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Membrane autopsy for fouling mitigation in reverse osmosis process of wastewater secondary effluent in a sewer mining plant

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ABSTRACT

Reverse osmosis (RO) plays a critical role in sewer mining plants for the reclamation of high-quality water from wastewater. However, membrane fouling remains a major operational challenge that limits the long-term performance and economic viability of RO systems in such applications. In this study, a comprehensive autopsy of fouled RO membrane elements from a full-scale sewer mining plant was performed to characterise foulant composition and elucidate fouling mechanisms. The results revealed a pronounced spatial distribution of foulants along the RO train, with the first element in the lead stage exhibiting more severe fouling than the tail elements due to higher hydraulic loading and contaminant exposure. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) analyses indicated that the foulant layers consisted of heterogeneous organic and inorganic deposits and biofilms. Additionally, total organic carbon (TOC) measurements and elemental analysis of foulant extracts confirmed the variation in foulant composition across elements. Cover densities of TOC, Si and Al foulants on the first membrane element were 1.6, 1.7, and 7.6-fold, respectively, higher than those on the last element. This spatial distribution of various foulant composition emphasised the necessity for combined acid and alkaline chemical cleaning strategies to achieve effective fouling control.

1. Introduction

The increasing global water scarcity and stricter environmental regulations have intensified the need for sustainable and locally sourced water solutions (Gebreslassie et al., 2024). Sewer mining, which involves extracting wastewater directly from sewers for on-site treatment and reuse, has emerged as a practical approach to decentralised freshwater reclamation and augmentation (Makropoulos et al., 2018; Rafaai et al., 2025). In sewer minging, reverse osmosis (RO) plays a critical role given its exceptional capability to remove dissolved salts, organic contaminants, trace chemicals, and microbial pathogens, producing high-quality freshwater suitable for non-potable and even indirect potable uses (Plevri et al., 2017, 2018). Integrating RO into sewer mining systems enables efficient freshwater recovery, reduces reliance on centralised supplies, and minimises environmental discharge, thus aligning with the principles of circularity in the water sectors (Wang et al., 2022; Cicekalan et al., 2025).

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Despite these advantages, membrane fouling remains one of the most vexing challenges limiting the widespread adoption of RO technology in sewer mining (Tay et al., 2020; Liu et al., 2020; Li et al., 2025). In sewer mining plants, RO is deployed to treat effluent from a secondary wastewater treatment process such as membrane bioreactors (MBRs) (Liu et al., 2020; Lee et al., 2021). Fouling occurs when organic matter, biofilms, inorganic salts, and particulate material remained in the effluent accumulate on the RO membrane surface and possibly within membrane pores, inevitably leading to water flux decline, increased operating pressures, more frequent membrane cleaning, and shortened membrane lifespan (Li et al., 2023; Liu et al., 2024; Tran et al., 2023). The complexity of fouling in the RO process of secondary wastewater effluent arises from the heterogeneous composition of the effluent, which often contains a mix of biodegradable organics, microorganisms, colloidal particles, and dissolved salts. Such complexity makes membrane fouling prediction and mitigation particularly challenging in the RO application in sewer mining (Ahmed et al., 2023; Chen et al., 2022; Matin et al., 2021).

To better understand and mitigate fouling, membrane autopsy has become an essential investigative approach in various RO applications (Park et al., 2023; Philibert et al., 2024). This post-operation analysis involves a suite of microscopic, spectroscopic, and chemical methods to characterise the fouling layer and identify foulant types. Common techniques include scanning electron microscopy (SEM) for morphological examination, energy-dispersive X-ray spectroscopy (EDS) for elemental analysis, Fourier-transform infrared spectroscopy (FTIR) for organic compound identification, and microbial assays for biofouling detection. Detailed characterisation of the fouling layer from membrane autopsy provides the information about the composition and distribution of fouling. This information is essential for understanding site-specific fouling behaviour and hence developing effective cleaning and pre-treatment protocols.

While previous studies have focused on autopsies of membranes treating seawater or brackish water (Philibert et al., 2024; Adel et al., 2022), there remains a relative paucity of research specifically addressing fouling in RO membranes exposed to secondary effluent in sewer mining. For example, Gonzalez-Gil et al (Gonzalez-Gil et al., 2021). proposed autopsy protocols and conducted autopsies of fouled membranes from a full-scale seawater RO desalination plant. The autopsy results revealed homogenous foulant layer consisted of organic matters, microbial cells, and inorganic scales in which iron was predominant. Complex mixture of organic and inorganic foulants was also reported in an autopsy study of fouled membranes in a brackish water RO desalination plant; however, the foulant layer mostly consisted of organic matters and aluminium silicate crystals which are prevalent in brackish water sources (Tran et al., 2007). These autopsy studies highlight the profound impacts of feed water sources on the formation and behaviour of foulant in the RO process. Given the complex and variable nature of wastewater, research on autopsy fouled membranes from RO treatment of wastewater secondary effluent are needed for process optimisation and fouling mitigation.

This study aims to enrich the knowledge of membrane fouling by conducting a comprehensive autopsy of fouled commercial RO membranes in the treatment of wastewater secondary effluent from MBRs in a sewer mining plant in Sydney, Australia. Using multiple analytical techniques and operational data, this study identified dominant fouling mechanisms and their sources. These insights are beneficial for developing effective cleaning protocols, improving membrane design, and optimising operations for RO sewer mining

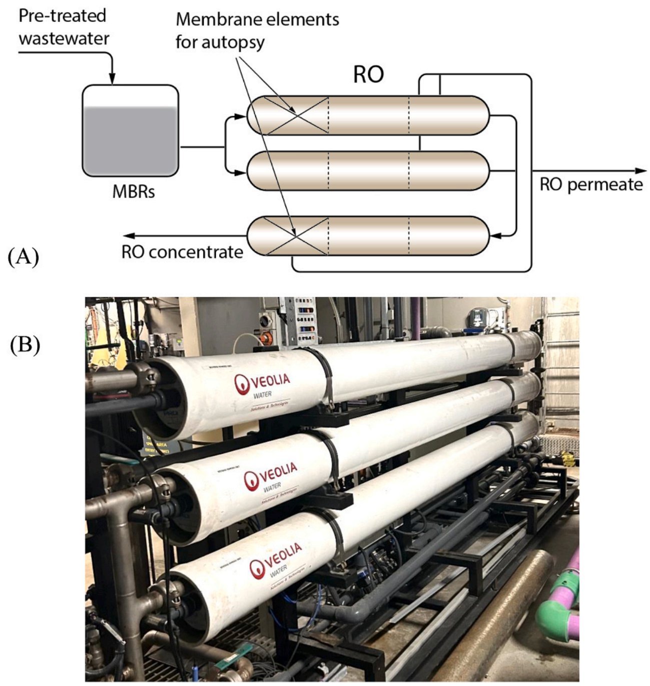

Fig. 1. (A) The schematic diagram and (B) the real image of the RO membrane elements deployed at the sewer mining plant. The RO membrane elements were configured in 2 stages, each stage was operated at 50 % water recovery, resulting in 75 % overall water recovery.

applications. Ultimately, the findings from this study might assist in enhancing the reliability, cost-effectiveness, and long-term sustainability of RO-based sewer minging and wastewater reclamation systems.

2. Materials and methods

2.1. Sewer mining plant and RO system description

Fouled RO membrane elements for autopsy were obtained from a sewer mining plant in a commercial building in Sydney, Australia. The plant processed wastewater generated within the building to produce high-quality recycled water for on-site non-potable reuse. The treatment chain included an MBR system equipped with submerged ultrafiltration (UF) membrane, followed by an RO system, UV disinfection, and chlorination. The sewer mining plant and the RO membranes had been in operation for 5 years prior to this autopsy study.

The RO system (Fig. 1) consisted of two stages: the first stage contained 6 membrane elements, and the second stage had 3 membrane elements (in spiral-wound configuration). The manufacture and operating specifications of RO membrane elements are provided in Table 1. Each stage was operated at water recovery of 50 %, resulting in the overall RO system recovery rate of 75 %. The system included antiscalant addition, pH adjustment (when required), and an automated clean-in-place (CIP) for membrane chemical cleaning. Prior to extracting RO elements for autopsy, the system was flushed with RO permeate water for 15 min, depressurised, and then all membrane elements were dismantled from the membrane housings. The first RO element of the first stage and the last one of the second stage were obtained for autopsy in this study (Fig. 1 A).

2.2. Membrane autopsy procedure

The fouled RO membrane elements were cut using a dry saw to prevent surface contamination. Approximately 100 cm^2 of flat-sheet membrane samples were obtained from each element for analysis. The membrane samples were stored in sealed containers at 4 °C and used for analysis within one week.

To evaluate their compositions, foulants were extracted from the fouled RO membranes using nitric acid (HNO_3) or sodium hydroxide (NaOH) solutions following the same procedures. Briefly, fouled membrane samples of 25 cm^2 were cut into smaller pieces (i.e. 1 cm^2 each) and submerged into 100 mL of 0.5 M HNO_3 or 0.5 M NaOH. Membrane pieces and the extracting solution were then sonicated for 90 min so that all foulants were extracted and dissolved into the extracting solution. After sonication, 50 mL liquid sample was collected from the extracting solution and proceeded to TOC and elemental analysis.

2.3. Analytical methods

Scanning electron microscopy (SEM) was used to assess the surface morphology of the fouled RO membranes. Membrane samples (~1 cm^2) were gradually dehydrated and then mounted on a sample holder. A thin gold layer was sputter-coated (Quorum Q150VS plus sputter coater, UK) on the membrane surface to ensure conductivity. The gold-coated membranes were examined using a Zeiss Supra 55VP field-emission SEM (Germany). Energy-dispersive X-ray spectroscopy (EDX) was performed, and the data were analysed using specialised software to identify foulant elements on the fouled RO membranes.

Elemental concentrations of liquid samples (i.e. extracts from HNO_3 and NaOH treatments) were measured using inductively coupled plasma mass spectrometry (ICP-MS; Agilent 7900, Agilent Technologies, USA) operated with helium collision gas. A multi-N/C UV TOC analyser (Analytik Jena, Germany) was used to determine the total organic carbon (TOC) of the liquid samples.

Other characteristics of the MBRs effluent and the permeate from the RO system were analysed following standardised procedures. Electrical conductivity and pH of these waters were measured using portable conductivity meters (Endress & Hauser Multiparameter handheld Liquiline Mobile CML18).

Table 1
Manufacture and operating specifications of the RO membrane elements.

Specifications	Value
Element diameter (inches)	8
Element length (inches)	40
Active membrane area (m^2)	39
Membrane material	Thin-film composite
Maximum operating pressure (bar)	41
Maximum feed flowrate (m^3/h)	16
Operating pH range	4–10
Salt rejection (%)	99.5
Permeate flowrate (L/day)	9000–12,000

3. Results and discussions

3.1. Performance of the RO membranes before membrane autopsy

The sewer mining plant and the RO membrane system had been operated for 5 years before all RO membrane elements were replaced, and the membrane autopsy was conducted. Characteristics of water streams in the sewer mining plant are provided in Table 2. The performance indicators of the RO system before and after membrane replacement are shown in Fig. 2. Despite being able to provide permeate of quality sufficient for recycle and on-site reuse (i.e., electrical conductivity of ∼36 μS/cm), the differential pressure over the first membrane stage of the RO system reached as high as 21 kPa due to irreversible membrane fouling. This increased differential pressure resulted in increase in specific energy consumption and the cost of fresh water extracted from sewer. Thus, the RO membrane elements were replaced. After membrane replacement, the differential pressure over the first stage of the RO system was reduced to 16 kPa and the permeate conductivity was stabilised around 15 μS/cm.

3.2. Visual observations of the fouled RO membranes

Consistent with the decreased performance before membrane replacement, the visual observations of the membrane confirmed severe fouling occurred on the outer and inside the spiral-wound RO membrane elements (Fig. 3). On the outer side, particularly near the head of the membrane element, slimy layers of organic and bio-foulants were observed (Fig. 3A&B). Inside the membrane elements, abundant foulants remained on the surface of the flat-sheet RO membrane even after rinsing with the RO permeate water for 15 min (Fig. 3C&D). The foulants depositing on the membrane surface added resistance to the transfer of water across the membrane, thus increasing the differential pressure over the membrane stages when the RO system was operated at constant permeate flux. The compositions of these foulant layers are discussed in the next section.

3.3. Fouling characterisation

SEM images demonstrate that the RO membranes were fouled with layers composed of inorganic scales, organic matters, and microorganisms, though they were not evenly distributed over the membrane surface (Fig. 4). The foulant in Fig. 4A appeared as a dense gel layer interspersed with particulate matter, indicating the probable presence of extracellular polymeric substance (EPS) produced by microbial communities (Wen et al., 2024; Chun et al., 2021). Such biofilms typically consist of proteins, polysaccharides, and lipids, which provide a matrix for trapping organic matter and fine colloids from the wastewater secondary effluent (Martínez-Campos et al., 2018; Yu et al., 2017). The presence of bacterial colony in Fig. 4A confirmed the formation of the biofilm on the RO membrane surface. This is consistent with previous reported studies on RO treatment of wastewater secondary effluents (Chun et al., 2021; Zhao et al., 2010). The combination of microbial cells, EPS, and adsorbed dissolved organic compounds created a dense, cohesive fouling structure that was difficult to completely remove during monthly chemical CIP (Martínez-Campos et al., 2018). The foulant remnants on the membrane surface served as a foundation for further scaling or particulate deposition, leading to the gradual performance deterioration of the RO system (Adel et al., 2022).

The resilience of bacteria and the subsequent formation of the biofilms were of particularly concern during the RO process of wastewater secondary effluent in sewer mining. The biofilms were formed on the feed side of the membrane, where the membrane surface was subjected to robust and turbulent flows under high hydraulic pressure. The ability of bacteria to thrive in such conditions underscores the complexity of microbial interactions within the RO system in sewer mining plants (Tang et al., 2014).

Fig. 4B provides further insights into the nature of fouling encountered in the RO process of the wastewater secondary effluent. It illustrates a particulate layer consisting of a complex of organic and inorganic compounds. Notably, structural features in Fig. 4C are characterised by edges that are neither distinctly sharp nor uniformly rounded. This morphology implies a diverse composition of the foulants formed on the RO membrane surface in the treatment of the wastewater secondary effluent. The compositions of foulants on the RO membrane applied in sewer mining are affected by various factors, including the characteristics of the wastewater fed into the

Table 2
Characteristics of water streams in the sewer minging plant ("-" not reported).

Water characteristics	Water stream			
	Raw wastewater	MBR effluent	RO permeate	
			Before replacement	After replacement
Total dissolved solids (mg/L)	330	443	< 10	< 10
pH	7.3	7.1	6.9	6.8
Electrical conductivity (μS/cm)		1578	36	15
Suspended solids (mg/L)	263	< 5	< 5	< 5
Turbidity (NTU)	-	0.23	0.1	-
Total hardness as $CaCO_3$ (mg/L)	60	74	1.1	-
Total alkalinity as $CaCO_3$ (mg/l)	186	4	0.2	-
Chemical oxygen demand (mg/L)	152	20	< 5	-
Biological oxygen demand (mg/L)	59	5	< 5	-
Faecal Coliforms & E.coli	-	-	< 1	< 1

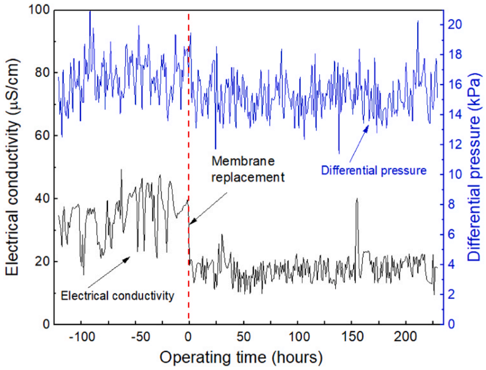

Fig. 2. Permeate electrical conductivity and the differential pressure of the first stage of the RO system before and after membrane replacement and autopsy. Negative operating time indicates time before the membrane replacement.

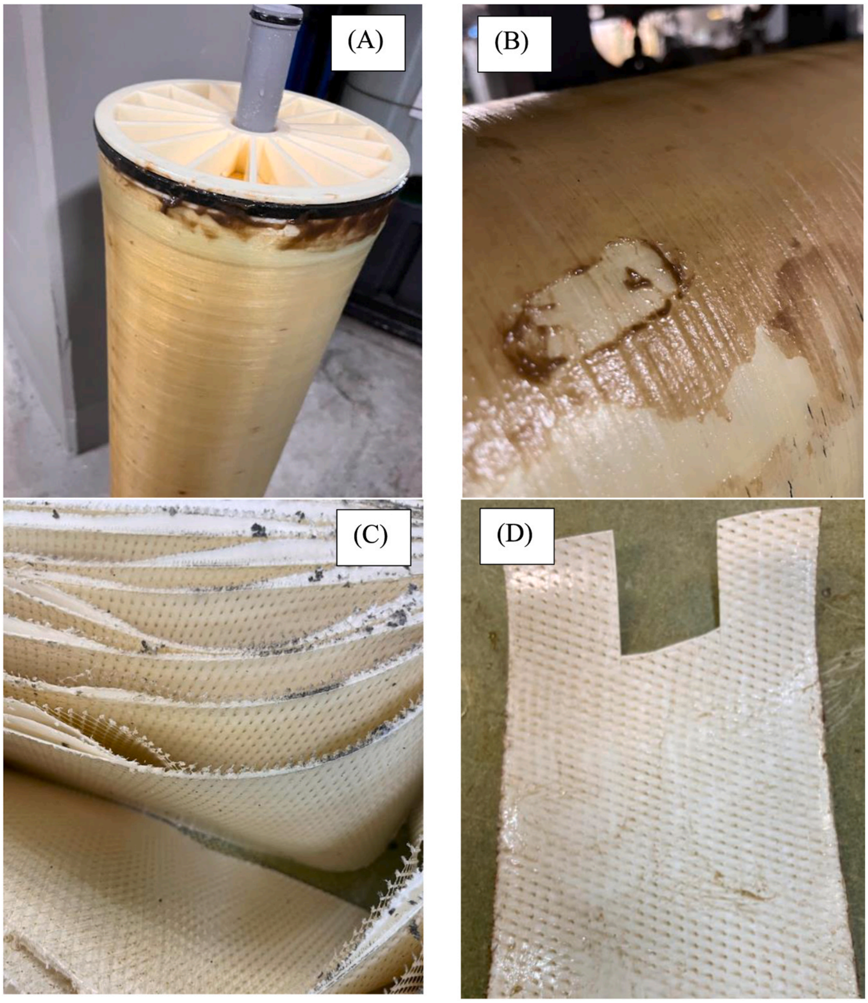

Fig. 3. Images of fouled RO membrane modules: (A&B) foulant on the outer side of the membrane element, and (C&D) membrane remnants on the flat-sheet membrane configuring the RO membrane element.

sewer mining plant and the secondary treatment processes prior to RO membranes. For example, Liu et al (Liu et al., 2020). reported that the foulants on the RO membrane of the integrated anaerobic fixed-film MBR/RO process were predominantly biofilms and organic substances; inorganic foulants were not evidenced. The absence of inorganic salts in the RO foulants in (Liu et al., 2020) might

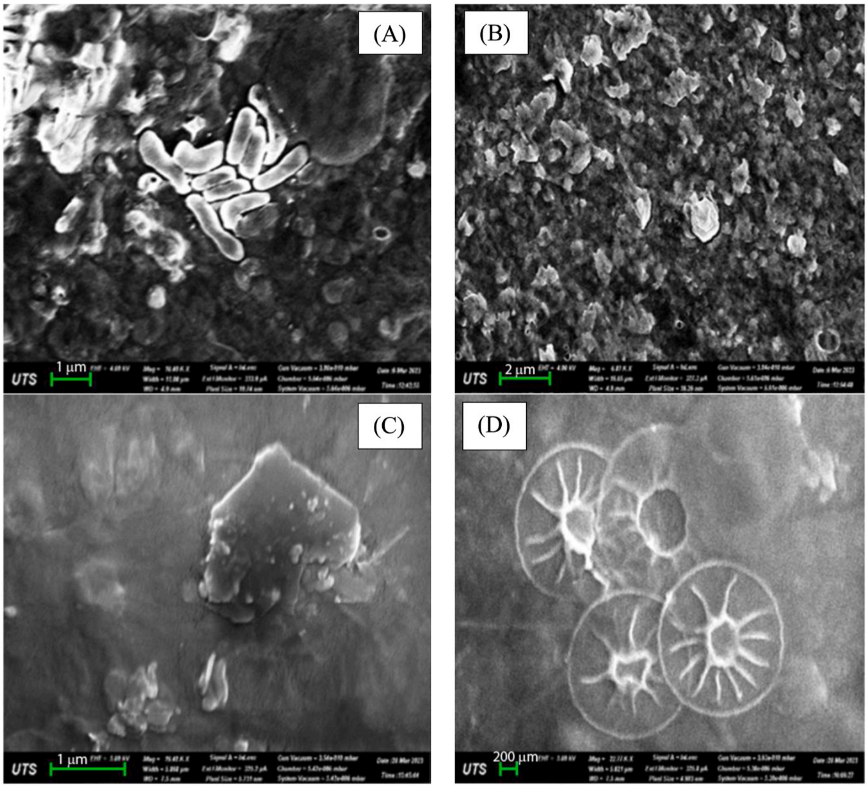

Fig. 4. Selected scanning electron microscopic images of the fouled membrane surface.

be attributed to the small pore sizes of the membranes (0.02 µm) deployed in the MBR unit of the sewer mining plant in that study.

Fig. 4D shows residues of microbes that penetrated the pre-treatment and the MBR process before being retained on the RO membrane. This figure shows the skeletal structure of diatoms adhering to the membrane. These organisms inhabit in both freshwater and marine environments but are rarely found in wastewater systems. The introduction of these organisms into the sewer mining plant and the RO system could occur through the inclusion of runoff water or seawater, especially when the sewer mining plant is situated in proximity to a coastal area. Moreover, the detection of diatoms in the RO foulant layer indicates possible membrane leakage in the MBR system given the larger size of diatoms compared to the pore sizes of the MBR membrane. It is noteworthy that previous membrane autopsy studies have hardly reported the presence of diatoms in the foulant layers of RO membranes in wastewater treatment or desalination applications.

EDX spectra of the foulant layer shown in Fig. 4B corroborate the visual observation and analysis mentioned above. The EDX spectra shown in Fig. 5 affirm the coexistence of organic and inorganic constituents of the foulant layers. The presence of inorganic components, such as aluminium and iron, in the foulant layer underscored the involvement of mineral-rich substances in the fouling

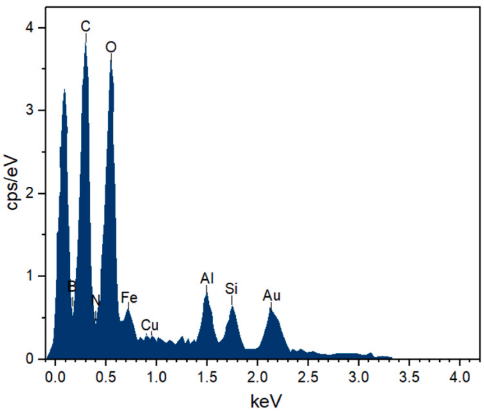

Fig. 5. EDS spectra of the foulant of RO membrane.

process. Notably, the EDX results also indicate a substantial presence of carbon, which is a characteristic signature of organic foulant constituents. It is worth noting that carbon originating from microbial cells is not expected given UF pretreatment prior to the RO system.

The analysis of the acid and base solutions after extracting foulants from the RO membrane samples provides a comprehensive assessment of the foulant composition. The cover density of the foulant constituents provided in Table 3 gives indicators for the extent of fouling on the RO membrane. As explained before, the fouling extraction process involved the utilisation of two distinct and potent solutions (0.5 M HNO_3 and 0.5 M NaOH), followed by sonication to ensure the maximum possible dissolution of foulant components from the membrane into the solution.

The results in Table 3 show significant concentration of TOC in the samples, particularly in the base extracting solutions (0.5 M NaOH). Given its strong alkaline nature, NaOH broke down and converted organic foulants into water-soluble products through hydrolysis, resulting in markedly high TOC concentrations in the base solutions. On the other hand, organic foulants were partly dissociated from the fouled membrane in the acid extraction; thus, TOC concentrations in the acid solutions were lower. Consequently, TOC cover densities on the first and last membrane element obtained from the acid extraction were noticeably lower than those obtained from the base extraction. Silicate was even not detected in the acid extraction, but its significantly high cover densities were on the first and last membrane following the base extraction procedure. Moreover, the higher silicate cover densities found in this study compared to those reported by Tang et al. (2014) highlight the site-specific nature of membrane fouling in RO treatment of wastewater secondary effluent in sewer mining plants.

The concentrations of most foulant components in base extracting solutions of the first membrane element were discernibly higher than those for the last membrane element (Table 3). This spatial variation in foulant components aligns with the expected distribution of flow, where the first membrane element handled a larger flow proportion (40–50 %) compared to the last membrane element (10–20 %). The higher cover densities of calcium and iron on the last membrane element compared to the first one in the acid extraction might be attributed to their higher concentrations in the feed water to the last membrane element. Concentration polarisation effect in the RO process might have exacerbated the deposition of calcium and iron. Given its higher feed concentration but lower turbulence, concentration polarisation effect was expected to be higher in the last membrane element compared to the first one.

The TOC and elemental analysis shown in Table 3 also indicates the need for combined acid/alkaline CIP during the RO treatment of the wastewater secondary effluent in the sewer mining plant. The complex compositions of the foulants on the RO membrane require both acid and alkaline cleaning agents. Indeed, the cleaning agents used in monthly chemical CIP of the RO system in the sewer mining plant were commercial products containing either acid or alkaline ingredients such as acetic acid, peroyacetic acid, citric acid, or potassium carbonate, tetrasodium ethylenediaminetetraacetate, and potassium hydroxide. Despite of the combined acid/alkaline based chemical CIP, foulant remnants on the membrane surface still induced gradual performance deterioration of the RO system. In this context, the process optimisation of the whole sewer mining plant, particularly pre-treatment to eliminate organic matters and silicate before the RO system, might be of vital importance to mitigate accumulative membrane fouling and the premature replacement of the RO membrane elements.

The autopsy results reported here highlight the complexity of RO membrane fouling in sewer mining with distinctive site-specific nature compared to that observed in brackish or seawater water desalination. The RO foulant layers in brackish or seawater desalination might also compose of biofouling, organic fouling, and inorganic scaling; however, their TOC and silicate compositions were negligible compared to those shown in Table 3. Indeed, autopsy studies of RO fouled membranes from brackish and seawater desalination revealed the predominant compositions of aluminium and iron (Gonzalez-Gil et al., 2021; Tran et al., 2007).

4. Conclusions

This study reported a systematic membrane autopsy of reverse osmosis (RO) membrane filtration used for sewer mining. Results show a clear spatial pattern of fouling along the RO membrane train, with the lead-stage elements experiencing more severe organic and colloidal fouling than the tail elements due to higher hydraulic loading and contaminant accumulation. Microscopic and spectroscopic analyses indicated foulant layers comprising of complex organic and inorganic constituents possibly with biofilm formation, confirming occurrence fouling mechanisms. Organic and elemental analyses of foulant extracts varied by membrane element position in the system, highlighting the need for tailored cleaning strategies and pretreatment. These results justified sequential acid and alkaline chemical clean-in-place (CIP) protocols to remove diverse foulant types and emphasised proactive fouling management, particularly for unattended, automated sewer-mining plants through optimisation of pretreatment and CIP regimes to improve RO reliability and cost-effectiveness.

CRediT authorship contribution statement

Amar Salih: Validation, Data curation, Conceptualization. **Claudio Kohn:** Writing – original draft, Validation, Investigation, Data curation. **Long Duc Nghiem:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Quang Viet Ly:** Validation, Methodology. **Hung Cong Duong:** Writing – review & editing, Methodology. **Cuong Ton-That:** Supervision, Methodology, Investigation.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing

Table 3
TOC and elemental density of the foulant layer on the RO fouled membrane surface (na = not detectable or not measured; values are mean and standard deviation of at least two replicate samples).

Foulants	Cover density (mg/m^2)			
	Acid extraction		Base extraction	
	First element	Last element	First element	Last element
TOC	**347.6 ± 26.9**	**371.5 ± 25.4**	**4918 ± 38**	**3026 ± 98**
Si	na	na	**13,762.5 ± 217.7**	**8283.5 ± 3148.8**
Al	**8.87 ± 1.46**	**8.33 ± 1.97**	**253.6 ± 73.9**	**33.5 ± 3.4**
Fe	**9.38 ± 2.04**	**12.96 ± 2.09**	na	na
Mg	**9.1 ± 1.05**	**8.88 ± 0.02**	na	na
Ca	**11.91 ± 0.61**	**12.39 ± 0.92**	na	na
Mn	3.09 ± 2.48	0.97 ± 0.19	na	na
Zn	1.72 ± 0.17	2.02 ± 0.78	na	na
Cu	2.47 ± 0.34	3.46 ± 0.19	na	na
Zn	1.72 ± 0.17	2.02 ± 0.78	na	na
Ba	1.87 ± 0.01	1.86 ± 0.01	na	na
Na	49.01 ± 13.7	20.62 ± 0.92	na	na
K	16.21 ± 5.04	14.34 ± 0.51	na	na
Be	1.87 ± 0.01	1.86 ± 0.01	na	na

interests: Long D. Nghiem serves as a co-editor of the journal but is not involved in the editorial or review process.

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Data availability

Data will be made available on request.

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