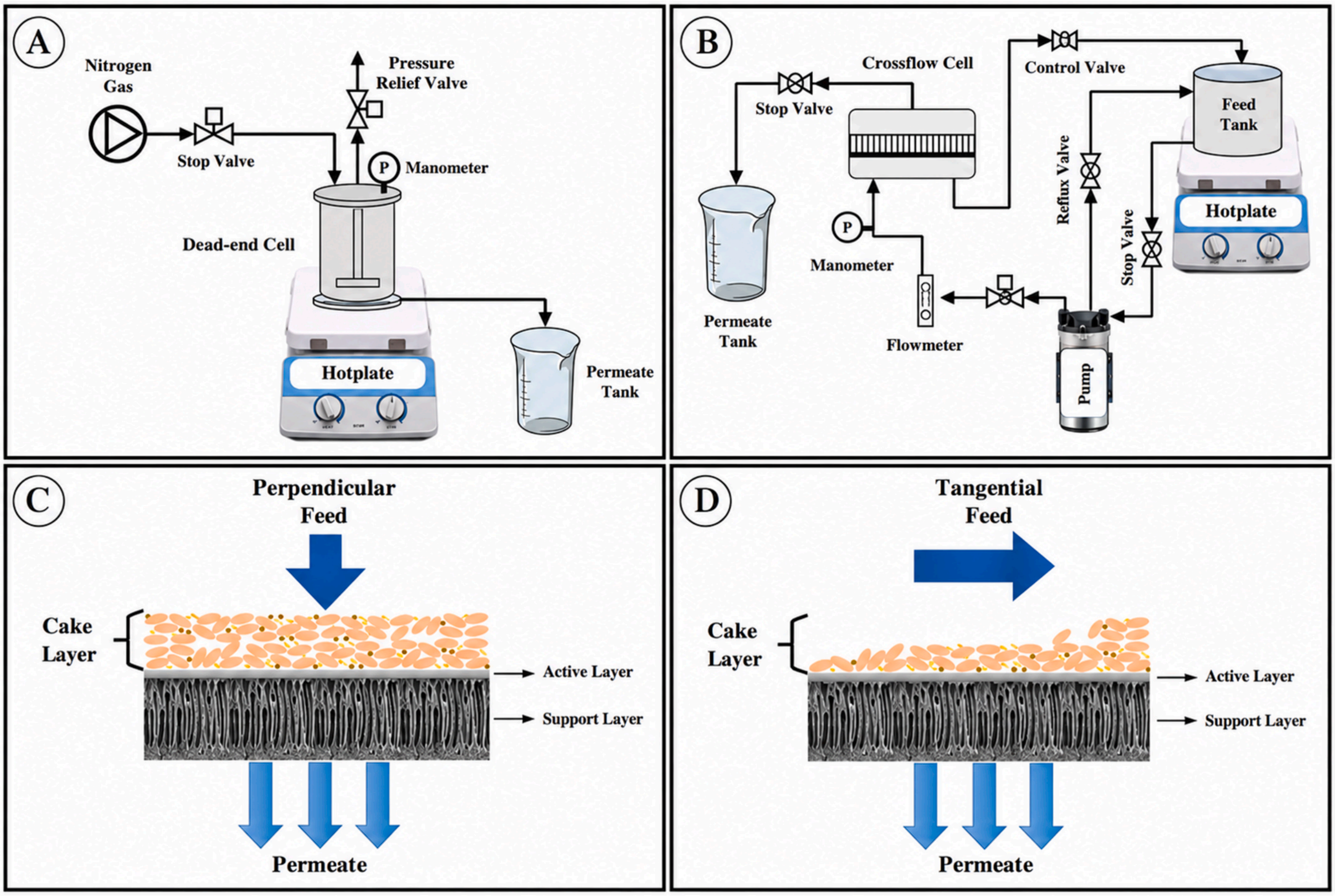

Fig. 1. Experimental setups and corresponding separation mechanisms for (a, c) dead-end stirred and (b, d) cross-flow filtration modes.

Permeate volume and the corresponding elapsed filtration time were recorded throughout filtration and used to calculate permeate flux according to Eq. (1).

$$J = \frac{V}{A.t} \tag{1}$$

where V, t, A are volume permeate (L), time (h), active membrane area (m^2), respectively.

Filtration duration varied according to the permeation behavior of each condition rather than being fixed across all experiments. For the PS cross-flow experiments conducted at 5 bar, cumulative permeate collection reached 80 mL after approximately 63.9 min for SF and 50.9 min for HA. The initial feed volume required to determine conventional permeate recovery could not be reliably reconstructed from the archived experimental records; therefore, recovery was not used as a basis for quantitative interpretation of the filtration results.

Foulant concentrations in the initial feed and permeate were quantified by UV–Vis spectrophotometry using foulant-specific calibration curves. SF and HA were analyzed at wavelengths of 280 and 255 nm, respectively, following the UV–Vis approach previously applied for aquaculture-relevant organic foulants [26]. For SF, the calibration relationship was A = 0.0754C–0.0053 (R^2 = 0.999), whereas for HA the relationship was A = 3.6623C–0.0343 (R^2 = 0.998), where A represents absorbance and C represents the corresponding foulant concentration (g L^{-1}). The concentrations determined for the initial feed and permeate were used as C_F and C_P, respectively, to calculate rejection according to Eq. (2).

$$R = \left(1 - \frac{C_P}{C_F}\right) \times 100\% \tag{2}$$

where C_P and C_F are the permeate concentration ($g.L^{-1}$) and the initial feed concentration ($g.L^{-1}$), respectively.

2.2.3. Membrane characterization

The membranes were systematically characterized to determine their pore size, hydrophobicity, morphology, and surface chemistry. Pore size was evaluated by the molecular weight cut-off (MWCO), defined as the molecular weight at 90% solute rejection. These tests were conducted at 25 °C and 5 bar using 200 mg/L organic solute solutions. Solute concentrations were quantified via the phenol-sulfuric acid method and measured with a UV-Vis spectrophotometer [Thermo Scientific™, Evolution™ 60S]. Membrane surface morphology was visualized by Scanning Electron Microscopy (SEM) [Hitachi, Flexsem 1000]. For SEM characterization, the SF concentration series (1.5, 2.0, and 2.5 g L^{-1}) and HA concentration series (0.2 and 0.4 g L^{-1}) were obtained under dead-end filtration at a fixed pressure of 5 bar. Configuration-associated morphology was evaluated at 5 bar using SF at 2.0 g L^{-1} and HA at 0.4 g L^{-1} under both dead-end and cross-flow operation. Surface hydrophobicity was determined by static water contact angle measurements [DataPhysics Instruments, OCA25]. Meanwhile, surface functional groups were identified using Fourier Transform Infrared (FTIR) spectroscopy [Shimadzu, IRTracer–100] over a wavenumber range of 4000–450 cm^{-1}. Post-filtration WCA and FTIR analyses were performed using PS membrane samples obtained at 5 bar. The SF-exposed sample corresponded to filtration with 2.0 g L^{-1} SF, whereas the HA-exposed sample corresponded to filtration with 0.4 g L^{-1} HA.

2.2.4. Statistical analysis

Quantitative filtration-performance experiments were performed in triplicate. Statistical analyses were performed using MATLAB R2025b (MathWorks, USA), with statistical significance defined at $p < 0.05$. For the dead-end filtration experiments, the effects of operating pressure and foulant concentration on permeate flux and rejection were evaluated separately for SF and HA using one-way analysis of variance (ANOVA), followed by Tukey's honestly significant difference (HSD) test for pairwise comparisons. For the comparison between dead-end and cross-flow filtration, two-way ANOVA was used to evaluate the effects of foulant type, filtration configuration, and their interaction on flux and rejection. Within-foulant comparisons between cross-flow and dead-end filtration were additionally evaluated using Welch's independent-samples *t*-test. Detailed statistical results are provided in Table S2–S5 of the Supplementary Data.

3. Results and discussion

3.1. Correlation of membrane characteristics with fouling phenomena

As previously mentioned, the characteristics of membranes are crucial for the successful implementation of ultrafiltration (UF) in the treatment of aquaculture wastewater. This analysis provides a comprehensive examination of the specific roles played by pore size, hydrophilicity, and surface chemistry [30,31]. The pore size of the membrane, indicated by its Molecular Weight Cut-Off (MWCO), serves as the principal mechanism for selectivity, operating through a size exclusion process.[32]. Nevertheless, the pore structure alone does not determine performance within this complex matrix. Membrane hydrophilicity is equally significant; a highly hydrophilic surface tends to reduce the adsorption of organic foulants, primarily proteins and carbohydrates, which are prevalent in aquaculture systems. This surface wettability is, in turn, influenced by the functional groups present on the membrane

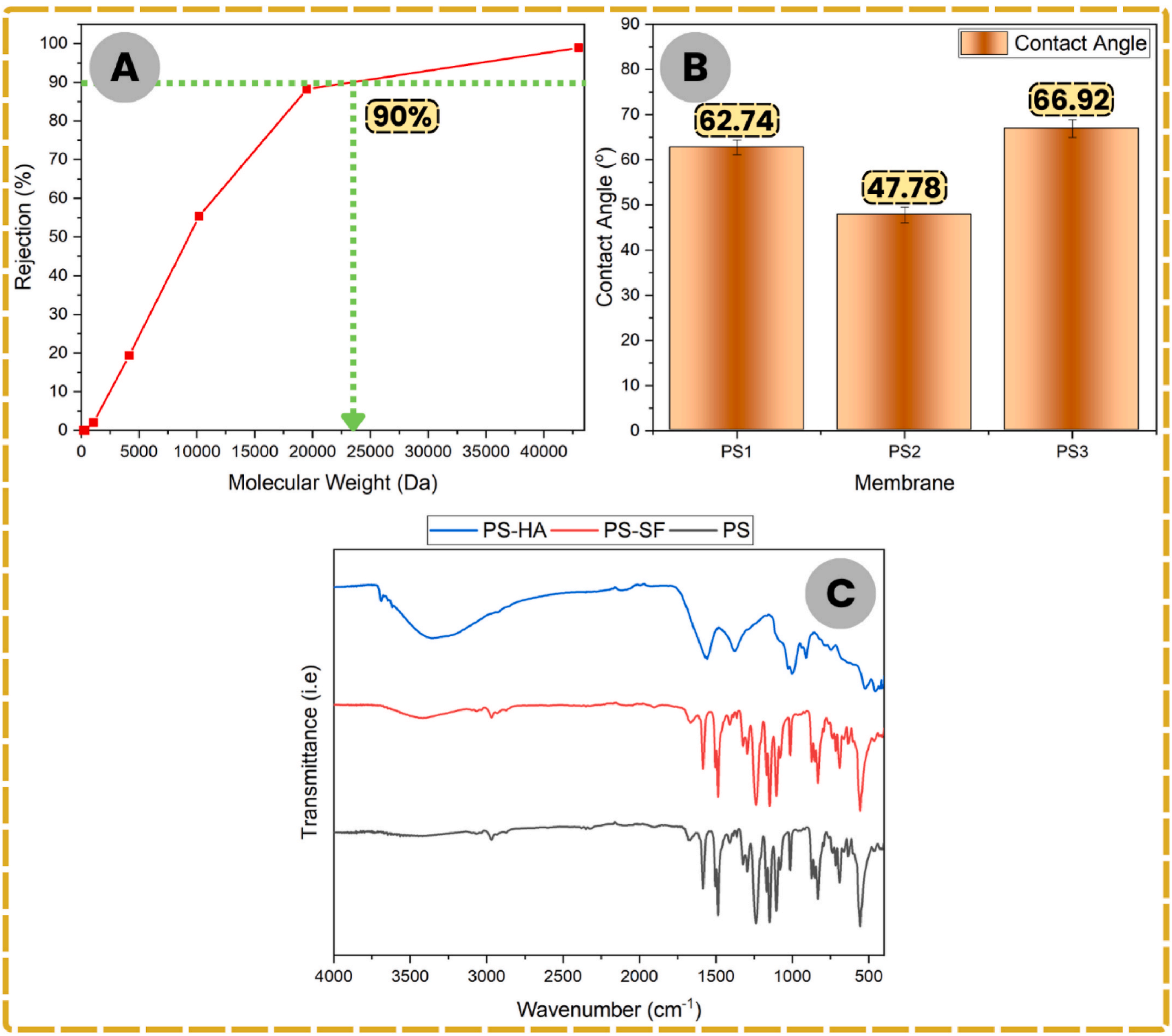

Fig. 2. (A) Rejection curve for MWCO determination of the pristine PS membrane at 5 bar; (B) water contact angle of pristine PS (PS1), PS after filtration with 2.0 g L^{-1} SF at 5 bar (PS2), and PS after filtration with 0.4 g L^{-1} HA at 5 bar (PS3); and (C) FTIR spectra of the corresponding pristine, SF-exposed, and HA-exposed PS membranes.

surface.[30,31]. The interdependence among these three factors is crucial for comprehending the overall processes of separation and fouling.

3.1.1. Pristine membrane characterization

Characterization of the pristine PS UF membrane established its baseline properties. The MWCO was experimentally determined to be ∼24 kDa (Fig. 2A), which is in close agreement with the manufacturer's reported value of 20 kDa. The slight discrepancy is likely attributable to differences in measurement methodologies and operating conditions. This MWCO confirms the membrane's theoretical capacity to reject large macromolecules, such as proteins and carbohydrates, via size exclusion [33].

Analysis by FTIR (Fig. 2C) confirmed the characteristic functional groups of the PS polymer. Key peaks were identified for the sulfone group (O═S═O), with symmetric and asymmetric stretching vibrations observed at 1159 cm^{-1} and 1231 cm^{-1}, respectively [34]. Other significant peaks included the asymmetric C-O-C stretch of the aromatic ether at ∼1248 cm^{-1}, aromatic C═C stretching at 1682 cm^{-1}, and aromatic C-H bending in the fingerprint region at 849 cm^{-1}. Aliphatic C-H stretching from methyl groups was also detected at 2869 cm^{-1} [35].

Wettability analysis (Fig. 2B) showed the pristine membrane surface to be moderately hydrophilic, leaning towards hydrophobic, with a water contact angle (WCA) of 62.74°. This inherent wettability is attributed to the sulfone (O═S═O) and ether (C-O-C) groups [34]. The oxygen atoms in these groups possess lone electron pairs, allowing the molecules as hydrogen bond acceptors for water molecules.

3.1.2. Post-operation analysis: SF-induced organic fouling

Membrane autopsy following operation with SF revealed a distinct surface transformation associated with the deposition of feed-derived organic material. As shown in Fig. 2C, several new FTIR features emerged after SF filtration, indicating changes in the chemical composition of the membrane surface. A prominent band at approximately 1550 cm^{-1} is consistent with Amide II-associated vibrations, involving N–H bending and C–N stretching, while the feature observed in the Amide III region at approximately 1350 cm^{-1} provides additional evidence of proteinaceous material associated with the fouled surface [36]. The broad O–H stretching band at approximately 3200 cm^{-1}, together with the C–O-associated feature around 1050 cm^{-1}, is consistent with hydroxyl- and carbohydrate/polysaccharide-like functionalities. FTIR-based characterization of organic membrane fouling similarly associates amide- and carbohydrate-related spectral regions with proteinaceous and polysaccharide-containing foulants [36].

Collectively, these spectral changes support the deposition of an SF-derived organic layer containing proteinaceous and polysaccharide-like components. However, FTIR identifies chemical functionalities and does not by itself establish the three-dimensional organization of these components as a protein–polysaccharide gel layer. The corresponding change in membrane wettability provides complementary evidence of surface transformation. For the SF-exposed membrane characterized after filtration at 5 bar, the WCA decreased from 62.74° for pristine PS to 47.78° (Fig. 2B), indicating increased surface hydrophilicity. The lower WCA is consistent with the presence of polar functionalities within the deposited organic material, which can increase the affinity of the surface toward water through interactions involving hydroxyl- and amide-containing groups [33,37]. Thus, the combined FTIR and WCA observations support an SF-induced transformation in both the surface chemistry and wettability of the PS membrane.

3.1.3. Post-operation analysis: HA fouling

In contrast, post-operation characterization following HA filtration revealed a surface transformation distinct from that observed for SF. For the HA-exposed membrane characterized at 5 bar, the WCA increased slightly from 62.74° to 66.92° (Fig. 2B), indicating a modest increase in surface hydrophobicity. FTIR showed a broad O–H band around 3350 cm^{-1}, O–H bending near 1410 cm^{-1}, and aliphatic C–H-associated features around 1468 cm^{-1}, consistent with HA-derived functionalities on the membrane surface [38]. Together, the FTIR and WCA results indicate that HA deposition altered the membrane–water interfacial properties in a manner distinct from SF exposure, although the spectral data do not independently establish the molecular organization of the deposited HA layer [39].

3.1.4. Morphological transformation and foulant-specific deposition

Surface morphological analysis provided complementary qualitative evidence of foulant-specific deposition on the PS membrane (Fig. 3). The pristine PS membrane exhibited a comparatively clean and uniform surface, whereas membranes exposed to either SF or HA showed clear deposition and substantial changes in surface morphology. For the dead-end configuration, the revised SEM comparison was conducted at a fixed pressure of 5 bar, enabling the effects associated with foulant concentration to be examined without simultaneously varying pressure. SF-exposed membranes at 1.5–2.5 g L^{-1} exhibited comparatively fine and dispersed surface deposits, with differences in surface coverage and deposit morphology across the investigated concentrations. In contrast, HA-exposed membranes exhibited more heterogeneous and aggregate-like deposits. These morphological differences are consistent with the known foulant-specific nature of organic membrane fouling, in which chemically distinct macromolecular and humic substances can exhibit different deposition, pore-blocking, and cake-growth behavior [40]. The influence of filtration configuration was further examined under matched conditions at 5 bar using SF at 2.0 g L^{-1} and HA at 0.4 g L^{-1}. For both foulants, the cross-flow samples exhibited deposition patterns that differed from the corresponding dead-end samples. This observation is consistent with the influence of tangential flow and membrane-surface shear on foulant accumulation and concentration polarization during cross-flow filtration [41,42]. However, SEM provides qualitative surface-morphology information rather than quantitative measurements of deposit mass, thickness, mechanical properties, or hydraulic resistance. The SEM observations are therefore interpreted together with the filtration-performance results rather than as independent proof of a specific fouling mechanism.

3.2. Membrane filtration performance

Membrane performance was evaluated according to the procedures previously described. The results are presented to correlate the membrane's physical and chemical characterization with its separation efficacy (flux and rejection) under varied operating pressures and foulant concentrations.

3.2.1. Performance with SF foulant

Filtration experiments utilizing the SF-containing solution demonstrated a distinct pressure-dependent flux behavior. At lower transmembrane pressures (3 and 5 bar), the membrane showed a comparatively smaller time-dependent change in flux, whereas a more pronounced decline was observed at 7 and 10 bar (Fig. 4A). Statistical analysis confirmed that operating pressure significantly affected permeate flux during SF filtration (one-way ANOVA, $F(3,8) = 24.36$, $p < 0.001$), whereas no statistically significant pressure effect was detected for SF rejection ($F(3,8) = 3.36$, $p = 0.076$). Tukey's HSD analysis showed that the flux at 3 bar differed significantly from those at 5, 7, and 10 bar, while the 10-bar flux was also significantly higher than that at 5 bar (Table S3). No significant difference was detected between 5 and 7 bar or between 7 and 10 bar. The pure-water controls also exhibited some pressure-dependent flux decline, particularly at 10 bar (Fig. S1), indicating that intrinsic membrane compaction may contribute to the observed behavior. During SF filtration, the additional time-dependent resistance may further arise from enhanced foulant transport, accumulation, and compression of the deposited organic layer at elevated pressure. Therefore, the observed flux decline is interpreted

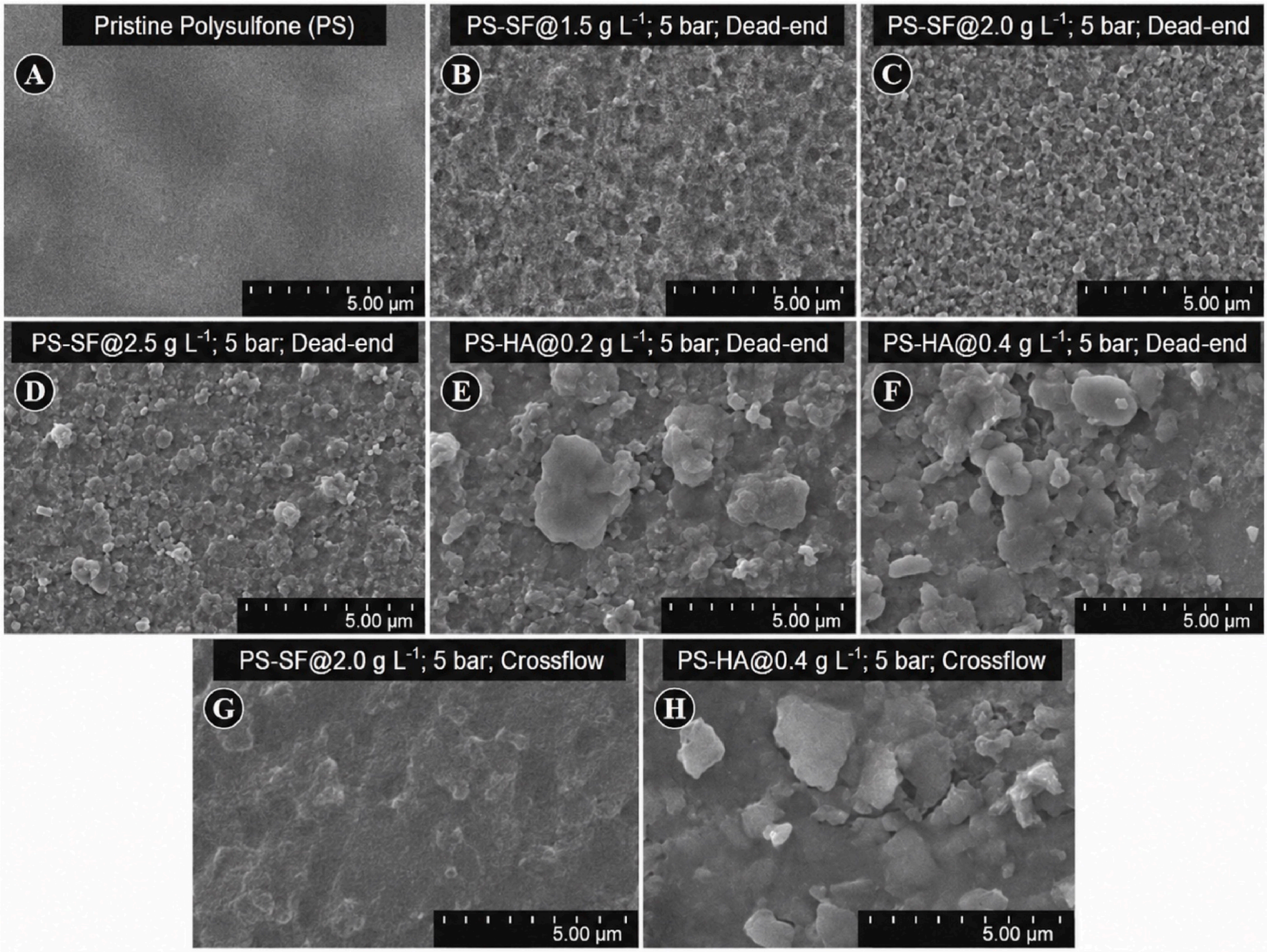

Fig. 3. Pristine and Post-treated Membrane surfaces characterization using SEM at 10,000× magnification.

as the combined effect of pressure-induced membrane response and foulant-associated resistance rather than membrane compaction alone. The protein-, carbohydrate-, and lipid-containing SF matrix may consequently form a compressible surface deposit, with increasing pressure potentially promoting closer packing of the accumulated foulant layer.

Variation in bulk SF concentration produced only limited changes in filtration performance within the investigated range. No statistically significant concentration effect was detected for either permeate flux ($F(2,6) = 3.07$, $p = 0.121$) or rejection ($F(2,6) = 1.89$, $p = 0.231$), and Tukey's HSD analysis identified no significant pairwise differences among 1.5, 2.0, and 2.5 g L^{-1} SF (Tables S2 and S3). These results indicate that, within the investigated concentration range, bulk SF concentration alone did not explain the observed variation in membrane performance. This observation indicates that the fouling mechanism is not exclusively reliant on the bulk foulant concentration. Other significant factors, such as protein-membrane and protein-protein interactions, are crucial determinants of fouling [43]. Moreover, the interactions between proteins and other constituents of the feed matrix, such as carbohydrates, lipids, and ash, are likely to contribute to the structure of the fouling layer. The inherent ash content of SF is known to contain ionized minerals (e.g., Ca, Mg, P, K, and Na), which can influence these interactions [44]. Furthermore, NaCl was incorporated into the feed solution to provide a simplified saline background relevant to low-salinity shrimp aquaculture rather than to reproduce the complete ionic composition of actual RAS water [27]. Ionic composition may influence organic fouling because membrane–foulant and foulant–foulant interactions can change in the presence of dissolved ions [45,46]. In the present study, however, NaCl concentration was fixed at 10 g L^{-1}; therefore, the effect of salinity or ionic strength was not independently investigated. Further studies incorporating systematic ionic-strength variation and multivalent ions are required to clarify ion–protein interactions in this system.

3.2.2. Performance with HA foulant

Using an identical testing methodology, the membrane's performance against the HA foulant was evaluated. The characteristic flux decline observed during HA filtration (Fig. 4D) indicates the progressive development of filtration resistance across the investigated pressures. Such behavior is consistent with the combined effects of concentration polarization, HA accumulation, and the development of additional foulant-associated resistance during ultrafiltration [29,47]. Operating pressure also significantly affected permeate flux during HA filtration ($F(3,8) = 18.47$, $p < 0.001$), whereas HA rejection did not exhibit a significant pressure-dependent response ($F(3,8) = 0.00$, $p = 1.000$) (Table S2). Tukey's HSD analysis showed significantly higher flux at 7 bar than at 3 bar and at 10 bar than at 3 and 5 bar, whereas the remaining pressure comparisons were not statistically significant (Table S3). Increasing pressure increased the absolute permeate flux, while time-dependent flux decline remained evident. Because the

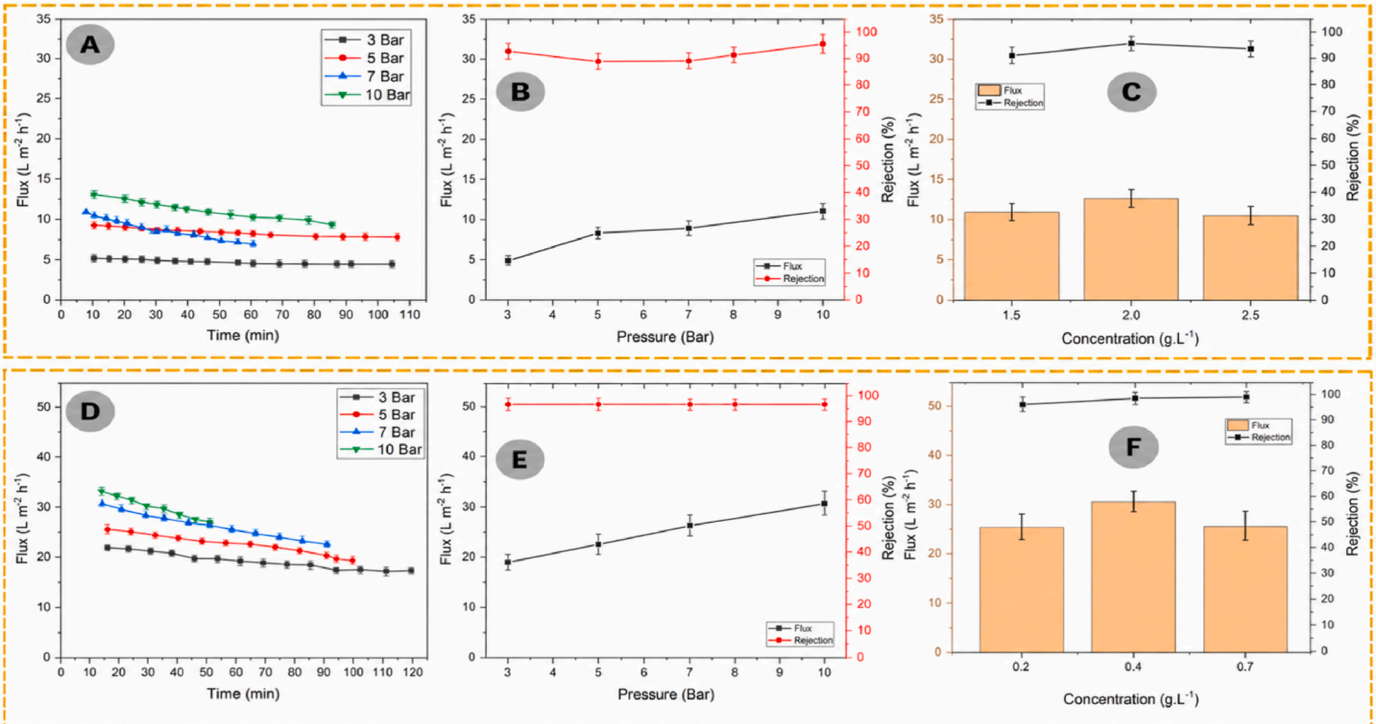

Fig. 4. Dead-end filtration performance of the PS membrane with (A-C) SF and (D-F) HA foulants. (A, D) Flux decline profiles; (B, E) Effect of operating pressure on flux and rejection; (C, F) Effect of feed concentration on flux and rejection.

pure-water controls also showed pressure-related flux changes, particularly at 10 bar, the present data cannot quantitatively distinguish intrinsic membrane compaction from HA-induced resistance. At elevated transmembrane pressure, both pressure-dependent membrane response and compression of accumulated foulant layers may contribute to the observed decline in permeability [47]. The observed behavior is therefore interpreted as resulting from combined contributions of membrane compaction, concentration polarization, and HA deposition. Hydrophobic interactions involving HA can further contribute to persistent or hydraulically irreversible membrane fouling [48]. Despite the decline in flux, the membrane exhibited excellent and consistent rejection performance, maintaining over 90% rejection across the entire pressure range. Similar to the SF trials, filtration performance showed only limited variation with bulk HA concentration within the investigated range.

3.2.3. *Effect of filtration configuration: dead-end vs. cross-flow*

Comparison of dead-end and cross-flow filtration revealed that the

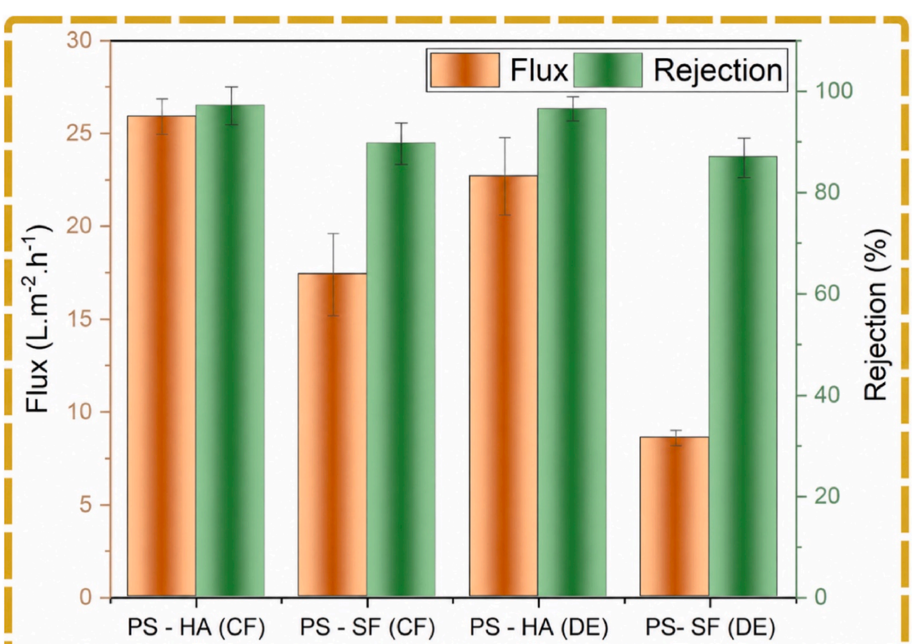

Fig. 5. Comparative Performance of Dead-End (DE) and Cross-Flow (CF) Filtration Modes at 5 bar.

configuration-associated effect on membrane performance depended on foulant type (Fig. 5). Two-way ANOVA showed significant effects of foulant type ($F(1,8) = 147.29$, $p < 0.001$) and filtration configuration ($F(1,8) = 40.87$, $p < 0.001$) on permeate flux, together with a significant foulant configuration interaction ($F(1,8) = 9.21$, $p = 0.016$) (Table S4). The significant interaction indicates that the magnitude of the configuration-associated flux difference was not identical for SF and HA. Subsequent within-foulant comparisons showed that cross-flow filtration produced a significantly higher flux than dead-end filtration for SF ($p = 0.018$), whereas the corresponding difference for HA was not statistically significant ($p = 0.110$) (Table S5). Thus, the configuration-associated enhancement in flux was particularly pronounced for SF. In contrast, filtration configuration did not significantly affect rejection ($F(1,8) = 0.49$, $p = 0.503$), and no significant foulant × configuration interaction was detected for rejection ($F(1,8) = 0.22$, $p = 0.652$). Foulant type itself significantly affected rejection ($F(1,8) = 14.00$, $p = 0.006$), with HA exhibiting higher mean rejection than SF under the investigated conditions. Within-foulant Welch tests likewise showed no significant CF–DE difference in rejection for either HA ($p = 0.855$) or SF ($p = 0.507$) (Tables S4 and S5).

These findings indicate that the principal configuration-associated response was observed in membrane productivity rather than solute rejection. The higher SF flux under cross-flow operation is consistent with reduced foulant accumulation and concentration polarization under tangential flow [49]. However, because the dead-end and cross-flow experiments employed different filtration cells and membrane areas, the statistically observed differences should be interpreted as configuration-associated effects rather than being attributed exclusively to hydrodynamics.

3.3. Practical significance and implications

The present results provide laboratory-scale evidence that operating pressure, foulant identity, and filtration configuration influence the behavior of PS ultrafiltration membranes under aquaculture-relevant model-foulant conditions. Within the investigated pressure range, elevated pressure did not necessarily translate into proportionally improved separation performance and was associated with greater time-dependent filtration resistance, particularly during SF filtration. These observations indicate that increasing transmembrane pressure alone may not be an effective strategy for sustaining UF performance when organic fouling is dominant. However, because energy consumption was not measured, the present study does not permit quantitative conclusions regarding energy efficiency or the energetic optimum of the filtration process.

The cross-flow experiments exhibited higher and more stable permeate flux than the corresponding dead-end experiments under the investigated conditions. This behavior is consistent with the ability of tangential flow to influence concentration polarization and foulant accumulation at the membrane surface. Nevertheless, the two configurations were operated using different filtration cells and membrane areas; therefore, the observed performance difference should not be attributed exclusively to hydrodynamics. The present results consequently support further evaluation of cross-flow operation for fouling control rather than demonstrating a definitive operational advantage for full-scale RAS.

The practical implications of the present study should also be interpreted within the limitations of the model-foulant experimental design. Actual RAS water contains feed- and fecal-derived organic matter, fine suspended and colloidal solids, microbial-derived compounds, dissolved organic matter, nitrogen species, and inorganic constituents that may interact during membrane filtration [23–25]. These multicomponent interactions are not reproduced by the isolated SF and HA solutions used here. Moreover, the elevated foulant concentrations represent accelerated challenge conditions rather than quantitative simulations of actual RAS water. Cleaning frequency, membrane lifetime, energy demand, long-term flux recovery, and operating costs were not evaluated and therefore cannot be inferred from the present experiments.

Accordingly, the findings are best interpreted as a controlled mechanistic basis for identifying operating variables and foulant-specific responses that should be considered in subsequent RAS-oriented UF studies. Validation using actual RAS water or appropriately characterized multicomponent model matrices, together with longer-duration cross-flow operation, cleaning and flux-recovery experiments, and energy assessment, will be required before recommendations regarding full-scale operating pressure, cleaning strategy, or process economics can be established.

4. Conclusions

This study provides a mechanistic understanding of foulant-specific transformations of polysulfone ultrafiltration membranes under controlled aquaculture-relevant model-foulant conditions. Shrimp-feed exposure produced an organic surface deposit containing proteinaceous and polysaccharide-like functionalities and was accompanied by increased membrane hydrophilicity, whereas HA exposure produced more heterogeneous aggregate-like deposits together with a modest increase in surface hydrophobicity. Elevated pressures were associated with greater filtration resistance for both foulant systems, with a more pronounced deterioration in flux stability observed during SF filtration at 7–10 bar. Pure-water controls indicated that intrinsic pressure-dependent membrane compaction may also contribute at high pressure; therefore, the observed flux decline cannot be attributed exclusively to foulant deposition. Cross-flow filtration exhibited higher and more stable flux than dead-end filtration under the investigated conditions, although differences in module geometry and membrane area prevent attribution of this performance difference exclusively to hydrodynamics. Collectively, these findings provide a controlled mechanistic basis for understanding foulant-specific UF behavior relevant to aquaculture water treatment; however, validation using actual RAS water is required before the observed trends can be extrapolated to long-term or full-scale operation.

CRediT authorship contribution statement

Candra Wijaya: Writing – original draft, Project administration, Methodology, Formal analysis, Conceptualization. **Putu Doddy Sutrisna:** Writing – review & editing, Supervision, Resources, Methodology, Conceptualization. **Pra Cipta Buana Wahyu Mustika:** Writing – review & editing, Investigation, Data curation, Conceptualization. **Marchis Wonder Limahardja:** Writing – review & editing, Investigation, Formal analysis. **Ivania Handoyo:** Writing – review & editing, Investigation, Formal analysis.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work the author(s) used ChatGPT 5.0 and Gemini 2.5 Pro in order to improve the language and writing. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.cscee.2026.101478.

Notation

RAS	:	Recirculating Aquaculture Systems	
PS	:	Polysulfone	
PVDF	:	Polyvinylidene fluoride	
PP	:	Polypropylene	
PES	:	Polyethersulfone	
SF	:	Shrimp Feed	
HA	:	Humic Acid	
Q	:	Flow rate	$L.h^{-1}$
T	:	Temperature	°C
P	:	Pressure	bar/kPa/mPa
S	:	Agitation speed	rpm
D	:	Diameter	m
J	:	Permeate Flux	$L.m^{-2}.h^{-1}$
V	:	Volume	L
A	:	Active area	m^2
t	:	Time	h
R	:	Rejection	%
C_P	:	Permeate Concentration	$g.L^{-1}$
C_F	:	Feed Concentration	$g.L^{-1}$
RS	:	Stokes Radii	nm
RP	:	Pore Radii	nm
MWCO	:	Molecular Weight Cut-Off	Da

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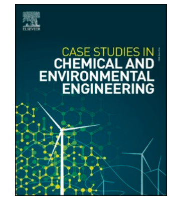

Foulant-specific transformations of polysulfone membrane: A mechanistic study of shrimp feed and humic acid fouling with implications for Recirculating Aquaculture Systems (RAS)

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ABSTRACT

The integration of ultrafiltration (UF) into recirculating aquaculture systems (RAS) is a promising strategy for managing organic wastewater contaminants, yet its efficacy is hindered by severe membrane fouling and physico-chemical transformation. This study investigates these challenges by evaluating polysulfone (PS) membrane under dead-end and cross-flow hydrodynamics. Performance was systematically assessed using shrimp feed (SF; 1.5–2.5 g L^{-1}) and humic acid (HA; 0.2–0.7 g L^{-1}) as aquaculture-relevant model foulants under varied operating pressures. The single-foulant systems were employed to isolate foulant-specific membrane responses under controlled conditions rather than to reproduce the complete physicochemical composition of actual RAS water. Pristine and fouled membranes were characterized (MWCO, SEM, FTIR, contact angle) to elucidate fouling mechanisms. Post-operation analyses revealed distinct, foulant-specific surface transformations. Higher operating pressures were associated with more pronounced time-dependent flux deterioration, particularly during SF filtration, whereas HA maintained consistently high rejection across the investigated pressure range. Furthermore, Cross-flow operation exhibited higher mean flux than dead-end filtration under the investigated conditions, with the configuration-associated enhancement being particularly pronounced for SF.

1. Introduction

The growth of the aquaculture industry continues to increase which simultaneously raises the demand for sustainable aquaculture wastewater management [1,2]. The Recirculating Aquaculture System (RAS) is an attractive option for contaminant removal through a series of integrated processes, which results in a reduction in the amount of water consumption [3–5]. The primary objective of this process is to eliminate any unwanted substances and pathogens for water recycling purposes [6–8]. The uneaten feed and the animal excretions need to be minimized as both are ammonia contributors in aquaculture systems and are harmful to the animals living in the system [9,10].

It is commonly known that organic contaminant in aquaculture
23 systems have diverse characteristics and sizes and are distributed randomly. Thus, conventional processes such as classification or centrifugation may not be effective in handling them [11]. In addition, the presence of protein in the system can lead to the formation of colloids, thus making the coagulation-flocculation process as one of the
12 potential techniques to solve this problem [12]. However, it is indispensable to minimize additional chemicals in the system. Therefore, the membrane filtration process which has no additional chemicals arises as an alternative to removing contaminants. Among the variety of pore size available, microfiltration (0.1 – 10 μm) and ultrafiltration (0.001 – 0.01 μm) have the potential to replace the coagulation – flocculation process [13]. In addition, nanofiltration and reverse osmosis can also be used in aquaculture systems, although both processes generally require more energy than microfiltration or ultrafiltration [10,14,15]. Besides, Yong et al. have applied membrane distillation in aquaculture systems, which showed success in recovering ammonia up to 465 mg L^{-1} (~23 times the initial concentration at around 19.83 mg L^{-1}) [16]. Simultaneously, almost all of the ammonia is rejected, resulting in a high separation factor of 148 (only 0.04 mg L^{-1} of ammonia is able to pass through the membrane).

Despite the advantages, the implementation of membrane processes in aquaculture remains limited by several challenges, including (1) low membrane selectivity, (2) high operating costs, (3) membrane fouling,

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and (4) membrane degradation [17]. Among those obstacles, membrane fouling is the most difficult barrier to overcome. In addition, various factors also affect the occurrence of membrane fouling namely: (1) wastewater characteristics, (2) operating conditions, (3) membrane properties [18,19].

The membrane properties, such as porosity, tortuosity, hydrophilicity, surface charge, pore size, and other physical properties, are crucial in separation processes. In general, membranes with hydrophilic properties are always preferred in aqueous-based separations, which cannot be separated from their high surface wettability and anti-fouling ability [20,21]. In addition, membranes with high surface wettability produce high flux without reducing their selectivity [22]. Furthermore, with certainty, the size ratio between contaminants and pore size can determine the existing fouling mechanism [18]. Overall, the relationship between membrane surface charge and isoelectric points of contaminants is affected by pH. Up to now, the electrostatic repulsion between the membrane and the contaminants has resulted in lower fouling and increased flux [18].

Several membrane fouling studies are summarized in Table S1, indicating that fouling behavior is strongly dependent on membrane properties, foulant characteristics, solution chemistry, and operating conditions. Actual RAS water contains a complex mixture of feed- and fecal-derived organic matter, fine suspended solids and colloids, microbial-derived compounds, dissolved organic matter, nitrogen species, and inorganic constituents [23–25]. Feed input and feed-derived particles have been directly associated with increased membrane fouling in marine RAS, while the continuous recirculation of water promotes the accumulation and transformation of dissolved organic matter, including humic- and fulvic-type compounds [23,24]. Consequently, a single synthetic solution cannot reproduce the complete fouling environment of an operating RAS. Nevertheless, controlled model-foulant systems are useful for isolating membrane responses to chemically distinct foulant classes, and previous aquaculture membrane studies have employed shrimp-feed-derived organic matter and humic acid for this purpose [26]. Therefore, in this study, shrimp feed (SF) and humic acid (HA) were employed as aquaculture-relevant model foulants representing feed-derived and humic-type organic matter, respectively. The objective was to investigate their individual effects on the filtration performance and physicochemical properties of a commercial polysulfone ultrafiltration membrane under controlled laboratory conditions, rather than to reproduce the complete composition of actual RAS wastewater. The effects of operating pressure, foulant concentration, and filtration configuration were evaluated, while MWCO, SEM, FTIR, and water contact angle analyses were used to characterize the corresponding membrane transformations.

2. Material and methods

2.1. Materials

Commercial polysulfone (PS) ultrafiltration membranes with a nominal molecular weight cut-off (MWCO) of 20,000 Da were supplied by RisingSun Membrane Technology. Shrimp feed (SF; IRAWAN 681 V, Central Proteina Prima Indonesia) and humic acid (HA; Sigma-Aldrich) were used as aquaculture-relevant model foulants. The shrimp feed contained protein (>30%), crude fiber (<4%), fat (>5%), moisture (<12%), and ash (<11%). Sodium chloride (NaCl), hydrochloric acid (HCl), sulfuric acid (H_2SO_4), phenol (C_6H_5OH), glucose ($C_6H_{12}O_6$), sucrose ($C_{12}H_{22}O_{11}$), and dextran standards with molecular weights of 1–40 kDa were used for feed preparation and membrane characterization. Deionized water produced in the laboratory, with a conductivity of approximately 15 μS cm^{-1} and total dissolved solids of approximately 11 mg L^{-1}, was used throughout the experiments.

2.2. Methods

2.2.1. Filtration feed preparation

Shrimp feed was ground and sieved to obtain particles smaller than 200 mesh. The sieved particles were then dispersed in deionized water at concentrations of 1.5, 2.0, and 2.5 g L^{-1}. The suspensions were homogenized using an IKA T25 digital ULTRA-TURRAX® at 20,000 rpm for 5 min at 25 ± 2 °C, followed by filtration to remove residual large particles. The resulting filtrate was exposed to UV-C irradiation to minimize microbial growth before use in the filtration experiments. Humic acid solutions were prepared separately at concentrations of 0.2, 0.4, and 0.7 g L^{-1}. NaCl was added to each feed solution at 10 g L^{-1} to provide a simplified saline background relevant to low-salinity shrimp aquaculture conditions [27]. The NaCl-based medium was used to control ionic background and was not intended to reproduce the complete ionic composition of actual RAS water.

SF and HA were investigated separately as single-foulant challenge solutions to enable controlled comparison of their individual membrane-fouling behavior. Previous aquaculture membrane studies have employed residual shrimp feed and HA as model organic foulants [26]. However, the SF concentrations employed in the present study (1.5–2.5 g L^{-1}) are higher than the 0.3–0.8 g L^{-1} shrimp-feed range previously investigated by Widiasa et al. [26]. Similarly, the HA concentrations used here (0.2–0.7 g L^{-1}) are within or above the upper range of high-concentration model-fouling studies; for example, HA concentrations of 50–150 mg L^{-1} and 500 mg L^{-1} have been employed in accelerated membrane-fouling evaluations [28,29]. Accordingly, the present concentrations are interpreted as accelerated fouling conditions designed for comparative mechanistic evaluation rather than as environmentally representative concentrations of actual RAS water. Therefore, the resulting fouling rates should not be quantitatively extrapolated to full-scale RAS operation.

2.2.2. Filtration procedure

The filtration experiments were organized into three experimental sets (See Fig. 1). The effect of operating pressure was evaluated under stirred dead-end filtration at 3, 5, 7, and 10 bar using SF at 2.0 g L^{-1} or HA at 0.4 g L^{-1}. The effect of foulant concentration was evaluated under dead-end filtration at a fixed pressure of 5 bar using SF concentrations of 1.5, 2.0, and 2.5 g L^{-1} and HA concentrations of 0.2, 0.4, and 0.7 g L^{-1}. Filtration-configuration effects were evaluated separately at 5 bar by comparing dead-end and cross-flow operation using SF at 2.0 g L^{-1} and HA at 0.4 g L^{-1}. All experiments were conducted at 25 ± 2 °C.

Prior to filtration, PS membrane coupons were immersed in deionized water for 24 h. Pure-water filtration was subsequently performed to establish the baseline membrane response. Dead-end filtration was conducted using a stirred cell with an effective membrane area of 15.9 cm^2, with N_2 gas providing the transmembrane pressure and continuous stirring at 500 rpm. Cross-flow experiments were conducted using a Sterlitech Explorer CF042P cell with an effective membrane area of approximately 42 cm^2 and a hold-up volume of 17 mL. The feed was recirculated at approximately 1.0 L min^{-1}, corresponding to an average cross-flow velocity of approximately 0.18 m s^{-1}, calculated from the volumetric recirculation rate and the cross-sectional area of the flow channel. Prior to foulant filtration, pure-water flux was measured using the pristine PS membrane under the corresponding operating pressures. For dead-end filtration, pure-water measurements were conducted at 3, 5, 7, and 10 bar to establish the pressure-dependent baseline membrane response. These measurements were used as clean-water controls for distinguishing pressure-related changes in membrane permeability from additional resistance arising during foulant filtration. The corresponding pure-water flux profiles are provided in Fig. S1. The upper pressure of 10 bar was included as a high-pressure challenge condition to evaluate the membrane response over an extended operating range and should not be interpreted as a recommended operating pressure for RAS ultrafiltration.

Case Studies in Chemical and Environmental Engineering 14 (2026) 101478

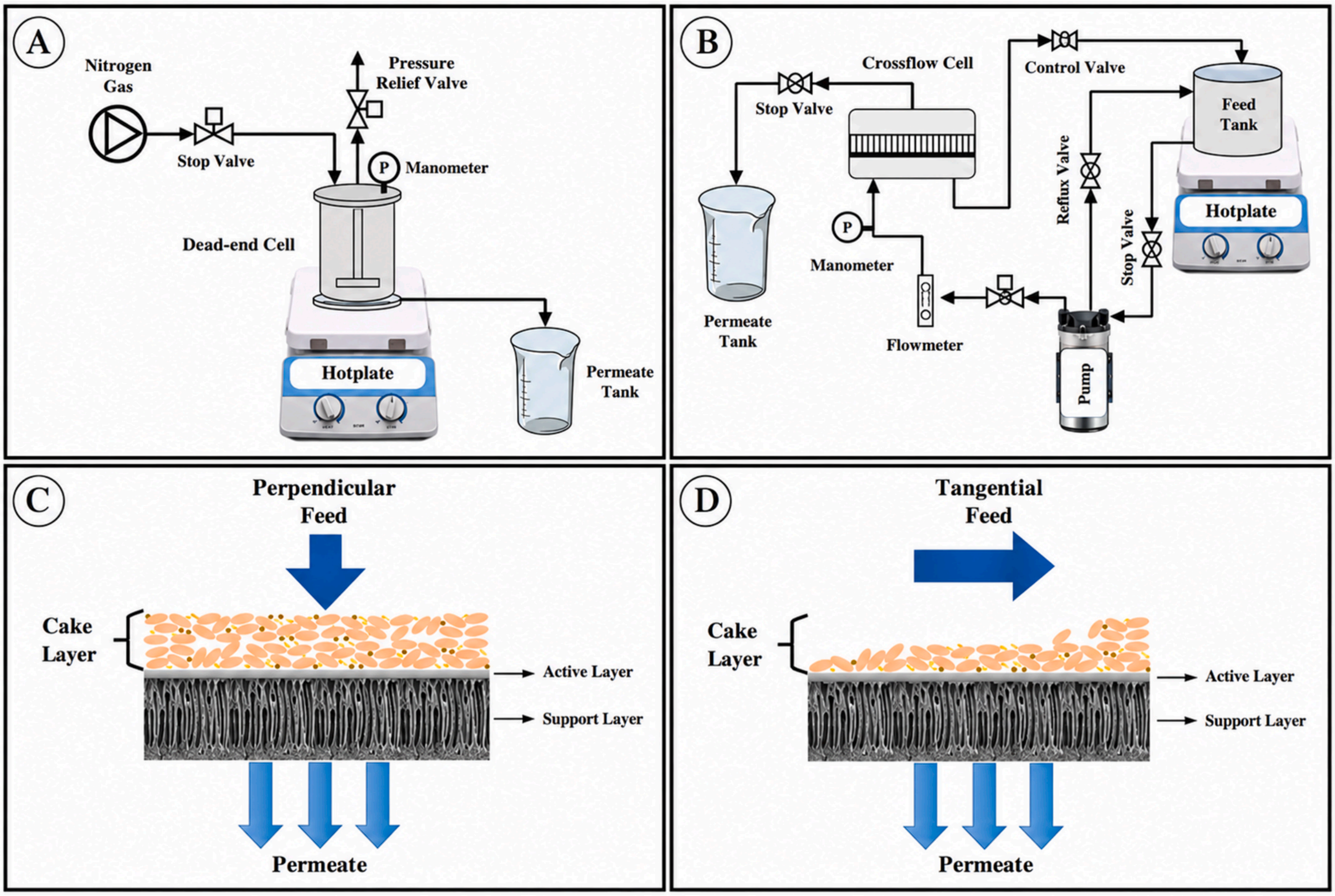


Fig. 1. Experimental setups and corresponding separation mechanisms for (a, c) dead-end stirred and (b, d) cross-flow filtration modes.

Permeate volume and the corresponding elapsed filtration time were recorded throughout filtration and used to calculate permeate flux according to Eq. (1).

$$J = \frac{V}{A.t} \tag{1}$$

where V, t, A are volume permeate (L), time (h), active membrane area (m^2), respectively.

Filtration duration varied according to the permeation behavior of each condition rather than being fixed across all experiments. For the PS cross-flow experiments conducted at 5 bar, cumulative permeate collection reached 80 mL after approximately 63.9 min for SF and 50.9 min for HA. The initial feed volume required to determine conventional permeate recovery could not be reliably reconstructed from the archived experimental records; therefore, recovery was not used as a basis for quantitative interpretation of the filtration results.

Foulant concentrations in the initial feed and permeate were quantified by UV–Vis spectrophotometry using foulant-specific calibration curves. SF and HA were analyzed at wavelengths of 280 and 255 nm, respectively, following the UV–Vis approach previously applied for aquaculture-relevant organic foulants [26]. For SF, the calibration relationship was $A = 0.0754C - 0.0053$ ($R^2 = 0.999$), whereas for HA the relationship was $A = 3.6623C - 0.0343$ ($R^2 = 0.998$), where A represents absorbance and C represents the corresponding foulant concentration (g L^{-1}). The concentrations determined for the initial feed and permeate were used as C_F and C_P, respectively, to calculate rejection according to Eq. (2).

$$R = \left(1 - \frac{C_P}{C_F}\right) \times 100\% \tag{2}$$

where C_P and C_F are the permeate concentration (g.L^{-1}) and the initial feed concentration (g.L^{-1}), respectively.

2.2.3. *Membrane characterization*

The membranes were systematically characterized to determine their pore size, hydrophobicity, morphology, and surface chemistry. Pore size was evaluated by the molecular weight cut-off (MWCO), defined as the molecular weight at 90% solute rejection. These tests were conducted at 25 °C and 5 bar using 200 mg/L organic solute solutions. Solute concentrations were quantified via the phenol-sulfuric acid method and measured with a UV-Vis spectrophotometer [Thermo Scientific™, Evolution™ 60S]. Membrane surface morphology was visualized by Scanning Electron Microscopy (SEM) [Hitachi, Flexsem 1000]. For SEM characterization, the SF concentration series (1.5, 2.0, and 2.5 g L^{-1}) and HA concentration series (0.2 and 0.4 g L^{-1}) were obtained under dead-end filtration at a fixed pressure of 5 bar. Configuration-associated morphology was evaluated at 5 bar using SF at 2.0 g L^{-1} and HA at 0.4 g L^{-1} under both dead-end and cross-flow operation. Surface hydrophobicity was determined by static water contact angle measurements [DataPhysics Instruments, OCA25]. Meanwhile, surface functional groups were identified using Fourier Transform Infrared (FTIR) spectroscopy [Shimadzu, IRTracer–100] over a wavenumber range of 4000–450 cm^{-1}. Post-filtration WCA and FTIR analyses were performed using PS membrane samples obtained at 5 bar. The SF-exposed sample corresponded to filtration with 2.0 g L^{-1} SF, whereas the HA-exposed sample corresponded to filtration with 0.4 g L^{-1} HA.

33 *2.2.4. Statistical analysis*

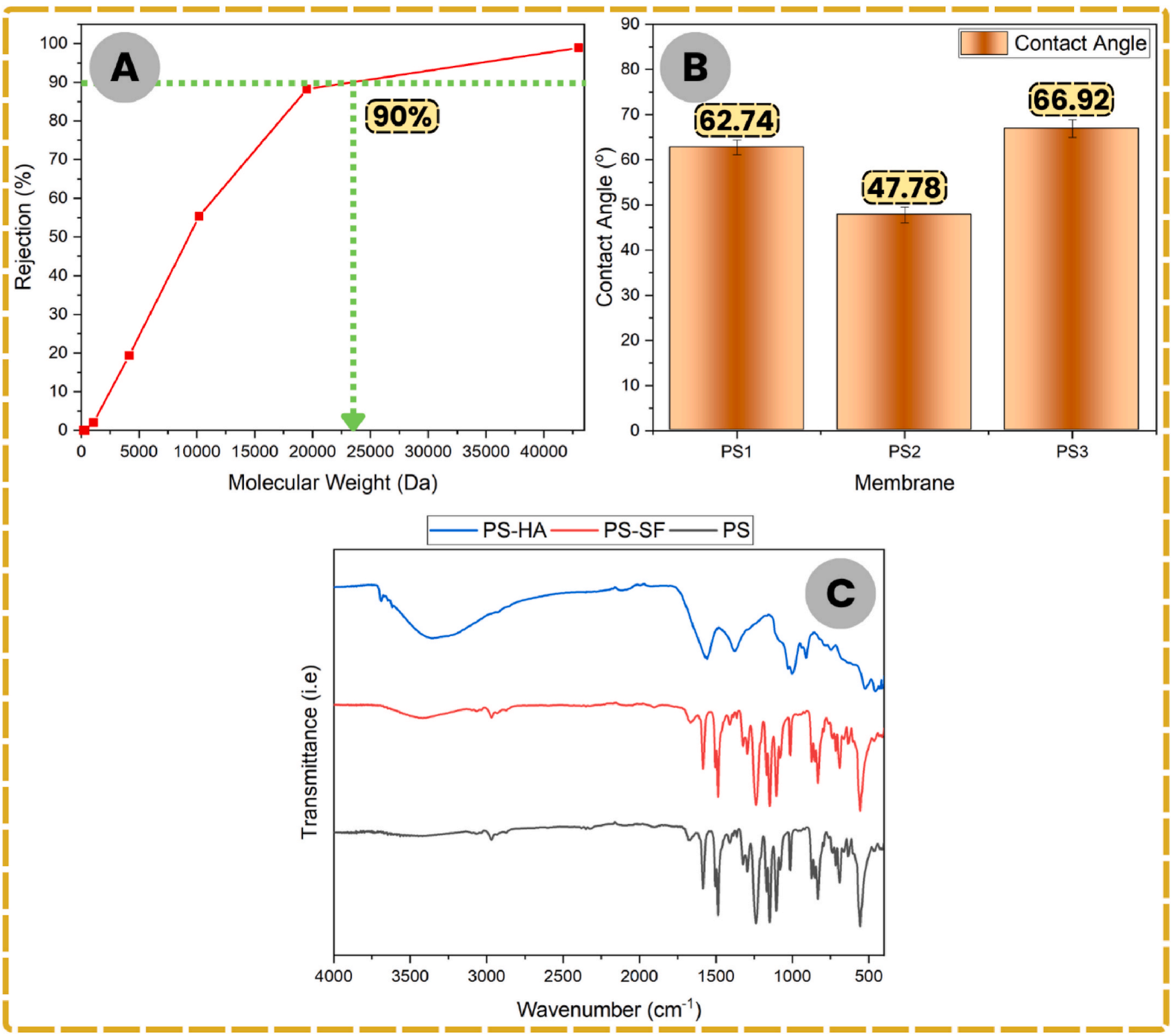
Quantitative filtration-performance experiments were performed in triplicate. Statistical analyses were performed using MATLAB R2025b
34 (MathWorks, USA), with statistical significance defined at $p < 0.05$. For the dead-end filtration experiments, the effects of operating pressure and foulant concentration on permeate flux and rejection were evalu-
10 ated separately for SF and HA using one-way analysis of variance (ANOVA), followed by Tukey's honestly significant difference (HSD) test for pairwise comparisons. For the comparison between dead-end and
30 cross-flow filtration, two-way ANOVA was used to evaluate the effects of foulant type, filtration configuration, and their interaction on flux and rejection. Within-foulant comparisons between cross-flow and dead-end filtration were additionally evaluated using Welch's independent-
49 samples *t*-test. Detailed statistical results are provided in Table S2–S5 of the Supplementary Data.

48

3. Results and discussion

3.1. Correlation of membrane characteristics with fouling phenomena

As previously mentioned, the characteristics of membranes are crucial for the successful implementation of ultrafiltration (UF) in the treatment of aquaculture wastewater. This analysis provides a comprehensive examination of the specific roles played by pore size, hydrophilicity, and surface chemistry [30,31]. The pore size of the membrane, indicated by its Molecular Weight Cut-Off (MWCO), serves as the principal mechanism for selectivity, operating through a size exclusion process.[32]. Nevertheless, the pore structure alone does not determine performance within this complex matrix. Membrane hydrophilicity is equally significant; a highly hydrophilic surface tends to reduce the adsorption of organic foulants, primarily proteins and carbohydrates, which are prevalent in aquaculture systems. This surface wettability is, in turn, influenced by the functional groups present on the membrane



35 **Fig. 2.** (A) Rejection curve for MWCO determination of the pristine PS membrane at 5 bar; (B) water contact angle of pristine PS (PS1), PS after filtration with 2.0 g L^{-1} SF at 5 bar (PS2), and PS after filtration with 0.4 g L^{-1} HA at 5 bar (PS3); and (C) FTIR spectra of the corresponding pristine, SF-exposed, and HA-exposed PS membranes.

surface.[30,31]. The interdependence among these three factors is crucial for comprehending the overall processes of separation and fouling.

3.1.1. Pristine membrane characterization

Characterization of the pristine PS UF membrane established its baseline properties. The MWCO was experimentally determined to be ~24 kDa (Fig. 2A), which is in close agreement with the manufacturer's reported value of 20 kDa. The slight discrepancy is likely attributable to differences in measurement methodologies and operating conditions. This MWCO confirms the membrane's theoretical capacity to reject large macromolecules, such as proteins and carbohydrates, via size exclusion [33].

Analysis by FTIR (Fig. 2C) confirmed the characteristic functional groups of the PS polymer. Key peaks were identified for the sulfone group (O═S═O), with symmetric and asymmetric stretching vibrations observed at 1159 cm^{-1} and 1231 cm^{-1}, respectively [34]. Other significant peaks included the asymmetric C-O-C stretch of the aromatic ether at ~1248 cm^{-1}, aromatic C═C stretching at 1682 cm^{-1}, and aromatic C-H bending in the fingerprint region at 849 cm^{-1}. Aliphatic C-H stretching from methyl groups was also detected at 2869 cm^{-1} [35].

Wettability analysis (Fig. 2B) showed the pristine membrane surface to be moderately hydrophilic, leaning towards hydrophobic, with a water contact angle (WCA) of 62.74°. This inherent wettability is attributed to the sulfone (O═S═O) and ether (C-O-C) groups [34]. The oxygen atoms in these groups possess lone electron pairs, allowing the molecules as hydrogen bond acceptors for water molecules.

3.1.2. Post-operation analysis: SF-induced organic fouling

Membrane autopsy following operation with SF revealed a distinct surface transformation associated with the deposition of feed-derived organic material. As shown in Fig. 2C, several new FTIR features emerged after SF filtration, indicating changes in the chemical composition of the membrane surface. A prominent band at approximately 1550 cm^{-1} is consistent with Amide II-associated vibrations, involving N–H bending and C–N stretching, while the feature observed in the Amide III region at approximately 1350 cm^{-1} provides additional evidence of proteinaceous material associated with the fouled surface [36]. The broad O–H stretching band at approximately 3200 cm^{-1}, together with the C–O-associated feature around 1050 cm^{-1}, is consistent with hydroxyl- and carbohydrate/polysaccharide-like functionalities. FTIR-based characterization of organic membrane fouling similarly associates amide- and carbohydrate-related spectral regions with proteinaceous and polysaccharide-containing foulants [36].

Collectively, these spectral changes support the deposition of an SF-derived organic layer containing proteinaceous and polysaccharide-like components. However, FTIR identifies chemical functionalities and does not by itself establish the three-dimensional organization of these components as a protein–polysaccharide gel layer. The corresponding change in membrane wettability provides complementary evidence of surface transformation. For the SF-exposed membrane characterized after filtration at 5 bar, the WCA decreased from 62.74° for pristine PS to 47.78° (Fig. 2B), indicating increased surface hydrophilicity. The lower WCA is consistent with the presence of polar functionalities within the deposited organic material, which can increase the affinity of the surface toward water through interactions involving hydroxyl- and amide-containing groups [33,37]. Thus, the combined FTIR and WCA observations support an SF-induced transformation in both the surface chemistry and wettability of the PS membrane.

3.1.3. Post-operation analysis: HA fouling

In contrast, post-operation characterization following HA filtration revealed a surface transformation distinct from that observed for SF. For the HA-exposed membrane characterized at 5 bar, the WCA increased slightly from 62.74° to 66.92° (Fig. 2B), indicating a modest increase in surface hydrophobicity. FTIR showed a broad O–H band around 3350 cm^{-1}, O–H bending near 1410 cm^{-1}, and aliphatic C–H-associated features around 1468 cm^{-1}, consistent with HA-derived functionalities on the membrane surface [38]. Together, the FTIR and WCA results indicate that HA deposition altered the membrane–water interfacial properties in a manner distinct from SF exposure, although the spectral data do not independently establish the molecular organization of the deposited HA layer [39].

3.1.4. Morphological transformation and foulant-specific deposition

Surface morphological analysis provided complementary qualitative evidence of foulant-specific deposition on the PS membrane (Fig. 3). The pristine PS membrane exhibited a comparatively clean and uniform surface, whereas membranes exposed to either SF or HA showed clear deposition and substantial changes in surface morphology. For the dead-end configuration, the revised SEM comparison was conducted at a fixed pressure of 5 bar, enabling the effects associated with foulant concentration to be examined without simultaneously varying pressure. SF-exposed membranes at 1.5–2.5 g L^{-1} exhibited comparatively fine and dispersed surface deposits, with differences in surface coverage and deposit morphology across the investigated concentrations. In contrast, HA-exposed membranes exhibited more heterogeneous and aggregate-like deposits. These morphological differences are consistent with the known foulant-specific nature of organic membrane fouling, in which chemically distinct macromolecular and humic substances can exhibit different deposition, pore-blocking, and cake-growth behavior [40]. The influence of filtration configuration was further examined under matched conditions at 5 bar using SF at 2.0 g L^{-1} and HA at 0.4 g L^{-1}. For both foulants, the cross-flow samples exhibited deposition patterns that differed from the corresponding dead-end samples. This observation is consistent with the influence of tangential flow and membrane-surface shear on foulant accumulation and concentration polarization during cross-flow filtration [41,42]. However, SEM provides qualitative surface-morphology information rather than quantitative measurements of deposit mass, thickness, mechanical properties, or hydraulic resistance. The SEM observations are therefore interpreted together with the filtration-performance results rather than as independent proof of a specific fouling mechanism.

3.2. Membrane filtration performance

Membrane performance was evaluated according to the procedures previously described. The results are presented to correlate the membrane's physical and chemical characterization with its separation efficacy (flux and rejection) under varied operating pressures and foulant concentrations.

3.2.1. Performance with SF foulant

Filtration experiments utilizing the SF-containing solution demonstrated a distinct pressure-dependent flux behavior. At lower transmembrane pressures (3 and 5 bar), the membrane showed a comparatively smaller time-dependent change in flux, whereas a more pronounced decline was observed at 7 and 10 bar (Fig. 4A). Statistical analysis confirmed that operating pressure significantly affected permeate flux during SF filtration (one-way ANOVA, $F(3,8) = 24.36$, $p < 0.001$), whereas no statistically significant pressure effect was detected for SF rejection ($F(3,8) = 3.36$, $p = 0.076$). Tukey's HSD analysis showed that the flux at 3 bar differed significantly from those at 5, 7, and 10 bar, while the 10-bar flux was also significantly higher than that at 5 bar (Table S3). No significant difference was detected between 5 and 7 bar or between 7 and 10 bar. The pure-water controls also exhibited some pressure-dependent flux decline, particularly at 10 bar (Fig. S1), indicating that intrinsic membrane compaction may contribute to the observed behavior. During SF filtration, the additional time-dependent resistance may further arise from enhanced foulant transport, accumulation, and compression of the deposited organic layer at elevated pressure. Therefore, the observed flux decline is interpreted

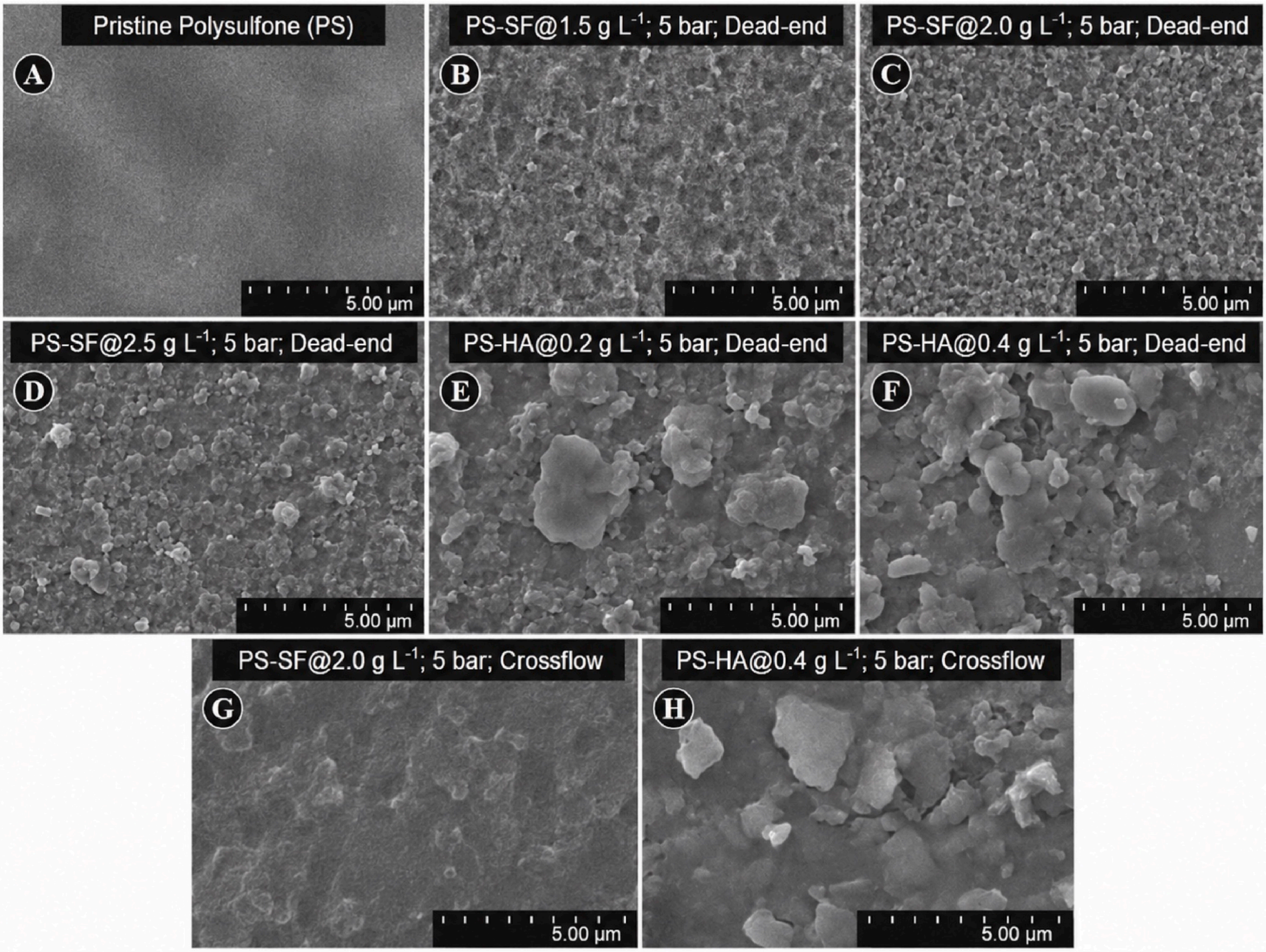


Fig. 3. Pristine and Post-treated Membrane surfaces characterization using SEM at 10,000× magnification.

as the combined effect of pressure-induced membrane response and foulant-associated resistance rather than membrane compaction alone. The protein-, carbohydrate-, and lipid-containing SF matrix may
29 consequently form a compressible surface deposit, with increasing pressure potentially promoting closer packing of the accumulated foulant layer.

Variation in bulk SF concentration produced only limited changes in filtration performance within the investigated range. No statistically significant concentration effect was detected for either permeate flux ($F(2,6) = 3.07$, $p = 0.121$) or rejection ($F(2,6) = 1.89$, $p = 0.231$), and Tukey's HSD analysis identified no significant pairwise differences among 1.5, 2.0, and 2.5 g L^{-1} SF (Tables S2 and S3). These results indicate that, within the investigated concentration range, bulk SF concentration alone did not explain the observed variation in membrane performance. This observation indicates that the fouling mechanism is not exclusively reliant on the bulk foulant concentration. Other significant factors, such as protein-membrane and protein-protein interactions, are crucial determinants of fouling [43]. Moreover, the interactions between proteins and other constituents of the feed matrix,
51 such as carbohydrates, lipids, and ash, are likely to contribute to the structure of the fouling layer. The inherent ash content of SF is known to contain ionized minerals (e.g., Ca, Mg, P, K, and Na), which can influence these interactions [44]. Furthermore, NaCl was incorporated into the feed solution to provide a simplified saline background relevant to low-salinity shrimp aquaculture rather than to reproduce the complete ionic composition of actual RAS water [27]. Ionic composition may influence organic fouling because membrane–foulant and foulant–foulant interactions can change in the presence of dissolved ions [45,46]. In the present study, however, NaCl concentration was fixed at 10 g L^{-1}; therefore, the effect of salinity or ionic strength was not independently investigated. Further studies incorporating systematic ionic-strength variation and multivalent ions are required to clarify ion–protein interactions in this system.

3.2.2. *Performance with HA foulant*

Using an identical testing methodology, the membrane's performance against the HA foulant was evaluated. The characteristic flux decline observed during HA filtration (Fig. 4D) indicates the progressive development of filtration resistance across the investigated pressures. Such behavior is consistent with the combined effects of concentration polarization, HA accumulation, and the development of additional foulant-associated resistance during ultrafiltration [29,47]. Operating pressure also significantly affected permeate flux during HA filtration ($F(3,8) = 18.47$, $p < 0.001$), whereas HA rejection did not exhibit a significant pressure-dependent response ($F(3,8) = 0.00$, $p = 1.000$) (Table S2). Tukey's HSD analysis showed significantly higher flux at 7 bar than at 3 bar and at 10 bar than at 3 and 5 bar, whereas the remaining pressure comparisons were not statistically significant (Table S3). Increasing pressure increased the absolute permeate flux, while time-dependent flux decline remained evident. Because the

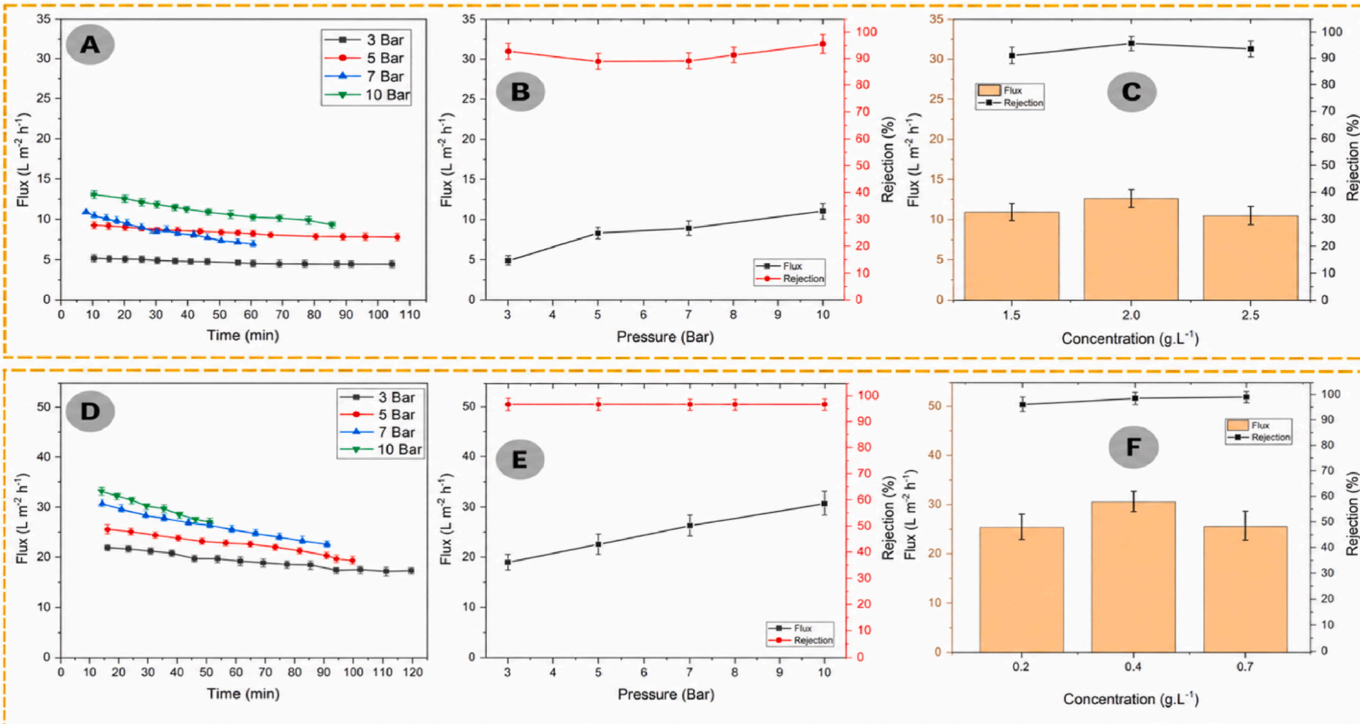


Fig. 4. Dead-end filtration performance of the PS membrane with (A-C) SF and (D-F) HA foulants. (A, D) Flux decline profiles; (B, E) Effect of operating pressure on flux and rejection; (C, F) Effect of feed concentration on flux and rejection.

pure-water controls also showed pressure-related flux changes, particularly at 10 bar, the present data cannot quantitatively distinguish intrinsic membrane compaction from HA-induced resistance. At elevated transmembrane pressure, both pressure-dependent membrane response and compression of accumulated foulant layers may contribute to the observed decline in permeability [47]. The observed behavior is therefore interpreted as resulting from combined contributions of membrane compaction, concentration polarization, and HA deposition.

Hydrophobic interactions involving HA can further contribute to persistent or hydraulically irreversible membrane fouling [48]. Despite the decline in flux, the membrane exhibited excellent and consistent rejection performance, maintaining over 90% rejection across the entire pressure range. Similar to the SF trials, filtration performance showed only limited variation with bulk HA concentration within the investigated range.

3.2.3. Effect of filtration configuration: dead-end vs. cross-flow

Comparison of dead-end and cross-flow filtration revealed that the

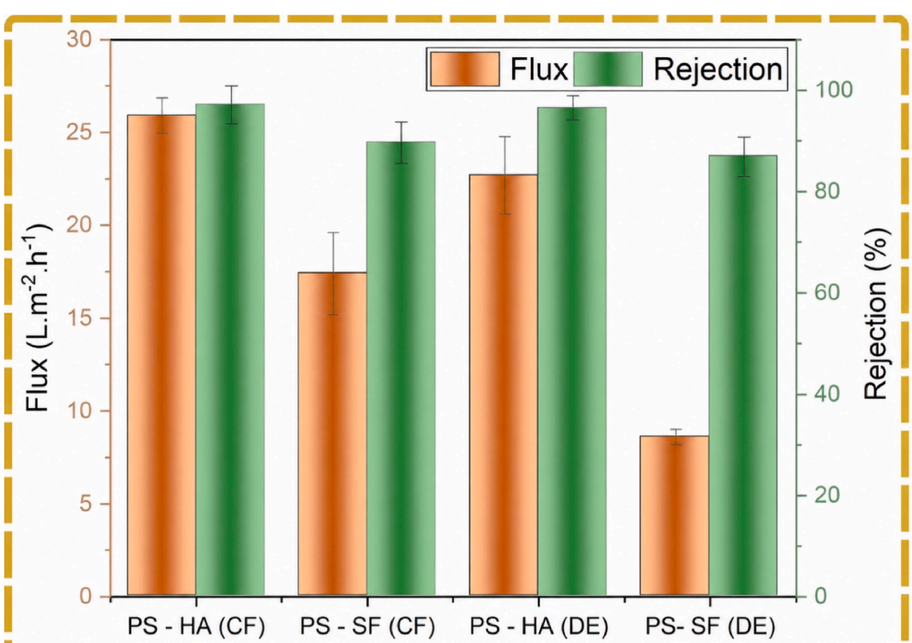


Fig. 5. Comparative Performance of Dead-End (DE) and Cross-Flow (CF) Filtration Modes at 5 bar.

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configuration-associated effect on membrane performance depended on foulant type (Fig. 5). Two-way ANOVA showed significant effects of foulant type ($F(1,8) = 147.29$, $p < 0.001$) and filtration configuration ($F(1,8) = 40.87$, $p < 0.001$) on permeate flux, together with a significant foulant configuration interaction ($F(1,8) = 9.21$, $p = 0.016$) (Table S4). The significant interaction indicates that the magnitude of the configuration-associated flux difference was not identical for SF and HA. Subsequent within-foulant comparisons showed that cross-flow filtration produced a significantly higher flux than dead-end filtration for SF ($p = 0.018$), whereas the corresponding difference for HA was not statistically significant ($p = 0.110$) (Table S5). Thus, the configuration-associated enhancement in flux was particularly pronounced for SF. In contrast, filtration configuration did not significantly affect rejection ($F(1,8) = 0.49$, $p = 0.503$), and no significant foulant × configuration interaction was detected for rejection ($F(1,8) = 0.22$, $p = 0.652$). Foulant type itself significantly affected rejection ($F(1,8) = 14.00$, $p = 0.006$), with HA exhibiting higher mean rejection than SF under the investigated conditions. Within-foulant Welch tests likewise showed no significant CF–DE difference in rejection for either HA ($p = 0.855$) or SF ($p = 0.507$) (Tables S4 and S5).

These findings indicate that the principal configuration-associated response was observed in membrane productivity rather than solute rejection. The higher SF flux under cross-flow operation is consistent with reduced foulant accumulation and concentration polarization under tangential flow [49]. However, because the dead-end and cross-flow experiments employed different filtration cells and membrane areas, the statistically observed differences should be interpreted as configuration-associated effects rather than being attributed exclusively to hydrodynamics.

3.3. Practical significance and implications

The present results provide laboratory-scale evidence that operating pressure, foulant identity, and filtration configuration influence the behavior of PS ultrafiltration membranes under aquaculture-relevant model-foulant conditions. Within the investigated pressure range, elevated pressure did not necessarily translate into proportionally improved separation performance and was associated with greater time-
9 dependent filtration resistance, particularly during SF filtration. These observations indicate that increasing transmembrane pressure alone may not be an effective strategy for sustaining UF performance when organic fouling is dominant. However, because energy consumption was not measured, the present study does not permit quantitative conclu-
2 sions regarding energy efficiency or the energetic optimum of the filtration process.

22 The cross-flow experiments exhibited higher and more stable permeate flux than the corresponding dead-end experiments under the investigated conditions. This behavior is consistent with the ability of tangential flow to influence concentration polarization and foulant
7 accumulation at the membrane surface. Nevertheless, the two configurations were operated using different filtration cells and membrane areas; therefore, the observed performance difference should not be attributed exclusively to hydrodynamics. The present results conse-
6 quently support further evaluation of cross-flow operation for fouling control rather than demonstrating a definitive operational advantage for full-scale RAS.

40 The practical implications of the present study should also be interpreted within the limitations of the model-foulant experimental design. Actual RAS water contains feed- and fecal-derived organic matter, fine suspended and colloidal solids, microbial-derived com-
2 pounds, dissolved organic matter, nitrogen species, and inorganic constituents that may interact during membrane filtration [23–25]. These multicomponent interactions are not reproduced by the isolated SF and
5 HA solutions used here. Moreover, the elevated foulant concentrations represent accelerated challenge conditions rather than quantitative simulations of actual RAS water. Cleaning frequency, membrane lifetime, energy demand, long-term flux recovery, and operating costs were not evaluated and therefore cannot be inferred from the present experiments.

Accordingly, the findings are best interpreted as a controlled mechanistic basis for identifying operating variables and foulant-specific responses that should be considered in subsequent RAS-oriented UF studies. Validation using actual RAS water or appropriately characterized multicomponent model matrices, together with longer-duration cross-flow operation, cleaning and flux-recovery experiments, and energy assessment, will be required before recommendations regarding full-scale operating pressure, cleaning strategy, or process economics can be established.

4. Conclusions

This study provides a mechanistic understanding of foulant-specific transformations of polysulfone ultrafiltration membranes under controlled aquaculture-relevant model-foulant conditions. Shrimp-feed exposure produced an organic surface deposit containing proteinaceous and polysaccharide-like functionalities and was accompanied by increased membrane hydrophilicity, whereas HA exposure produced more heterogeneous aggregate-like deposits together with a modest increase in surface hydrophobicity. Elevated pressures were associated with greater filtration resistance for both foulant systems, with a more pronounced deterioration in flux stability observed during SF filtration at 7–10 bar. Pure-water controls indicated that intrinsic pressure-dependent membrane compaction may also contribute at high pressure; therefore, the observed flux decline cannot be attributed exclusively to foulant deposition. Cross-flow filtration exhibited higher and more stable flux than dead-end filtration under the investigated conditions, although differences in module geometry and membrane area prevent attribution of this performance difference exclusively to hydrodynamics. Collectively, these findings provide a controlled mechanistic basis for understanding foulant-specific UF behavior relevant to aquaculture water treatment; however, validation using actual RAS water is required before the observed trends can be extrapolated to long-term or full-scale operation.

CRediT authorship contribution statement

Candra Wijaya: Writing – original draft, Project administration, Methodology, Formal analysis, Conceptualization. **Putu Doddy Sutrisna:** Writing – review & editing, Supervision, Resources, Methodology, Conceptualization. **Pra Cipta Buana Wahyu Mustika:** Writing – review & editing, Investigation, Data curation, Conceptualization. **Marchis Wonder Limahardja:** Writing – review & editing, Investigation, Formal analysis. **Ivania Handoyo:** Writing – review & editing, Investigation, Formal analysis.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work the author(s) used ChatGPT 5.0 and Gemini 2.5 Pro in order to improve the language and writing. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

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Declaration of competing interest

The authors declare that they have no known competing financial

2 interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

2 Supplementary data to this article can be found online at https://doi.org/10.1016/j.cscee.2026.101478.

Notation

RAS	:	Recirculating Aquaculture Systems	
PS	:	Polysulfone	
PVDF	:	Polyvinylidene fluoride	
PP	:	Polypropylene	
PES	:	Polyethersulfone	
SF	:	Shrimp Feed	
HA	:	Humic Acid	
Q	:	Flow rate	L.h^{-1}
T	:	Temperature	°C
P	:	Pressure	bar/kPa/mPa
S	:	Agitation speed	rpm
D	:	Diameter	m
J	:	Permeate Flux	L.m^{-2}.h^{-1}
V	:	Volume	L
A	:	Active area	m^2
t	:	Time	h
R	:	Rejection	%
C_P	:	Permeate Concentration	g.L^{-1}
C_F	:	Feed Concentration	g.L^{-1}
RS	:	Stokes Radii	nm
RP	:	Pore Radii	nm
MWCO	:	Molecular Weight Cut-Off	Da

18

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Foulant-specific transformations of polysulfone membrane: A mechanistic study of shrimp feed and humic acid fouling with implications for Recirculating Aquaculture Systems (RAS)

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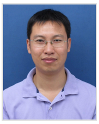

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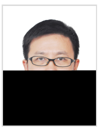

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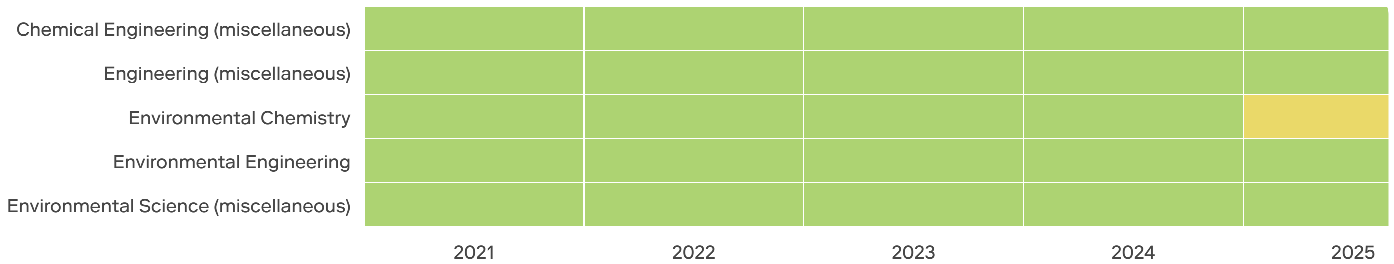

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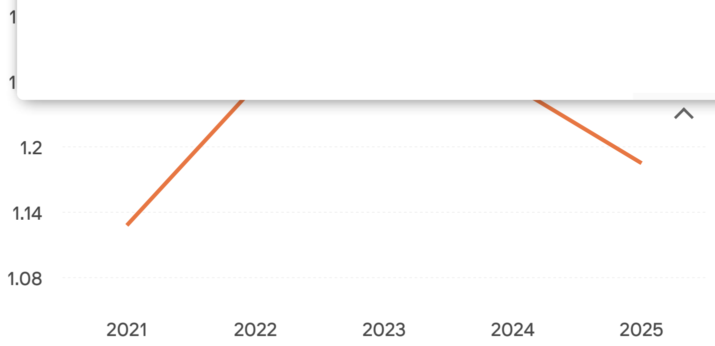

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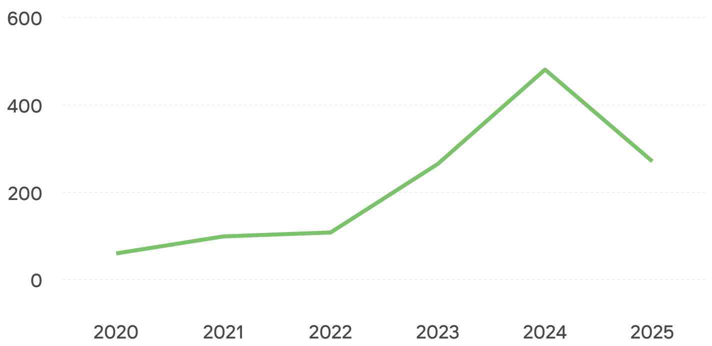

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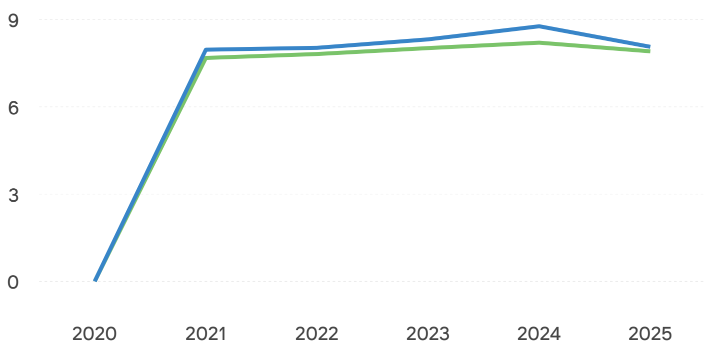

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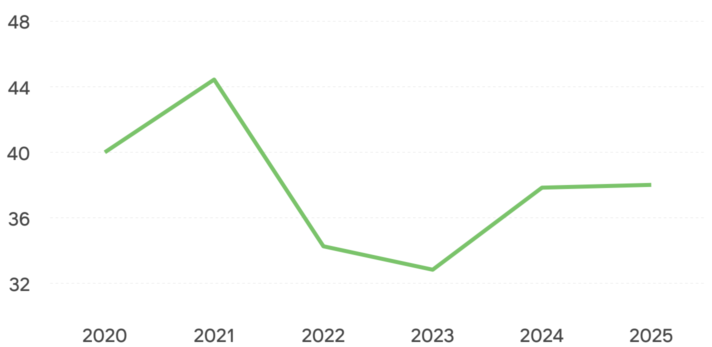

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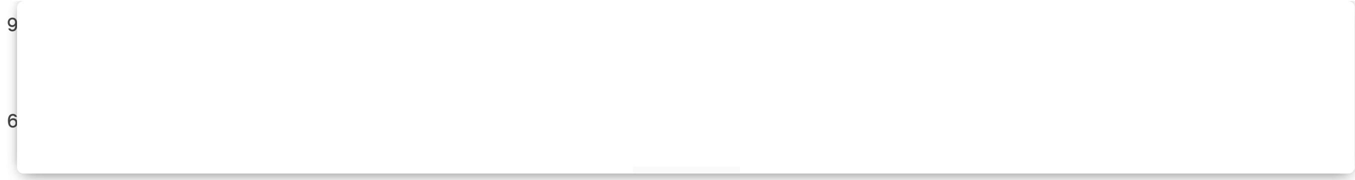

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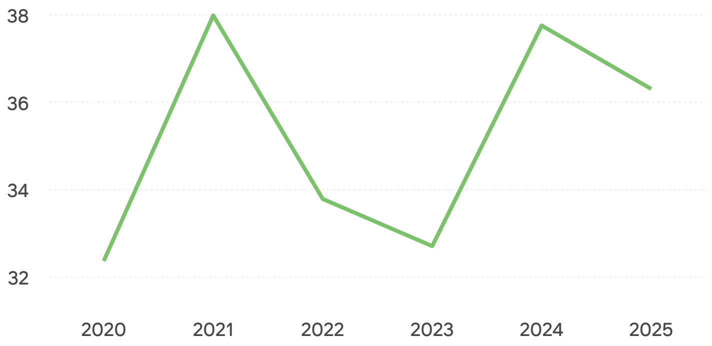

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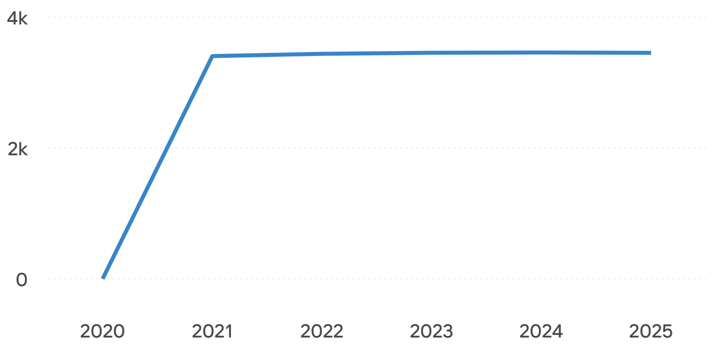

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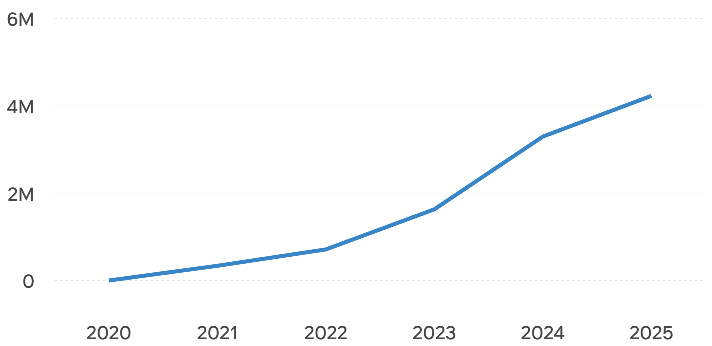

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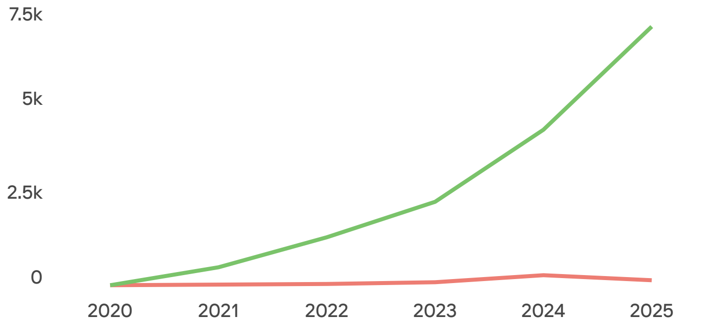

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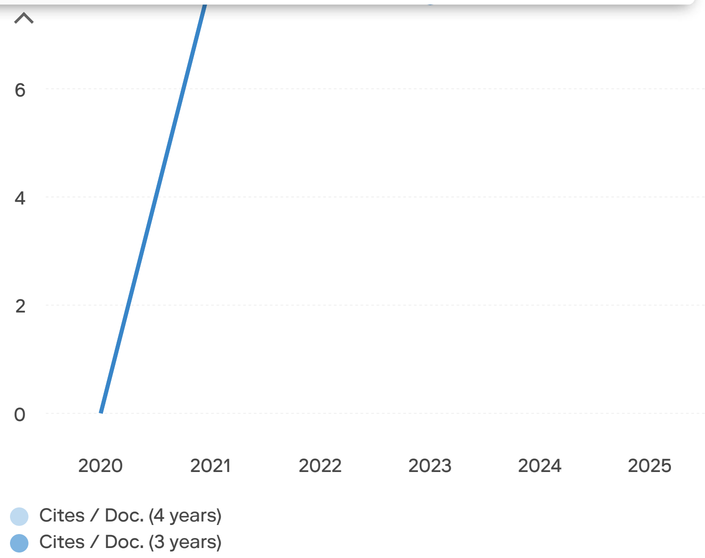
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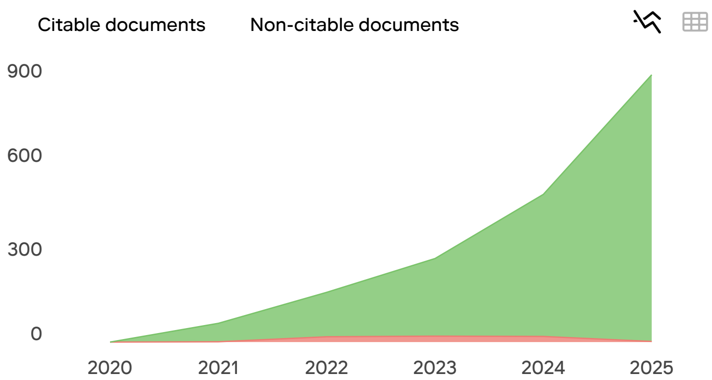
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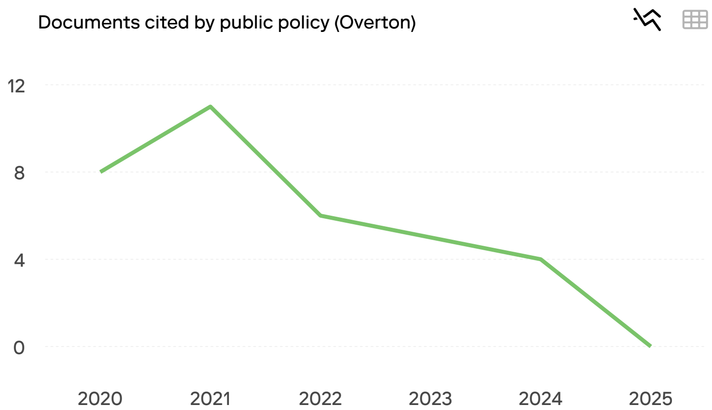
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Case Studies in Chemical and Environmental Engineering • Article • *Open Access* • 2026 •
DOI: 10.1016/j.cscee.2026.101478

Wijaya, Candra ; Sutrisna, Putu Doddy ; Mustika, Pra Cipta Buana Wahyu ; Limahardja, Marchis Wonder ; Handoyo, Ivania
Department of Chemical Engineering, Universitas Surabaya (UBAYA), Surabaya, Indonesia

Abstract

The integration of ultrafiltration (UF) into recirculating aquaculture systems (RAS) is a promising strategy for managing organic wastewater contaminants, yet its efficacy is hindered by severe membrane fouling and physico-chemical transformation. This study investigates these challenges by evaluating polysulfone (PS) membrane under dead-end and cross-flow hydrodynamics. Performance was systematically assessed using shrimp feed (SF; 1.5–2.5 $g\,L^{-1}$) and humic acid (HA; 0.2–0.7 $g\,L^{-1}$) as aquaculture-relevant model foulants under varied operating pressures. The single-foulant systems were employed to isolate foulant-specific membrane responses under controlled conditions rather than to reproduce the complete physicochemical composition of actual RAS water. Pristine and fouled membranes were characterized (MWCO, SEM, FTIR, contact angle) to elucidate fouling mechanisms. Post-operation analyses revealed distinct, foulant-specific surface transformations. Higher operating pressures were associated with more pronounced time-dependent flux deterioration, particularly during SF filtration, whereas HA maintained consistently high rejection across the investigated pressure range. Furthermore, Cross-flow operation exhibited higher mean flux than dead-end filtration under the investigated conditions, with the configuration-associated enhancement being particularly pronounced for SF.

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I have completed my evaluation of your manuscript. The reviewers recommend reconsideration of your manuscript following revision. I invite you to resubmit your manuscript after addressing the comments below. Please resubmit your revised manuscript by **Aug 18, 2026**.

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Editor and Reviewer Comments:

Reviewer #1: The manuscript addresses a relevant topic by comparing shrimp-feed- and humic-acid-induced fouling of polysulfone ultrafiltration membranes under different pressures and hydrodynamic conditions. The combination of filtration performance with SEM, FTIR, contact-angle, and MWCO analyses is potentially useful. However, substantial concerns remain regarding the representativeness of the simulated RAS conditions, incomplete reporting of the experimental design and cross-flow parameters, unclear foulant quantification methods, insufficient statistical analysis and replication, and overinterpretation of the fouling mechanisms and practical implications. Therefore, I recommend major revision before the manuscript can be considered for publication.
1. The manuscript separately disperses shrimp feed and humic acid in deionized water and adds 10 g/L NaCl to simulate aquaculture water. However, real RAS water typically contains a complex mixture of feed residues, excreta, microbial metabolites, proteins, polysaccharides, inorganic particles, ammonium nitrogen, and various ions. Therefore, the use of only two single-model foulant systems is insufficient to support the claims regarding "RAS application" and commercial-scale operation.
2. The experimental design and cross-flow operating parameters are incompletely reported.
3. The Methods section provides only the rejection equation but does not explain how Cp and Cf were measured for SF and HA. This is a critical issue affecting the reliability of the results.
4. The Abstract states that: "High operating pressure significantly exacerbated fouling by SF, a phenomenon not observed with HA." However, Section 3.2.2 suggests that HA underwent more severe compaction and pore blockage at elevated pressures, while the Conclusions state that:
"Elevated pressures (7-10 bar) intensified both types of fouling."These three statements are clearly incons[illegible]t.
5. The SEM images in Fig. 3 were not obtained under fully matched experimental conditions. For example, both pressure and concentration were varied for the SF samples, different concentrations were also used for the HA samples, and the cross-flow samples were tested under another set of operating conditions. Therefore, the observed morphological differences cannot be attributed independently to pressure, concentration, or filtration mode.
6. Table 1 is overly lengthy and uses inconsistent language and units. It should be substantially condensed or moved to the Supplementary Materials.

7. The graphical abstract does not effectively highlight the core findings of the study and should be redesigned.

Reviewer #2: The manuscript investigated fouling of a commercial polysulfone ultrafiltration membrane using shrimp feed and humic acid. The comparison between two foulants was potentially relevant to aquaculture wastewater treatment. Surface characterization also provided useful observations of the deposited layers. However, the simulated solutions were not demonstrated to represent actual RAS wastewater. The dead-end and cross-flow experiments were conducted using different devices without sufficient hydrodynamic information. Replicate experiments and statistical analyses were not reported. Membrane compaction was also not separated from foulant resistance at high pressure. Several mechanistic conclusions therefore exceeded the available evidence. Therefore, I suggest the manuscript has to be reviewed after a Major Revision. The specific comments are as follows:

Major Concerns:
1) Page 3, Lines 60-70. The manuscript was submitted as a case or pilot study, but the experiments used artificial solutions in laboratory filtration cells. No full-scale RAS application was examined.
2) Page 4, Lines 88-92. The selected foulant concentrations were not justified using actual RAS water-quality data. Their environmental relevance therefore remained unclear.
3) Page 4, Lines 95-106. The cross-flow velocity was not reported. This parameter was necessary for interpreting concentration polarization and comparing the two filtration configurations.
4) Page 4, Lines 95-106. Dead-end and cross-flow experiments used different membrane areas and apparatuses. The resulting flux differences could not be attributed solely to hydrodynamics.
5) Page 4, Lines 95-111. The experimental duration and permeate recovery were not reported. These factors directly affected the observed flux decline and foulant concentration.
6) Page 4, Lines 94-111. The manuscript did not report experimental replicates or statistical analysis. Claims of significant differences were therefore unsupported.
7) Page 4, Lines 96-104. Pressures up to 10 bar were unusually high for ultrafiltration. No clean-water control was presented to separate membrane compaction from fouling.
8) Page 5, Lines 113-122. The post-operation sampling conditions were unclear. The manuscript did not specify which pressure or filtration mode corresponded to each characterized membrane.
9) Page 5, Lines 155-170. The FTIR assignments alone did not demonstrate the formation of a protein-polysaccharide gel layer. The claimed layer composition required more direct evidence.
10) Page 9, Lines 247-260. The practical claims concerning energy consumption and cleaning intervals were not supported by measured data. These outcomes were not evaluated in the experiments.

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Dear Dr. Wijaya,

We would like to remind you that on **Jul 28, 2026** we asked you to revise your above referenced manuscript and your revision is due by **Aug 18, 2026**.

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Kind regards,
Long Nghiem, PhD
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Editor and Reviewer Comments:

Reviewer #1: The authors have carefully addressed my comments. I think this work can now be accepted for publication.

Reviewer #2: The authors adequately addressed the previous concerns. The revised manuscript clarified the experimental scope and strengthened the statistical analysis. The reconstructed SEM comparison and pure-water controls also improved the reliability of the findings. However, several presentation issues remained, particularly the manuscript classification, language errors, broken cross-reference, and graphical abstract. Therefore, I suggest the manuscript has to be reviewed after a Minor Revision. The specific comments are as follows:

Page 2, Lines 43-44. The expression "are also can be used" was grammatically incorrect.
Page 2, Lines 49-53. "The challenges to implementing membranes remain struggling on" was grammatically incorrect and unclear.
Page 3, Line 63. "Error! Reference source not found" remained in the revised manuscript.
Graphical Abstract. The image showed typical characteristics associated with AI-generated content and required careful redrawing. If AI-generated elements were retained, their use should comply with journal requirements and be formally declared.

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