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Basics, membrane fouling and fouling control**

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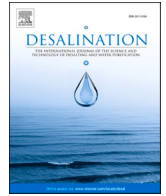
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# Ceramic membrane filtration for oily wastewater treatment: Basics, membrane fouling and fouling control

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## HIGHLIGHTS

- Ceramic membrane technology is promising for oily wastewater treatment.
- The factors affecting ceramic membrane fouling during oily wastewater separation are discussed.
- The commonly used strategies for fouling control of ceramic membranes are reviewed.
- Opportunities for improved fouling control are identified.

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## ABSTRACT

Membrane technology presents an effective solution for treating oily wastewater, a significant environmental hazard stemming from industries such as food processing, metalworking, and oil extraction. Compared to polymeric membranes, ceramic ones exhibit superior mechanical, chemical, and thermal stability, enabling more effective oil removal and easier cleaning. Despite their advantages, membrane fouling remains a challenge, impacting the efficiency of oily wastewater treatment. This review explores oily wastewater characteristics and ceramic membrane applications in treatment processes. It examines the factors influencing ceramic membrane fouling, including wastewater properties (e.g., oil concentration, pH), membrane characteristics (e.g., surface hydrophilicity, charge), and operational parameters (e.g., cross-flow velocity, permeate flux). Strategies to mitigate fouling, such as pretreatment, backpulsing/backwashing for sustained operation, and chemical cleaning for fouling removal, are discussed. By using pretreatment, membrane fouling can be reduced. Backpulsing/backwashing is effective to maintain a long-term operation. Chemical cleaning is effective in removing irreversible fouling and restoring the performance of the ceramic membranes. Moreover, membrane modification techniques that enhance performance are highlighted. Ultimately, the review identifies that effective fouling control is crucial for optimizing ceramic membrane use in oily wastewater treatment, underscoring the need for ongoing research in this area.

## 1. Introduction

Due to the rapid industrial growth, vast amounts of oily wastewater are produced by a wide range of industries, such as oil and gas, petrochemical, food, textile, leather and metal finishing [1,2]. Oily wastewater, especially produced water, generated from oil and gas wells, contains a high level of pollutants. It may cause a series of problems if directly discharged into the aquatic environment [3,4]. Therefore, these oil/water mixtures should be treated to meet the stringent discharge regulations and reduce the environmental impacts [5]. Furthermore,

Water scarcity poses a global challenge due to the explosive growth of the world's population and economy; therefore, the reuse of industrial oily wastewater, for example, as fracking fluid for oil extraction, should also be considered [6].

Oily wastewater can be classified into three types, i.e., free, dispersed and emulsified oils, by considering the droplet size. Free oil has a droplet size  $>150\ \mu\text{m}$  with a lower specific gravity than water and can thus easily be removed by traditional techniques like flotation. Dispersed oil, with oil droplet sizes ranging from  $25\ \mu\text{m}$  to  $150\ \mu\text{m}$ , is more stable than free oil and will only coalesce by an external force induced e.g., by

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centrifugal processes [7]. Oily wastewater with a droplet size of  $<20\ \mu\text{m}$ , is classified as emulsified oil [8,9]. Emulsified oils are usually stabilized by surfactants and are, therefore, resistant to coalescence to form larger oil droplets [8]. This makes them difficult to be removed from wastewater. Various technologies have been used to separate oily wastewater based on the oil droplet size [10]. These include coagulation and flocculation, gravitational settling, dissolved air flotation, hydrocyclone, and adsorption. However, when dealing with stable oil-in-water (O/W) emulsions, the above-mentioned methods have difficulties, i.e., too low separation efficiency to meet reinjection and reuse purposes [11]. Moreover, the traditional methods have high capital and operational costs and require a large operation space [12–15]. The advantages and disadvantages of the commonly used methods for oily wastewater treatment have been compared in Table 1.

Membrane separation is one of the most promising methods for oily wastewater treatment, especially O/W emulsions [16,17]. The separation of oily wastewater by membranes has several advantages, including a high oil removal efficacy, no chemical addition, and a more compact design, compared to traditional technologies [12]. The high mechanical, thermal and chemical stability of ceramic membranes make these membranes particularly suitable for industrial wastewater treatment, such as oily wastewater (see also Table 2) [18–22]. The initial

**Table 1**  
Comparison of oily wastewater treatment methods [13,14].

Methods	Principle	Advantages	Disadvantages
Coagulation/flocculation	Break the emulsion and promote droplet coalescence via chemical additives	Enhanced oil coalescence and aggregation, increase the removal of solids and organic compounds	Not effective for dissolved oil removal, a large volume of sludge is produced
Gravitational settling	Separation of free oil ( $>150\ \mu\text{m}$ ) based on the specific gravity of oil droplets	No energy requirement, cheaper, effective for large oil droplets and suspended particles removal	Not suitable for stable emulsion and dissolved oil removal
Dissolved air flotation	Oil droplets attach to the dissolved air bubbles and float to the surface	Suitable for the removal smaller particles and oil droplets $>20\ \mu\text{m}$	Difficult to remove dissolved oil, not cost effective to produce large amount of air that are smaller than oil droplets
Hydrocyclone	Free oil separation under centrifugal force	Compact modules, higher efficiency for smaller oil droplets	Fouling and clogging, energy requirement, not effective to remove stable oil emulsion
Adsorption	Retain contaminants by porous adsorbent	Compact modules, high quality effluent, cheaper	High retention time, generated chemical waste related to the regeneration of adsorbent
Polymeric membrane	Remove particles, colloids and oil droplets ( $<20\ \mu\text{m}$ ) based on size exclusion	High quality permeate, small footprint, little chemical requirement	Membrane fouling, susceptible to degradation at temperatures higher than $50\ ^\circ\text{C}$ , sensitive to chemical cleaning
Ceramic membrane	Remove particles, colloids and oil droplets ( $<20\ \mu\text{m}$ ) based on size exclusion	High quality permeate, small footprint, little chemical requirement, high chemical, thermal and mechanical stability	Membrane fouling, Higher cost than polymeric membranes, brittleness, defects

manufacturing cost of ceramic membranes is higher compared to their polymeric counterparts. However, the performance of ceramic membranes in water and wastewater treatment, including extended lifespan and reduced maintenance requirements, can offset the overall water production cost [23]. Besides, because of the hydrophilic surface, the high porosity and the narrow pore size distribution, ceramic membranes are considered to have a higher permeate flux and lower membrane fouling [24–26]. Moreover, they are known as materials with good tolerance to high oil content and other foulants in the feed. After fouling, the membranes can be cleaned in-place (CIP) with more aggressive chemicals at a higher temperature [27–29], while, polymeric membranes normally suffer from degradation when the temperature is higher than  $50\ ^\circ\text{C}$  [13]. A schematic review of the advantages of ceramic membranes for water treatment over polymeric counterparts can be found in a recent review [26]. As a result, ceramic membranes have been widely studied in oily wastewater treatment. As shown in Fig. 1, the number of publications about ceramic membranes for oily wastewater treatment, as well as the number of citations of papers per year, increased considerably, from 20 and 733 to 126 and 5493 respectively, between the years 2012 and 2022.

The membranes, however, suffer from fouling problems during the filtration process, in which a layer of oil droplets, suspended particles and other components of wastewater is formed on the membrane surface and/or membrane pores are plugged or blocked by small oil droplets and particles [30,31]. Membrane fouling causes a decrease in permeance, resulting in a lower flux or higher operating pressures [32]. As a consequence, it is important to control membrane fouling effectively during the filtration process [33]. Reducing membrane fouling can lead to a decrease in operating costs and thus to a potential increase in ceramic membrane applications on the oily wastewater treatment market.

In their previous work, Dickhout et al. [34] explored the interaction between the membrane and produced water emulsion from a colloidal perspective. Huang et al. [35] conducted a review of antifouling membranes for oily wastewater treatment, focusing on the interplay between wetting and membrane fouling. More recently, Tanudjaja et al. [16] provided an overview of various aspects related to the practical applications of membrane filtration for oily wastewater treatment. However, to date, no review papers have specifically addressed fouling of ceramic membrane filtration in the treatment of oily wastewater. In order to achieve a comprehensive understanding of ceramic membrane fouling by oily wastewater, in this review, we first introduce the composition of oily wastewater and then the ceramic membranes (materials and type) for oily wastewater filtration. Afterwards, the factors affecting ceramic membrane fouling during oily wastewater separation are discussed from the aspects of feed characteristics, membrane properties, and operational parameters. Finally, the commonly used strategies (pretreatment, backpulsing/backwashing, chemical cleaning and membrane modification) for fouling control of ceramic membrane filtered by oily wastewater are reviewed and opportunities for improved fouling control are identified.

## 2. Ceramic membrane filtration for oily wastewater treatment

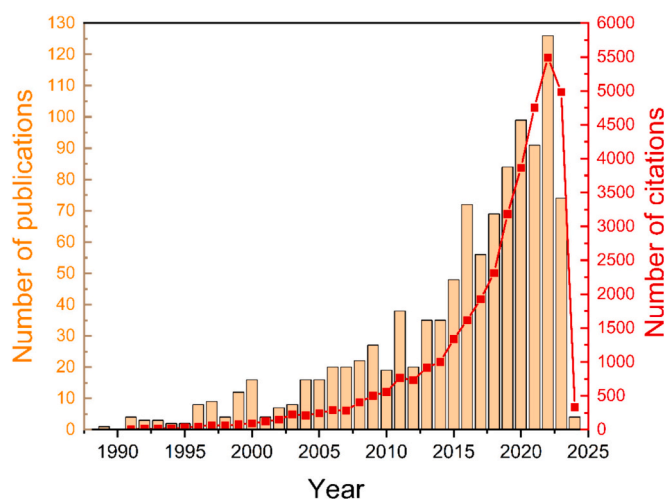
### 2.1. Composition of oily wastewater

Oily wastewater mainly contains oil and grease and contaminants such as suspended particles, dissolved organic substances, chemical additives (e.g., surfactants), and inorganic substances (e.g., heavy metals, and salts) [36]. The various components of oily wastewater can affect the separation process. For instance, surfactants can influence the electrostatic interactions between the membrane surface and oil droplets, thereby affecting membrane fouling [37]. The concentration of surfactants can impact both fouling and rejection of the membrane. A high concentration of surfactants also reduces the interfacial tension of the emulsion, making oil droplets more deformable, increasing their

**Table 2**  
Comparison of commercial ceramic membrane filtration in the treatment of oily wastewater.

Membrane			Feed condition					Performance			Refs
Material	Pore size ( $\mu\text{m}$ )	Configuration	Oil type	$d_{\text{oil}}$ ( $\mu\text{m}$ )	$C_f$ ( $\text{mg L}^{-1}$ )	CFV ( $\text{m s}^{-1}$ )	TMP (bar)	Initial flux (LMH/bar)	Final flux (LMH/bar)	$R_d$ (%)	
$\alpha\text{-Al}_2\text{O}_3$	0.2	Tubular	Crude oil	1–10	250	0.24	0.69	760	61	98.5	[91]
$\alpha\text{-Al}_2\text{O}_3$	0.8	Tubular	Crude oil	1–10	250	0.24	0.69	982	48	99.9	[91]
$\alpha\text{-Al}_2\text{O}_3$	0.1–0.2	Tubular	Rolling emulsion	10–20	10,000	3–5	1–2	54	~	99.8	[52]
$\text{ZrO}_2$	0.1–0.2	Tubular	Rolling emulsion	10–20	10,000	3–5	1–2	125	62.5	99.9	[52]
$\alpha\text{-Al}_2\text{O}_3$	0.05	Tubular	Synthetic oily wastewater	~	500	1.68	2	~	85	98	[94]
$\text{ZrO}_2$	0.2	~	Refinery oil	~	6000	2.56	1.1	218	109	99.4	[95]
$\alpha\text{-Al}_2\text{O}_3$	0.2	Tubular	Refinery oil	~	26	2.25	1.25	350	260	84.6	[45]
$\text{ZrO}_2$	0.1	Tubular	Produced water	1–100	100	2	2	908	87.5	98	[19]
$\text{ZrO}_2$	0.1	Tubular	Produced water	~	$25 \pm 2$	3	1.5	700	200	88	[20]
$\text{TiO}_2$	50 kDa	Triangular	Produced water	~	20–57	2	0.4	200–250	100	71–88	[21]
$\text{TiO}_2$	2	Triangular	Produced water	~	30–84	2	0.4	350–600	300	80–93	[21]
$\text{TiO}_2$	0.05	Tubular	Produced water	~	565	0.6–1.3	1	70	5	99	[22]
SiC	0.04	Tubular	Olive mill wastewater	~	275	2	67 <sup>b</sup>	2083	347	97	[96]
SiC	0.04	Tubular	Produced water	~	20–57	2	0.4	1500–1900	500	61–85	[21]
SiC	0.5	Tubular	Produced water	~	38–57	2	0.4	7200–8400	2500	73–86	[21]
NF <sup>a</sup> -TiO <sub>2</sub>	1000 Da	Tubular	Produced water	~	565	~	1	20	20	99.53	[93]

$C_f$  - oil concentration in the feed, CFV - cross-flow velocity,  $d_{\text{oil}}$  - oil droplet size,  $R_d$  - oil rejection, ~ not mentioned, a - UF pretreatment, b - constant flux, LMH.



**Fig. 1.** Numbers of publications and citations on ceramic membranes for oily wastewater treatment from 1989 to 2024. The data was extracted from Web of Science in March 2024, with the keywords of “ceramic membrane AND (oily wastewater OR oil-in-water emulsion OR produced water)”.

likelihood of penetrating the membrane [38]. Additionally, the presence of salts in oily wastewater can weaken the charge effect on both the membrane and oil droplets, further complicating the separation process [39]. Moreover, the composition of oily wastewater varies considerably in various industries. A high content of organic carbon is commonly observed in the food and drinking industry, while the oil content in metal processing industry is very low [16]. To this end, Meneceur et al. [40] developed green nanocomposite adsorbent to eliminate the heavy metals, oil and grease, total suspended solids, and organic pollutants from petrochemical wastewater. Shokri et al. [41] reviewed electrocoagulation for oil removal from various industries, noting its enhanced efficiency when combined with other processes such as membrane filtration and adsorption.

Produced water during oil and gas extraction is the largest stream of oily wastewater in the world with a global estimated 3:1 volume-to-product ratio, adding up to an estimated annual global volume of 54 billion cubic meters in 2020 [42,43]. To prevent operational problems and improve oil recovery, surfactants and other chemical additives are

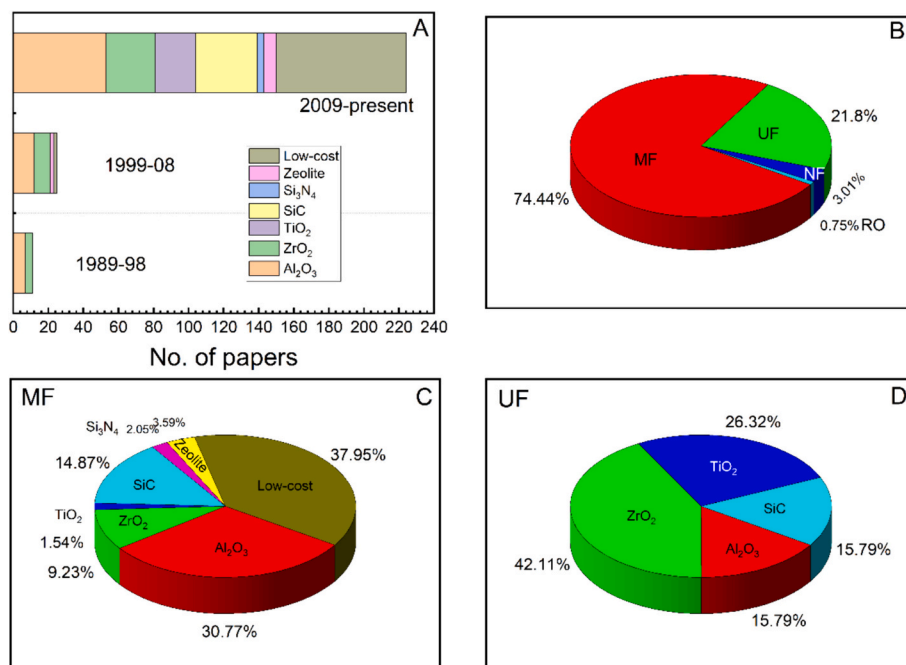
commonly used in the oil and gas industry [34,44]. Produced water also contains other constituents from the subsurface [45]. The physical and chemical properties of the produced water vary considerably, depending on the geographic location, operational conditions and age of the oil and gas fields. For example, salinity and hardness level, mainly composed of  $\text{Cl}^-$ ,  $\text{Na}^+$ ,  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ , vary from 0 to 300,000  $\text{mg L}^{-1}$  [44]. The total organic carbon (TOC) concentration is normally in a range of 500–1000  $\text{mg L}^{-1}$  [43]. Typically, the total dissolved solids (TDS), and major ions (e.g.,  $\text{Cl}^-$ ,  $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{Sr}^{2+}$ ) increase with well lifetime, while alkalinity, pH and TOC decrease with time [46]. In addition, a high water to oil production ratio is often found in many aging oil wells [47].

## 2.2. Materials used to produce ceramic membranes

The ceramic membranes widely used in oily wastewater treatment are alumina ( $\text{Al}_2\text{O}_3$ ), zirconia ( $\text{ZrO}_2$ ), titania ( $\text{TiO}_2$ ), silicon carbide (SiC) and low-cost ceramic membranes (e.g., mullite, and kaolin) (Fig. 2A).  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  membranes were first used for oily wastewater treatment due to their commercial availability on the market [48,49] (Fig. 2A). Membrane filtration with an  $\text{Al}_2\text{O}_3$  membrane was considered as the best available technology among the treatments of oilfield brines by the US EPA in 1992 [50]. Compared with  $\text{Al}_2\text{O}_3$  membrane,  $\text{ZrO}_2$  membrane has a higher and more stable flux in addition to be less susceptible to fouling [51–53]. The higher hydrophilic and negatively charged surface of  $\text{ZrO}_2$  membrane leads to the weak attachment of oil droplets. Therefore, higher fluxes and lower fouling have been observed [52,54].

Afterwards, zeolite ceramic membranes were synthesized by in situ crystallization on the inner surface of tubular  $\alpha$ -alumina substrates for oily wastewater treatment [55,56]. It was found that the zeolite layer can improve the fouling resistance of the membrane due to the enhanced surface hydrophilicity. In addition, the nano-channel pathways along the zeolite pores favour the transport of water molecules [57,58].

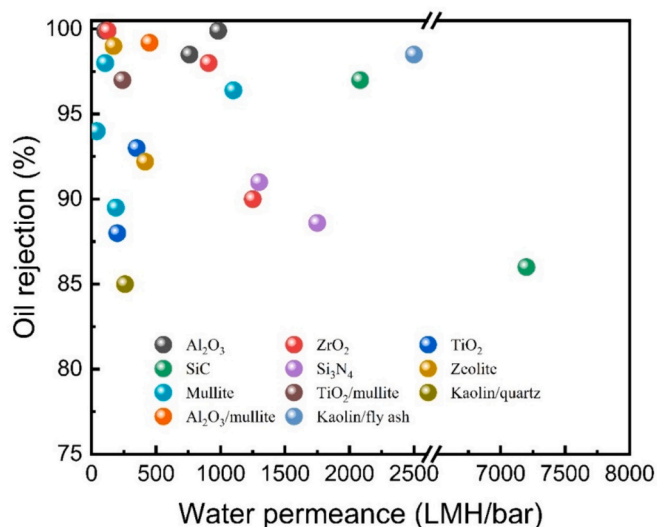
In the present decade (since 2009),  $\text{TiO}_2$  and SiC membranes have been developed for the separation of oily wastewater (Fig. 2A). Zhang et al. [59] first studied the  $\text{TiO}_2$ -doped  $\text{Al}_2\text{O}_3$  composite membrane with an average pore size of 0.2  $\mu\text{m}$  for oily wastewater filtration, a higher and more stable permeate flux was observed for the  $\text{Al}_2\text{O}_3$ - $\text{TiO}_2$  composite membrane than that of  $\text{Al}_2\text{O}_3$  membrane. Ebrahimi et al. [22] used  $\text{TiO}_2$  membrane for oil-field produced water filtration with an oil rejection of up to 99 %.



**Fig. 2.** (A) Number of papers of different ceramic membranes in three divided decades (updated in March 2024), (B) percentage of each type of ceramic membranes, (C) percentage of different ceramic MF membrane materials, and (D) percentage of different ceramic UF membrane materials for oily wastewater or produced water treatment. Note that some papers report on several ceramic materials or types in each, so the number of ceramic membrane materials and types rather than the number of papers is presented here.

Compared with the Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, and TiO<sub>2</sub> membranes, SiC membranes have been considered to have a higher mechanical strength, chemical resistance and a lower thermal expansion coefficient [60,61]. Furthermore, SiC membranes have a lower irreversible fouling and higher permeate flux than polymeric membranes and traditional ceramic membranes [39,62–65]. As a result, SiC membranes have been demonstrated for pilot and full-scale produced water treatment [66,67]. More recently, silicon nitride (Si<sub>3</sub>N<sub>4</sub>) ceramic membranes were developed and studied for O/W separation. The membrane with a pore size of 0.68 μm was found to have a high oil rejection (95 %) and permeate flux (390 L m<sup>-2</sup> h<sup>-1</sup> (LMH)) at a transmembrane pressure (TMP) of 1 bar in an alkaline environment [68,69].

One of the major limitations for industrial application of the ceramic membranes is their higher costs than their polymeric counterparts, because of the utilization of expensive inorganic precursors (Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, TiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub> and SiC), smaller surface area per module and high sintering temperatures, associated with high energy consumption [70]. For example, commercial SiC ceramic membranes are usually sintered at a temperature up to 2000 °C in an argon atmosphere [71]. Therefore, considerable effort has been invested in reducing the sintering temperature of SiC membranes by using low-temperature sintering aids [72,73]. Other strategies, such as co-sintering, to reduce the sintering costs of conventional ceramic membranes have also been studied [74]. In addition, clay-based or clay-bonded and solid-state waste-based low-cost ceramic membranes have been developed for the separation of O/W emulsion in the last decade (Fig. 2A) [75–81]. These low-cost ceramic membranes are usually prepared with e.g., kaolin, quartz, mullite, feldspar, bauxite and coal fly ash, at a temperature below 1500 °C [82–85]. As shown in Fig. 3, the performance of the low-cost ceramic membranes is comparable with traditional commercial ceramic membranes in terms of permeate flux and oil rejection [5,86–88]. However, the chemical stability of these membranes may be lower due to the existence of impurity and higher porosity in the membrane matrix, thus leading to a shorter lifetime [89]. Therefore, they are mainly studied with synthetic O/W emulsions at a lab-scale; no pilot or full-scale applications have been demonstrated yet.



**Fig. 3.** Relationship of oil rejection and initial water permeance of traditional ceramic membranes and low-cost ceramic membranes. Here we chose 22 samples from literature (Table S1, supporting information) to have a general performance comparison of traditional ceramic membranes with low-cost ceramic membranes.

### 2.3. Type of ceramic membranes used for oily wastewater filtration

The type of ceramic membranes used for oily wastewater treatment include microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and zeolite reverse osmosis (RO) (Fig. 2B). Ceramic MF and UF membranes are the most (>96 %) studied ones for oily wastewater treatment among the four types of membranes (Fig. 2B). In MF, Al<sub>2</sub>O<sub>3</sub>, SiC and low-cost ceramic membranes are the top three membranes studied in literature (Fig. 2C), while in UF, also ZrO<sub>2</sub>, and TiO<sub>2</sub> membranes are used, where ZrO<sub>2</sub> membrane accounts for the highest percentage (Fig. 2D).

TiO<sub>2</sub> and zeolite are respectively used as ceramic NF and RO membranes for produced water treatment [55,90].

Table 2 lists the configuration of the commercial ceramic membranes used for oily wastewater treatment as reported in literature. Considering the high fouling potential and variable composition of oily wastewater, tubular ceramic MF and UF are the most used because they have a higher fouling resistance and are easy to clean [52,91]. For model O/W emulsions and refineries, these membranes have shown a high oil rejection (> 98 %) [21]. Although a lower flux has been found at a higher oil concentration in the feed, the rejection of the membrane has not been compromised. For real oily wastewater, like produced water, MF or UF treatment alone is not sufficient [42,46]. For example, despite having a much higher permeate flux than other ceramic membranes, the oil rejection of SiC MF membrane is only 73–86 % for produced water treatment [21], and the TiO<sub>2</sub> UF only reaches a maximum rejection of 88 % [21]. Unlike the model O/W emulsions, oil droplet size distribution could be much wider in real oily wastewater. The presence of small oil droplets enables them to pass through the membrane easily. Additionally, the salinity of real oily wastewater, such as produced water, is very high, compromising the effectiveness of the membrane's surface charge in oil rejection.

Given that low rejection is commonly observed by ceramic MF/UF for real oily wastewater, especially produced water, NF and RO should then be considered as a post-treatment step. Because the temperature of produced water could be as high as 85 °C, ceramic membranes are more suitable to use than polymeric membranes, since they would encounter degradation issues at these high temperatures [92]. The adoption of ceramic NF and zeolite RO membranes for oily wastewater treatment started later compared to ceramic MF and UF membranes, with their earliest known application being in 2009 [22]. Ebrahimi et al. [22] reported the use of commercial NF membranes, preceded by either MF or UF for the treatment of model (synthetic) and real produced water. The ceramic membrane systems showed up to 99 % oil removal, but severe flux reduction of NF membranes was observed during produced water filtration. In another study, a total oil removal of up to 99.5 % was also obtained using ceramic UF followed by ceramic NF as a final treatment of oilfield produced water [93]. Besides the high oil rejection, ceramic NF also exhibited high TOC (75–90 %) rejection and 100 % total suspended solids (TSS) rejection. Furthermore, most divalent cations and anions (e.g., Ba<sup>2+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup> and SO<sub>4</sub><sup>2-</sup>) can be efficiently removed from produced water [90]. Zeolite RO ceramic membranes are normally synthesized at lab-scale. Because of the uniform sub-nanometer- or nanometer-scale pores of zeolites, these membranes could be used to reject dissolved organic pollutants and salts from produced water [55].

### 3. Ceramic membrane fouling in oily wastewater treatment

Membrane fouling, causing a decreased permeate flux and increased operating costs, is one of the primary challenges influencing the long-term operation of membranes [12,34,97]. As shown in Fig. 4, the type of fouling depends on feed composition (foulant component, pH, ionic

strength, and temperature), membrane structural and physicochemical properties (pore size, hydrophilicity/hydrophobicity, surface charge and roughness), and operational conditions (TMP, permeate flux, and cross-flow velocity). The factors that affect the ceramic membrane's fouling in oily wastewater treatment are further discussed in this section.

#### 3.1. Effect of oily wastewater characteristics on membrane fouling

Membrane fouling is strongly affected by the characteristics of oily wastewater such as oil concentration, pH, ionic strength, surfactant type and concentration, and temperature [37,39]. With an increase in oil concentration in the feed, larger permeate flux decline and higher oil rejection are normally observed as a thicker oil layer is formed over the membrane surface that is difficult to be removed by hydraulic cleaning [5,94,98,99]. In the cross-flow microfiltration of oily wastewater using an  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> membrane, Hua et al. [94] observed that the steady state permeate flux of the membrane decreased from 165 LMH to 135 LMH with an increase in oil concentration from 250 to 1000 mg L<sup>-1</sup>. Kumar et al. [5] also found a larger flux decline in the microfiltration of synthetic oily wastewater when oil concentration increased from 50 to 200 mg L<sup>-1</sup>. At the highest oil concentration (200 mg L<sup>-1</sup>), oil rejection reached 99.64 %. The type of oil can affect the droplet behaviour near a membrane surface. Oils with a higher viscosity are not easily removed by cross-flow and are thus more likely to attach on the membrane surface [100].

The effect of pH on oil emulsion depends on the surfactants used for emulsion preparation. For instance, the properties (zeta potential and droplet size) of the O/W emulsion, stabilized by dioctyl sulfosuccinate sodium salt and Span 80, respectively, have not been affected by pH, based on studies by Lobo et al. [101] and Zhang et al. [59]. The effect of solution pH on membrane fouling is mainly caused by the electrostatic interactions between the charged membrane surface and the charged oil droplets [59]. A higher stable permeate flux and higher oil rejection has been found at higher pH due to the stronger electrostatic repulsion between the oil droplets and membrane surface [68]. In addition, Hua et al. [94] observed a higher negative charge of oil droplets at a higher pH. In that case, the higher permeate flux of the Al<sub>2</sub>O<sub>3</sub> membrane was the result of a more "open" cake layer at high pH, due to the inter-droplet interaction.

Ceramic membrane filtration is frequently used for high temperature oil emulsion separation due to its high thermal stability. At higher temperatures the permeate flux of ceramic membranes increase, because of the decrease of the viscosity of water [45,88,98,102]. However, membrane fouling can also be higher as oil droplets can deform to some degree and block the membrane pores due to lower viscosities of the oil at a higher temperature [98,102].

Salts, commonly present in produced water, are known to affect both the characteristics of oil droplets and the interaction at the interface of oil and membrane and, thereby, membrane fouling [103]. A higher concentration of salts in the feed leads to more fouling and lower

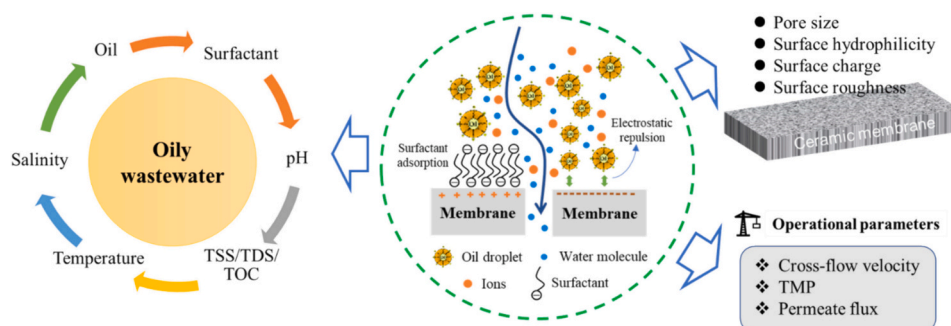


Fig. 4. Schematic diagram of the three main factors on ceramic membrane fouling during oily wastewater treatment.

rejection, due to the effect of compressed electrical double layer and reduced oil-water interfacial tension [38,94,104]. For instance, Abbasi et al. [105], e.g., found that the permeate flux of ceramic membrane decreases with an increase in the salt concentration from 25 to 200 g L<sup>-1</sup>. However, Weschenfelder et al. [106] found that the presence of salts can improve permeate flux when the membrane and oil emulsion have opposite charges.

Oil emulsions are normally stabilized by surfactants and, therefore, the type and concentration of the present surfactant are important in determining the interaction between oil droplets and membrane. A higher permeate flux has been observed when ZrO<sub>2</sub> membranes were used to separate an anionic (glycolic acid ethoxylate oleyl ether) surfactant-stabilized emulsion, while a lower permeate flux was observed for a cationic (hexadecyl trimethyl ammonium bromide) surfactant-stabilized emulsion. This difference in flux can be attributed to the effects of electrostatic repulsion and electrostatic attraction between the membrane surface and droplets [107]. However, an opposite conclusion was drawn by Lu et al. [108] who found that irreversible membrane fouling could be alleviated during ultrafiltration of O/W emulsions with a TiO<sub>2</sub> ceramic membrane oppositely charged to the stabilization surfactant, due to synergetic steric hindrance and demulsification. It was speculated that the electrostatic attraction of positively charged surfactants on the negatively charged membrane surface and pores form a surfactant barrier which prevents oil droplets' penetration [108]. In addition, due to less positively charged surfactants in the solution, small oil droplets become unstable and tend to coalesce into larger droplets, which are more likely to be rejected by the membrane. Besides the surfactant type, its concentration in oil emulsions can also affect membrane fouling either positively or negatively, depending on the added amount. When the surfactant concentration exceeds a specific threshold—the critical micelle concentration (CMC)—micelles begin to form and the additional surfactants in the solution will not adsorb onto the surface of oil droplets [109]. In a recent study, Chen et al. [39] found that an increase in surfactant concentration (below the CMC) may then enhance the interaction between oil and membrane, and thereby improve membrane performance (Fig. 5). With an increase in anionic

surfactant concentration, the irreversible fouling of both positively charged and negatively charged ceramic membranes was reduced. The primary reason for fouling alleviation on the positively charged membrane surface was attributed to the adsorption of surfactant. On the other hand, the reduction in fouling on the negatively charged membrane was attributed to the enhanced electrostatic repulsion between the membrane and oil droplets. Irrespective of the type of surfactants, a decline of permeate flux has been observed for SiC UF when the concentration of surfactant increases (above the CMC) [110]. Matos et al. [107] found that the permeate flux of ZrO<sub>2</sub>/TiO<sub>2</sub> UF (50 kDa) membrane decreased with the concentration of cationic surfactant stabilized emulsions. For emulsions stabilized by nonionic surfactant, the membrane only showed a decrease of permeate flux when the concentration of surfactant was above the CMC. The permeate flux of the membrane was enhanced for emulsions stabilized by anionic surfactants with a concentration below the CMC. Another effect of increasing surfactant concentration is the lower oil-water interfacial tension, which means that the oil droplets are more deformable and thus easier squeeze through the membrane pores. For example, a transition from high to low oil rejection was observed for three types (anionic, cationic and nonionic) of surfactant-stabilized emulsions filtered by SiC MF membrane with increased surfactant concentration [38].

Unlike synthetic O/W emulsions, the real oily wastewater (e.g., produced water) contain many other components such as suspended particles, dissolved organic compounds and solids, which may accelerate membrane fouling. For example, Tomczak and Gryta [27] observed a larger decrease of permeate flux when applying ultrafiltration to real oily wastewater than that of synthetic O/W emulsion made from the same oil. The interaction of oil emulsions with suspended particles could thus be a major reason for causing ceramic membrane fouling. Abdalla et al. [111] also studied the effect of suspended inorganic particles on the microfiltration of oil emulsion. Compared with the emulsion alone, the addition of suspended solids (bentonite) in the oil emulsion resulted in a permeance decrease of 3.5–5 times. In contrast, Tomczak and Gryta [27] found that pre-filtration adversely affect membrane fouling during ultrafiltration of oily wastewater. Without

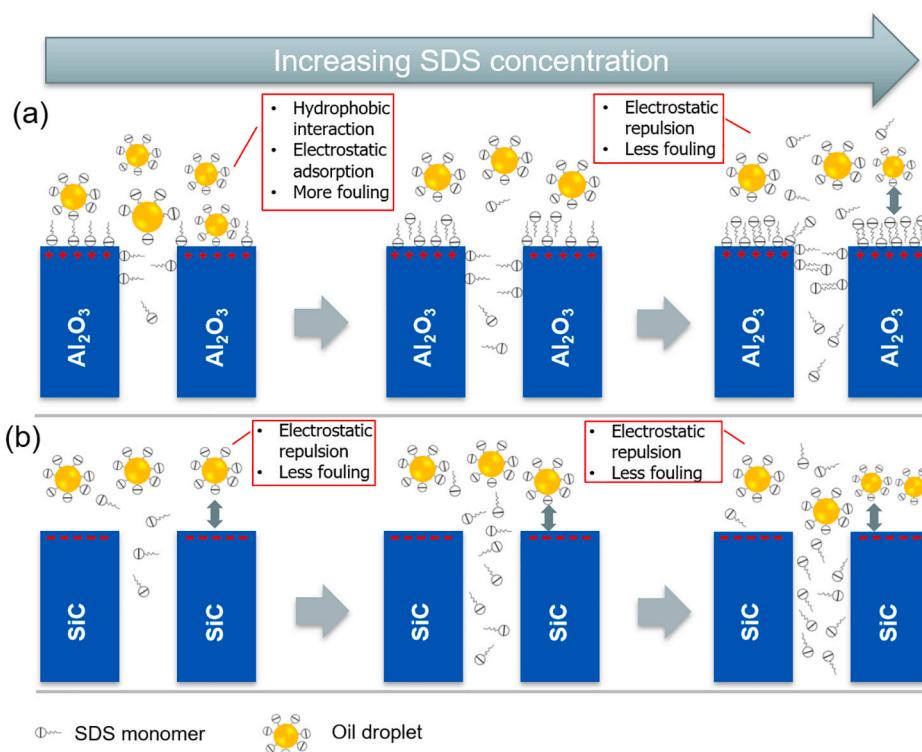


Fig. 5. Effect of SDS concentration on ceramic membrane fouling during filtration of oil emulsions. Adapted with permission from [39].

pre-filtration, the suspended particles could form a cake layer on the membrane surface, preventing the occurrence of internal fouling and leading to a higher stable permeate flux. In addition, the particulate matter present in produced wastewater is more likely aggregated due to the high salinity of the solution. Membrane fouling becomes more serious when the aggregates break into smaller particles at elevated shear force, leading to intermediate pore blocking [112]. Polymeric additives used to enhance oil recovery via increasing the viscosity of water injected into the reservoirs can also have a negative effect on membrane fouling. Weschenfelder et al. [113] found that the flux decline of  $ZrO_2$  membranes reached 84 % in 40 min operation for feed prepared with  $0.1 \text{ g L}^{-1}$  polymer and  $100 \text{ g L}^{-1}$  NaCl. The flux reduction was still high (83 % decline) even for a feed prepared with only  $1 \text{ g L}^{-1}$  polymer. The saturated dissolved solids could form crystalline particles (e.g.,  $CaCO_3$  and  $BaSO_4$ ) in the bulk of oily wastewater and then deposit on the membrane surface, even though it can be alleviated by adjusting the pH of the feed [114]. The gradual buildup of the precipitates on the membrane surface would result in a decline in permeate flux [115]. Characterization of flux and permeate quality during oily wastewater treatment is generally used to monitor the performance degradation of the membrane due to fouling. However, it cannot provide further information on membrane fouling due to the effect of interactions of different components in produced water and their interactions with the membranes. Therefore, detailed characterizations of the autopsied membranes would provide valuable insights into the fouling processes in ceramic membranes. Thibault et al. [116] proposed a possible fouling formation sequence in ceramic ultrafiltration of produced water, based on the so-called micro-characterizations such as backscattered electron (BSE) imaging and wavelength-dispersive (WDS) X-ray maps (Fig. 6). Firstly, the local surface linear defects, arising from the sol-gel synthesis of the selective layer, are responsible for fouling initialization. These defect areas can act as nucleation sites, favouring the precipitation of saturated species (e.g.,  $Mg(OH)_2$ ,  $SiO_2$ , and  $BaSO_4$ ). Following this initial inorganic precipitation, oil compounds may then preferentially adsorb on the precipitates or co-precipitation of dissolved organic matter and silica may occur. Eventually, the filtration performance decreased, and the operating life of the ceramic membrane was shortened. In addition, reaction with anatase in the selective layer was observed during the crystallization of  $BaSO_4$ , suggesting that other types of ceramic membranes should be considered when dealing with produced water with high levels of dissolved Ba.

In order to gain a better insight on the fouling mechanism of oil on the membrane surface, many other non-invasive techniques have been developed to visualize membrane fouling process. One such technique, known as direct observation through membrane (DOTM), involves using a microscope equipped with a video camera. Tummons et al. [117] reported using DOTM to reveal the fouling behaviour of sodium dodecyl

sulfate (SDS) stabilized O/W emulsions on the surface of an anodic alumina Anopore membrane ( $0.2 \mu\text{m}$ ). The study found that membrane fouling by oil droplets occurred in three distinct stages: (1) droplets attachment and clustering, (2) droplet deformation, (3) droplet coalescence. In addition, Electrical impedance spectroscopy (EIS), ultrasonic time-domain reflectometry (UTDR) and optical coherence tomography (OCT) have been shown to provide valuable information on fouling process of droplets on membrane surfaces [16,100]. However, these techniques are still limited in their applications either due to the requirement of a special membrane module or the low image resolution of the equipment.

### 3.2. Effect of membrane properties on membrane fouling

#### 3.2.1. Membrane pore size

The rejection mechanism of MF and UF membranes is mainly based on the size-sieving effect. A smaller pore size gives a better rejection as well as less irreversible fouling [118]. Oil droplets larger than the membrane pore size may accumulate on the membrane surface blocking the pores and eventually forming a cake layer or continuous layer (Fig. 7 (a), (b) and (d)). Therefore, complete pore blocking occurs easier on a membrane with smaller pore sizes [37,119]. However, since oil droplets are deformable, and they can squeeze through the membrane when TMP is higher than the critical pressure ( $P_{crit}$ ), leading to a lower oil rejection but reduced fouling (more discussion in Section 3.3.3) (Fig. 7 (e) and (f)). Conversely, when the pore size of the membrane is larger than the size of oil droplets, internal pore blocking can occur, as depicted in Fig. 7 (c). As a consequence, considering the size of oil droplets relative to the membrane pore size is crucial when selecting a membrane for oily wastewater treatment to mitigate fouling [37]. Ebrahimi et al. [93] observed a lower decline of the flux of the membranes with a pore size of  $0.1 \mu\text{m}$  and 20 kDa compared to a membrane with a pore size of  $0.2 \mu\text{m}$ . Whereas, Jiang et al. [120] found that an MF ceramic membrane with a larger pore size can mitigate fouling for the treatment of flowback water from a shale gas well.

#### 3.2.2. Membrane surface hydrophilicity

Hydrophilicity is one of the most important surface properties of the filter medium [121,122], since the affinity between foulants and the membrane surface under water is important for membrane fouling. A hydrophilic surface could effectively prevent the deposition or adsorption of the hydrophobic oil droplets from the emulsions [34] and thus improve the membrane permeate flux [32,102,123–125]. Ceramic membranes are normally prepared with metal oxides and all of them are hydrophilic because of the surface hydroxyls, making them, in principle, suitable for oily wastewater treatment [32]. In addition, the surface hydrophilicity of the ceramic membranes was found to be related to the open porosity of the membranes. A higher hydrophilic surface was observed for membranes with a higher open porosity, leading to lower fouling due to smaller adhesion force of oil droplets [65]. However, an explanation for this phenomenon was not found. Among the ceramic membranes, SiC membranes, which feature a thin  $SiO_2$  layer on their surface, exhibit superhydrophilic properties with a static water contact angle below  $5^\circ$ , resulting in a high water permeance and a low fouling potential for O/W separation [126,127].

#### 3.2.3. Membrane surface charge

Membrane surface charge becomes increasingly important when charged foulants are present in the feed [128]. Electrostatic interaction will dominate the membrane fouling at low salinity in the feed water. However, with a high concentration of salts presented, the electrical double layer thickness of the membrane is compressed, leading to a lower zeta potential [59,112]. In addition, the membrane surface charge is strongly dependent on iso-electric point (IEP) of the membrane materials and the pH of the solution. Oil droplets in produced water are usually negatively charged, also due to the addition of anionic

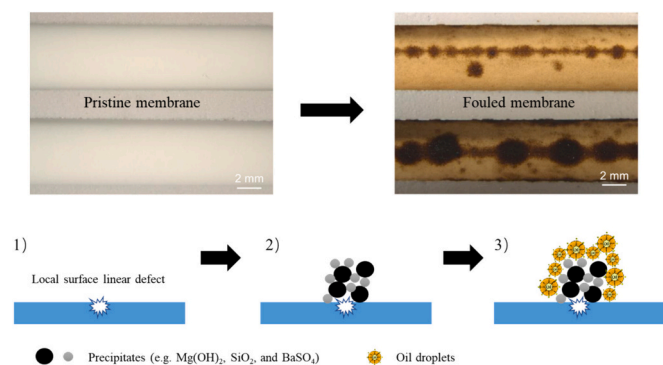


Fig. 6. Possible sequence of fouling formation on the ceramic membrane surface for ultrafiltration of produced water. The surface linear defects are the possible reason leading to the initialization and propagation of fouling on the membrane surface. Adapted with permission from [116].



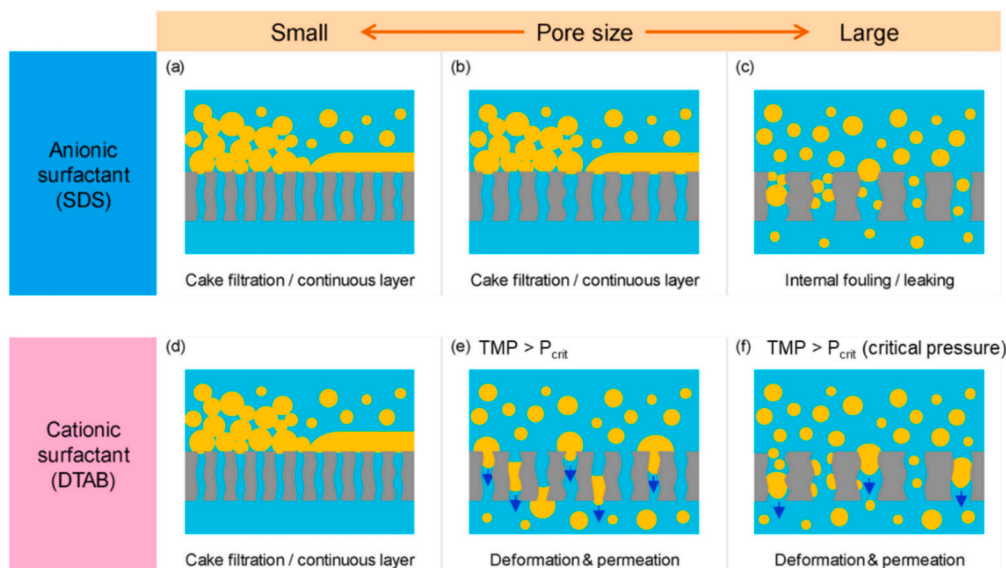


Fig. 7. Effect of pore size of porous ceramic membranes on fouling during filtration of surfactant-stabilized emulsions. Adapted with permission from [37].

surfactants to improve oil recovery. Therefore, the selection of a membrane with the same surface charge to oil droplets can mitigate membrane fouling. This becomes more important when the membrane pore sizes are comparable to that of the oil droplets [37]. SiC membranes have the lowest IEP (pH 2.6) among ceramic membranes, making them thus more suitable for oily wastewater treatment with a low salt concentration or ionic strength [62,129]. Zsirai et al. [21] compared performance of SiC and TiO<sub>2</sub> membranes in a pilot-scale study of produced water filtration and the results suggested that the SiC MF membranes had the highest fouling propensity among the ceramic membranes. This was explained by the following two reasons: firstly, the high salinity of the produced water screened the electrostatic interaction between SiC membrane and oil droplets. Secondly, all membranes were filtered at the same pressure, a higher fouling propensity could happen in SiC MF as the initial flux was >10 times higher than that of other ceramic membranes.

### 3.2.4. Membrane surface roughness

The effect of surface roughness on membrane fouling is under debate. In some studies, increased surface roughness was found to increase membrane fouling due to the enhanced interaction between oil droplets and the rougher membrane surface [130,131]. However, a decreased fouling tendency was observed for (super)hydrophilic membranes with a rough surface (hierarchical structure) than the one with a smooth surface. The proposed mechanism is that water can be trapped in the micro-/nanoscale structure and in this way, reducing the contact area of the membrane to which oil-droplets can attach [34,35].

## 3.3. Effect of operational parameters on membrane fouling

### 3.3.1. Cross-flow velocity

Membrane separation can be operated in either a dead-end or cross-flow mode. The use of cross-flow filtration with turbulent flow can effectively reduce membrane fouling by sweeping away the deposited pollutants on the membrane surface, permitting longer filtration cycles. Turbulent flow is easier to be obtained in membrane feed channels with a wider diameter at lower crossflow velocities compared to capillary membranes. At a very high crossflow velocity, turbulent flow can also be achieved in capillary membranes (e.g., 0.7 mm inner diameter) while leading to high energy consumption (Table 3). Also, a higher permeate flux can be observed at higher cross-flow velocities due to stronger turbulence formed on the membrane surface. For oily wastewater

Table 3

Correlation of feed channel diameter and crossflow velocity to achieve turbulent flow in membrane filtration.

Feed channel diameter	Crossflow velocity	Flow type	Energy consumption
Small	High	Turbulent	High
Big	Small	Turbulent	Low

separation, the operation with ceramic membranes in the cross-flow mode is increasing [21]. However, with increasing cross-flow velocity, the shape of oil droplets is strongly deformed and the flocs are prone to breakup on the membrane surface or in the cross-flow pump due to the high shear forces. In these cases, the oil droplet and particles can pass or block the membrane pores, leading to more severe fouling and/or low rejection [112,132].

### 3.3.2. Permeate flux

So far, few papers have paid attention to the effect of permeate flux on ceramic membrane fouling for oily wastewater treatment. Fraga et al. [96] assessed a SiC membrane for the treatment of olive mill wastewater at constant flux filtration. A flux of 67 LMH was decided for the test as the lowest TMP increase was observed. Loganathan et al. [115] found that the optimal permeate flux range is 125–130 LMH for ultrafiltration of oily wastewater in a pilot study. In this flux range, stable and sustained operations were achieved. However, several authors have studied the effect of permeate flux on polymeric membrane-based oily wastewater treatment [133–139]. According to these studies, the threshold flux, defined as the flux at which a high fouling rate is observed, can be determined by the well-known flux stepping protocol [139]. In most studies, the results indicate that TMP increases gradually and then approaches a steady state if the flux is below the threshold flux [136,138,140]. However, a three-stage fouling is developed when the flux is above threshold flux (Fig. 8). In stage 1, a gradual increase of TMP has been observed followed by a sharp TMP jump (stage 2). Finally, the TMP enters a pseudo-steady state region. Below the threshold flux, modest increase in fouling resistance was observed for both constant flux and constant pressure filtration conditions, while above the threshold flux, fouling was more severe in the constant flux system [139]. Fux and Ramon [141] have found that during operation at low flux, the oil droplets are spherical, being easily removed by cross-flow cleaning, while a high flux would lead to droplets' deformation and irreversible deposition on the membrane surface or in the membrane

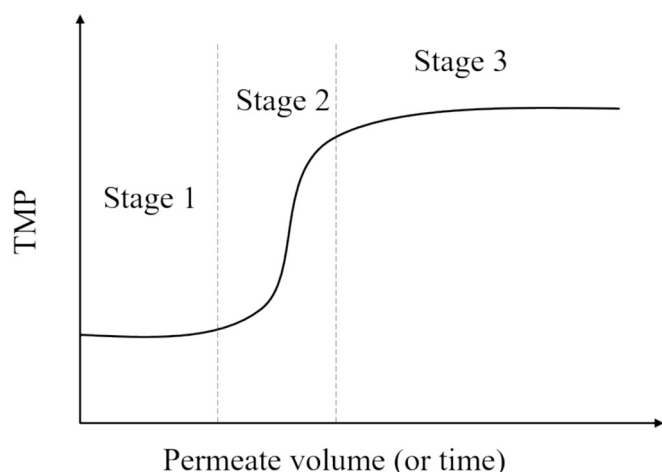


Fig. 8. Fouling develops in three stages in constant flux cross-flow filtration with flux above the threshold flux. Adapted with permission from [133].

pores.

### 3.3.3. Operational pressure

During the filtration of O/W emulsions by a membrane, the rejection of solutes could be different from the rigid particulate as the shape of oil droplets varies, depending on the applied pressure. Therefore, oil droplets can coalesce or break into smaller ones, entering or penetrating the membrane pores that are narrower than the average size of oil droplets [133,142]. A model was put forward by Nazzal and Wiesner [143] to describe the TMP required for a droplet to deform and permeate the membrane (critical pressure). The model was later corrected by Cumming et al., [144] and validated by Darvishzadeh and Priezjev [132]. The critical pressure,  $P_{crit}$  can be described as:

$$P_{crit} = \frac{2\sigma\cos\theta}{r_p} \sqrt[3]{1 - \frac{2 + 3\cos\theta - \cos^3\theta}{4(r_d/r_p)^3 \cos^3\theta - (2 - 3\sin\theta + \sin^3\theta)}} \quad (1)$$

where  $\sigma$  is the interfacial tension of the oil phase and aqueous phase,  $r_d$  is the oil droplet size,  $r_p$  is the diameter of membrane pores,  $\theta$  is the contact angle of oil and membrane.

Table 4 summarizes the critical pressures for ceramic membranes in oily wastewater treatment. As can be seen, the critical pressures are usually located in the range of 1–3 bars for MF and UF ceramic membranes. Higher critical pressures are observed for larger oil droplets filtered by membranes with the same or similar pore sizes. In addition, membranes with smaller pore sizes have been found to have a higher critical pressure for rejecting oil droplets with comparable sizes. When TMP is higher than the critical pressure, a lower oil rejection but a higher permeate flux can be observed [37]. Furthermore, when the

Table 4

Critical pressures for O/W wastewater treatment using ceramic MF/UF membranes in different studies.

Pore size ( $\mu\text{m}$ )	Membrane	Critical pressure (bar)	Method	Mean droplet size ( $\mu\text{m}$ )	Refs
0.05	$\alpha\text{-Al}_2\text{O}_3$	2	Experiments	0.45	[94]
0.2	ZrO <sub>2</sub>	1.55	Experiments	–	[95]
0.2	$\alpha\text{-Al}_2\text{O}_3$	1.25	Experiments	3	[45]
0.2	TiO <sub>2</sub> - Al <sub>2</sub> O <sub>3</sub>	2.4	Experiments	6	[102]
0.68	Si <sub>3</sub> N <sub>4</sub>	1	Experiments	0.68	[68]
0.15	–	1	Analytic	0.9	[132]
0.5	$\alpha\text{-Al}_2\text{O}_3$ / ZrO <sub>2</sub>	2.8	Analytic	11	[123]

– not reported.

nominal pore size of membranes was similar, the membrane with a larger pore size distribution was found to have a lower oil rejection as oil droplets can pass through some larger pores of the membrane [145].

## 4. Fouling control of ceramic membrane for oily wastewater filtration

Membrane fouling is gradually formed during the oily wastewater filtration process. A cake layer (reversible fouling) can be formed on the membrane surface when the oil droplet size is larger than the pore size of membrane and can be removed by regular backpulsing/backwashing. Internal clogging may occur when the particles are smaller than the membrane pore size which further reduces the pore volume, thus resulting in constriction of pore flow (irreversible fouling) [146,147]. Chemical cleaning is widely used to recover the membrane performance after accumulation of irreversible fouling. In Fig. 9, we can see that the commonly used methods for fouling control of ceramic membranes in oily wastewater treatment include: pretreatment, backpulsing/backwashing, and chemical (acid and/or base) cleaning [24]. In addition, membrane modification can be an effective way to alleviate membrane fouling.

### 4.1. Pretreatment

Pretreatment to enhance oily wastewater treatment efficacy by ceramic membranes include coagulation/flocculation [95,148], electrocoagulation [149] and adsorption [150]. Aluminum and iron salts are commonly used for coagulation and the concentration of coagulant is important to increase membrane flux and oil rejection [148,151,152]. Flocculants can also be used to decrease membrane fouling and improve permeate quality. Zhong et al. [95] compared five types of flocculants (inorganic and polymeric) for the pretreatment of refinery wastewater. The most effective flocculant was 3530S (a derivative of polyacrylamide). Changmai et al. [149] utilized electrocoagulation-microfiltration for the treatment of oily wastewater. The concentration of oil and grease could considerably be reduced in 20 min and then ceramic membranes were used to remove the flocs. Due to the increase in the size of oil droplets after coagulation/flocculation as well as the formation of large flocs on the membrane surface, a highly porous cake layer and low attractive energy between the cake layer and the membrane surface was formed and the extent of pore plugging was decreased [95]. However, when the flocculant is overdosed, sludge flocs could be formed and could block the membrane pores and thus increase membrane mass transfer resistance [148].

Oily wastewater treatment can also be enhanced by a hybrid adsorption-membrane filtration process. A combination of powdered activated carbon (PAC) with membrane filtration is generally used [150,153,154]. Abbasi et al. [153] studied the effect of the concentration of PAC on the performance of ceramic membranes. Permeate flux of the membranes increased at a low concentration of PAC loading (200–400 ppm), while a decrease of permeate flux of the membranes was observed at high PAC concentrations due to the deposition of PAC particles on the membrane surface. Yang et al. [154] also used PAC (100 ppm) as an additive in the microfiltration-PAC system for emulsion filtration. Membrane fouling was effectively mitigated due to the mechanical scouring effect of PAC particles. Other adsorbents such as zeolite can also be used in the pretreatment step to reduce membrane fouling [150].

### 4.2. Backpulsing/backwashing

In Table 5, an overview is given of the performance of ceramic MF/UF membranes with and without backpulsing/backwashing for oily wastewater treatment. The fundamental difference between backpulsing and backwashing is the utilized frequency and duration of the reversed force (Fig. 10). Backpulsing is typically done at a very short period (<1

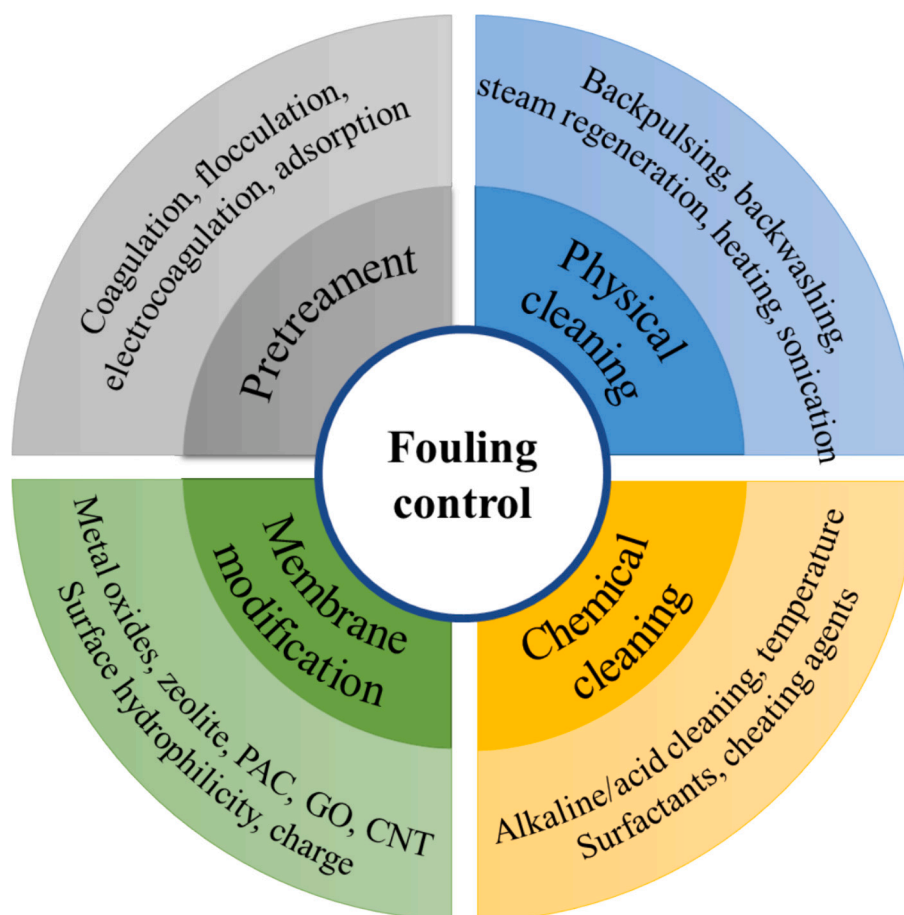


Fig. 9. Fouling control of ceramic membranes for oily wastewater treatment. The strategies include pretreatment, physical cleaning, chemical cleaning and membrane modification.

**Table 5**  
Backpulsing/backwashing performance of ceramic membranes for oily wastewater treatment.

Feed	Membrane	Pore size ( $\mu\text{m}$ )	Backpulsing/backwashing conditions	Results	Refs
Dilute oil-in-water suspension	$\alpha\text{-Al}_2\text{O}_3$	0.2/0.8	$P = 1.4$ bar, $t_b = 0.2, 0.5$ s, $f = 0.01\text{--}0.1$ Hz	The permeate flux was increased up to 25 times with backpulsing.	[159]
Unstable secondary emulsion	$\text{ZrO}_2$	0.1	$P = 3$ bar, $t_b = 0.7$ s, $t_i = 1$ min	Steady state flux was increased from 450 to 1000 LMH after backpulsing	[123]
Oil/water emulsion	$\text{ZrO}_2$	0.1	$P = 4$ bar, $t_b = 0.2, 0.5, 1$ s	The permeate flux with backpulsing was 3-fold higher than that without backpulsing	[160]
Olive mill wastewater	SiC	0.04	$P = 3$ bar, $t_b = 0.8$ s, $t_i = 10$ min	By backpulsing, the final TMP was reduced from 0.53 to 0.48 bar after 24 h filtration at a constant flux of 67 LMH	[96]
Real and synthetic produced water	$\alpha\text{-Al}_2\text{O}_3$	0.1/0.2/0.5	$P = 2$ bar, $t_b = 1$ min, $t_i = 30$ min/60 min	A 22% and 30% improvement of permeate effective flux could be achieved when backwashing was applied every 60 and 30 min respectively	[19]
Refinery wastewater	$\alpha\text{-Al}_2\text{O}_3$	0.2	$P = 2$ bar, $t_b = 15$ s, $t_i = 280$ s	The periodic backwashing of ceramic membrane can maintain its performance in a long-term operation	[45]
Oilfield produced water	$\text{ZrO}_2$	0.1	$P = 2$ bar, $t_b = 30$ s, $t_i = 40$ min	The flux increased 35% immediately after the first backwashing	[20]

P - backpulsing/backwashing pressure,  $t_b$  - backpulsing/backwashing duration,  $t_i$  - backpulsing/backwashing interval,  $f$  - backpulsing/backwashing frequency ( $f = 1/t_b$ ).

s) and high frequency, while backwashing takes a few seconds or even minutes every 15 min or longer. In addition, there is no interruption to the process of membrane filtration when using backpulsing [155]. During the long-term filtration, backpulsing/backwashing has a positive effect on MF/UF membrane filtration performance, which is normally conducted at 1–4 bar [21,93]. It can remove the surface deposits and unblock the pores. In addition, backpulsing/backwashing would allow a higher average flux, requiring a lower membrane surface area and, as a consequence, resulting in more compact systems with reduced footprints

[20]. The frequency and duration of backwashing can be adjusted to achieve an improved recovery [156]. Weschenfelder et al. [19] found that a higher effective permeate flux was obtained at a higher backwashing frequency. In addition, a combination of backpulsing and backwashing led to the highest effective permeate flux. Zsirai et al. [157] also observed that a higher flux can be sustained via the application of backpulsing during the cleaning-in-place process. Furthermore, backwashing can be enhanced (so called enhanced chemical backwashing (ECB)) with chemicals such as NaOH and acid to extend

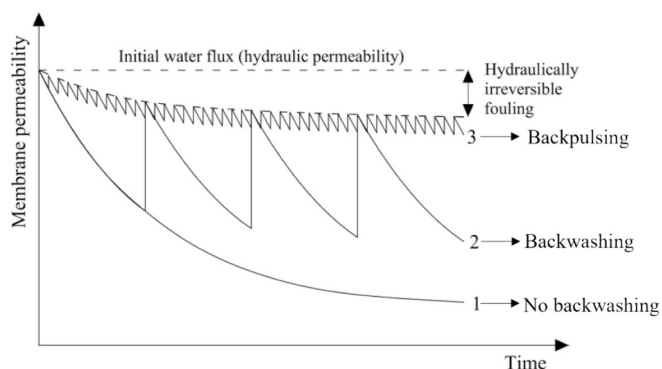


Fig. 10. Schematic illustration of the difference between backpulsing and backwashing and their effect on the decline of membrane permeability. Adapted with permission from [155].

the timespan between chemical cleaning (so called CIP) being required for produced water treatment [115]. More recently, Hashimoto et al. [158] studied the effect of fine bubbles on membrane fouling treating produced water. It was found that flushing the membrane surface with fine bubbles was more efficient than traditional backwashing in both dead-end and cross-flow filtration modes.

### 4.3. Chemical cleaning

Although the decline of permeate flux can be prevented by using a periodic backpulsing/backwashing process, it is necessary to use chemical cleaning when the loss of permeate flux reaches 50–60 % [45]. In Table 6, the chemical cleaning efficiency of different methods have been compared for oily wastewater treatment by ceramic membranes. As can be observed, the cleaning efficiency is affected by many factors, e. g., membrane properties, oily wastewater characteristics, cleaning chemicals, cleaning temperature, pressure, and duration. Usually, ceramic membranes can be easily cleaned after fouling by a synthetic oil emulsion. Zhu et al. [86] used 0.1 % NaOH aqueous solution to back-flush the soybean oil fouled TiO<sub>2</sub>-mullite ceramic membrane, and found that over 96 % of the original flux can be recovered after the first generation cycle. Abadikhah et al. [161] tested SiO<sub>2</sub> nanoparticles modified Si<sub>3</sub>N<sub>4</sub> ceramic hollow fiber membrane with gasoline prepared emulsion, the membrane recovery was up to 92 % by soaking the fouled membrane in HCl (0.5 M) and NaOH (0.5 M) aqueous solutions for 15 min, respectively. However, the membrane cleaning efficiency is much lower when challenged with the combined emulsion/suspension than with emulsion alone. It was found that the residual permeance of ZrO<sub>2</sub> membrane is 16-fold lower after fouling by a combined emulsion/bentonite suspension based on six short runs with a standard chemical cleaning between each run [111]. Due to the complex composition of real oily wastewater, such as produced water, the flux recovery of ceramic membrane is, in general, lower than for model O/W emulsion

Table 6  
Cleaning of ceramic membranes by various methods for oily wastewater treatment.

Oily water source	Membrane	Pore size (μm)	Scale	Cleaning methods	Operation condition (tem, time, TMP)	Permeance recovery (%)	Refs
Crude oil	α-Al <sub>2</sub> O <sub>3</sub>	0.1	b	1%v- Derquim (pH 9.8) + 1%v-Ultrasil 73 (pH 2.65)	80 °C, 0.85 ± 0.1 bar	98	[162]
Crude oil	α-Al <sub>2</sub> O <sub>3</sub>	0.1	b	1%v-Ultrasil 73 (pH 2.65) + 1%v- Derquim (pH 9.8)	80 °C, 0.85 ± 0.1 bar	99	[162]
Crude oil	α-Al <sub>2</sub> O <sub>3</sub>	0.1	b	1%v-Surfactron CD50 (pH 2.25) + 1%v-Derquim (pH 9.8)	45 °C, 0.85 ± 0.1 bar	91	[162]
Crude oil	α-Al <sub>2</sub> O <sub>3</sub>	0.2	b	1%v-Ultrasil 73 (pH 2.65) + 1%v- Derquim (pH 9.8)	80 °C, 0.85 ± 0.1 bar	90	[162]
Crude oil	α-Al <sub>2</sub> O <sub>3</sub>	0.5	b	1%v-Ultrasil 73 (pH 2.65) + 1%v- Derquim (pH 9.8)	80 °C, 0.85 ± 0.1 bar	60	[162]
Soybean oil	mullite	1.02	b	0.1 % NaOH	Room temperature	65	[86]
Soybean oil	TiO <sub>2</sub> -mullite	0.11	b	0.1 % NaOH	Room temperature	96	[86]
Rolling emulsion	ZrO <sub>2</sub>	0.1–0.2	p	1 % NaOH + 1 % citric acid	70 °C	78	[52]
Produced water	TiO <sub>2</sub>	1000 Da	b	lye solutions (1 % (w/w) NaOH solution, Ultrasil P3-14, Ultrasil P3-10)	30 to 60 min	41	[93]
Gasoline emulsion	Si <sub>3</sub> N <sub>4</sub>	0.68	b	Heating at 400 °C	1 h	99	[68]
Produced water	ZrO <sub>2</sub>	0.1	b	Alkaline solution (1000 mg L <sup>-1</sup> NaOH + 100 mg L <sup>-1</sup> NaClO) + acid solution (8 mg L <sup>-1</sup> citric acid + 8 mg L <sup>-1</sup> glycolic acid)	70 °C, recirculated 15 min	95	[19]
Produced water	ZrO <sub>2</sub>	0.1	p	Alkaline solution (1000 mg L <sup>-1</sup> NaOH + 100 mg L <sup>-1</sup> NaClO) + acid solution (8 mg L <sup>-1</sup> citric acid + 8 mg L <sup>-1</sup> glycolic acid)	70 °C, recirculated 15 min	83	[19]
Produced water	SiC	0.04	p	6 % NaOH + 6 % citric acid	30 min, 0.55–0.6 bar	50	[157]
Olive mill wastewater	SiC	0.04	p	4 % NaOH + 2 % citric acid	25 °C	23	[96]
Olive mill wastewater	SiC	0.04	p	4 % NaOH + 2 % citric acid	60 °C	100	[96]
Produced water	ZrO <sub>2</sub>	0.1	b	Hot water rinsing	50 °C, 10 min, 0.5 bar	40	[20]
Produced water	ZrO <sub>2</sub>	0.1	b	Hot water rinsing + KOH (10 mL/L)/alkylbenzene sulphonate (7 mL/L)	50 °C, 10 min + 40 °C, 30 min, 0.5 bar	58	[20]
Produced water	ZrO <sub>2</sub>	0.1	b	Hot water rinsing + KOH (10 mL/L)/alkylbenzene sulphonate (7 mL/L) + Citric acid (8 mL/L)/glycolic acid (8 mL/L)	50 °C, 10 min + 40 °C, 30 min + 60 °C, 30 min, 0.5 bar	86	[20]
Real oil emulsion	α-Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub>	0.05	b	Water flushing + alkaline solution, ASG (PRU 06-03, Gütling, Germany) + HCl (pH = 3)	40 °C (alkaline cleaning), 1 h, 1 bar	99.9	[114]
Oily wastewater	~	8 kDa	p	Water flushing + 3 % NaOH + 3 % H <sub>3</sub> PO <sub>4</sub>	50 °C, 1 h + 50 °C, 1 h	100	[27]
Oil sand wastewater	TiO <sub>2</sub>	0.1	p	Citric acid (pH = 2.5) + NaOH (pH = 13)	~	100	[115]

b-bench scale, p-pilot scale, ~-not mentioned, tem-temperature.

(Table 6).

The membrane properties such as pore size can affect the cleaning efficiency too. Silalahi and Leiknes [162] compared the flux recovery of Al<sub>2</sub>O<sub>3</sub> ceramic membrane with a nominal pore sizes of 0.1, 0.2 and 0.5 μm, respectively, for crude oil emulsion filtration. Membranes with a smaller pore size had a higher flux recovery in almost all cleaning combinations as oil droplets are mainly present on the surface of the membrane. Similar results have also been reported by other authors [93,118]. When challenged with produced water, membranes with a pore size of 0.1 μm had a flux recovery of 61 %, whereas the cleaning efficiency was only 33 % for the membranes with a pore size of 0.2 μm [93]. Ghidossi et al. [118] found that chemical washing could not regenerate the ZrO<sub>2</sub>-TiO<sub>2</sub> membrane with a pore size of 0.1 μm for oily wastewater treatment due to internal fouling. A 300-kDa membrane was observed to have a high permeance recovery after chemical cleaning. From these studies, a membrane with smaller pore sizes seemed to be more suitable for oily wastewater treatment to obtain a higher flux recovery with chemical cleaning.

Alkaline and acid solutions are widely used for ceramic membrane cleaning due to its chemical stability [26,163]. Alkaline solutions can remove the organic compounds from the membrane surface, while inorganic compounds (e.g. scaling materials) can be effectively removed by acid solutions [164]. To improve the cleaning efficiency, a combination of the two solutions is commonly utilized. For produced water applications, the most effective cleaning cycle is alkaline solution cleaning followed by an acid rinse [49]. This is because a thin continuous oil layer can easily be formed on the outer surface of the membranes, which is quite impermeable to cleaning agents. Therefore, using alkaline cleaning to remove the top oil layer can help to remove the inorganic scaling from the membrane surface or within the pores. Weschenfelder et al. [20] studied the cleaning efficiency of ZrO<sub>2</sub> membranes after filtration with produced water. The flux recovery of the membrane was only 40 % after hot water rinsing, but could be increased up to 58 % after alkaline cleaning and to 86 % of the original flux after subsequent acid cleaning. By optimizing the chemical cleaning protocol, Fraga et al. [96] found that the flux of SiC membrane can be recovered to 100 % after hot water rinsing, acid/alkaline and alkaline/acid cleaning after filtering olive mill wastewaters. Simonič [114] also reported that the best strategy to fully restore the ceramic UF membrane performance was, firstly, using alkaline solution ASG (PRU 06-03, Gütling, Germany) at 40 °C for 1 h at 1 bar. Then the membrane was flushed with distilled water. Afterwards, the membrane was cleaned with HCl (pH = 3) for 30 min followed by final flushing with water.

In addition, the cleaning efficiency of the ceramic membrane can be further improved with chemicals at a higher temperature. Silalahi and Leiknes [162] found that after filtering with crude oil emulsion, the permeance recovery of Al<sub>2</sub>O<sub>3</sub> membranes can reach 91 % when cleaned at 45 °C with alkaline and acid solutions. A higher permeance recovery (99 %) was observed when cleaned at 80 °C. Fraga et al. [96] also compared the chemical cleaning efficiency at room temperature and 60 °C. The membrane could only be recovered to 23 % permeance when cleaned at room temperature, while the permeance was totally restored after cleaning at 60 °C. Complete recovery of membrane performance after fouling by real oily wastewater was also achieved by 1–3 wt% NaOH and H<sub>3</sub>PO<sub>4</sub> solutions at 50 °C [27]. Nonetheless, the integrity of ceramic membranes may be compromised during chemical cleaning at higher temperatures. Observations have shown that the durability of ceramic NF membranes is adversely affected by long-term cleaning with sodium hypochlorite at room temperature [165]. Thus, it can be inferred that chemical cleaning, especially with aggressive acids at elevated temperatures, could further impact the quality of ceramic membranes.

Other chemicals such as surfactants (e.g., SDS) and chelating agents (e.g., ethylene diamine tetra acetic acid (EDTA)) have also been used for ceramic membrane cleaning. Surfactants can detach the oil from the membrane surface due to reduced oil-water surface tension. Chelating agents are expected to form complexes with oil droplets and separate

them from the membrane surface [166]. Garmsiri et al. [166] compared the cleaning efficiency of SDS with three other chemicals (H<sub>2</sub>SO<sub>4</sub>, NaOH and EDTA), and, with concentration of 10 mM, it was found to be the best cleaning agents to recover ceramic membrane performance after filtration of a simulated O/W emulsion.

Since ceramic membranes are resistant to oxidation, oxidants, such as sodium hypochlorite (NaClO), ozone (O<sub>3</sub>) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), are widely used to remove organic and biological foulants on the membrane surface and/or in the pores through oxidation and/or disinfection [164,167]. However, these oxidants are not commonly used to clean the ceramic membranes after fouling by oil droplets because they are found to be inefficient to degrade the oil droplets [168].

#### 4.4. Other cleaning methods

To minimize the chemical waste after chemical cleaning, other cleaning methods have been explored. As an alternative to chemical cleaning, Atallah et al. [169] applied a steam regeneration technique to recover the performance of ceramic membranes in the filtration of produced water at 80–85 °C. It was found that the permeate flux recovery of the membrane was only 50 % with backflushing only, but it could be increased even up to 200 % with the injection of steam directly into the feed channels in conjunction with backflushing. This high flux recovery was attributed to a sudden change in the viscosity of the constituents in the cake layer. The viscosity of the filter cake was reduced for the duration of the steam exposure time, leading to a more efficient release from the membrane surface. After steam regeneration, the membrane still maintained a high temperature, leading to the reduction of feed viscosity and flux recovery of over 100 %.

Heat treatment can also be used for the recovery of ceramic membrane performance via heating it in an oven. Abadikhah et al. [68] restored 99 % of the flux of emulsion fouled Si<sub>3</sub>N<sub>4</sub> membranes after heating at 400 °C for 1 h, while Chen et al. [170] recovered the membrane performance up to 100 % after oil fouling at 600 °C for 2 h.

To enhance the ZrO<sub>2</sub> ceramic membrane separation process, an ultrasonic field was applied by Shu et al. [171] in the treatment of O/W emulsion wastewater. The flux recovery ratio of the membrane was around 40 % with water cleaning under sonication, but it could be increased to 70 % combining with the use of chemical agents. Other fouling control strategies, such as applying high-frequency pulsation to the feed pressure, have been investigated by Mohamed et al. [172,173]. They found that the permeate flux of the UF membrane can be improved by employing a pulsative cleaning method.

As observed in Table 6, the cleaning efficiency of ceramic membranes is related to the scale of the filtration experiment too. A higher permeance recovery has been found at bench scale than at pilot scale. This efficiency difference is caused by flow paths and uniform distribution of cleaning solution, which is more efficient on a membrane surface with smaller module sizes [19].

#### 4.5. Membrane modification

In the filtration of O/W emulsions, the interaction between oil droplets and membrane surface is vital for membrane fouling. Reducing oil droplets adhesion on the membrane surface and/or in the pores can be achieved by precoating a thin layer of nano-materials [174–177] or by changing surface properties such as charge, roughness and hydrophilicity [3,34,178–181]. Thus, surface modification of ceramic membranes can make the membranes less susceptible to fouling [24]. For more details about this strategy, readers are encouraged to refer to our previous review paper [182].

Improving surface hydrophilicity and enabling a higher surface negative charge are two of the mostly used strategies for ceramic membrane modification [182]. To improve surface hydrophilicity, nano-sized metal oxides such as TiO<sub>2</sub>, ZrO<sub>2</sub>, γ-Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> have been used to enhance permeate flux and oil rejection in addition to

the fouling alleviation of ceramic membranes [102,122,183–192]. Besides, zeolites, PAC and graphene oxide have also been used to improve the separation efficiency of ceramic membranes during oil separation [58,193,194]. Since oil droplets are stabilized with charged surfactants, electrostatic interaction between membrane surface and oil droplets is important for membrane fouling too. To reduce membrane fouling, Zhang et al. [59] doped TiO<sub>2</sub> in Al<sub>2</sub>O<sub>3</sub> powder to prepare a composite microfiltration ceramic membrane. The doping not only improved membrane surface hydrophilicity, but also shifted the isoelectric point of the membrane towards lower pH, increasing the negative charge. In addition, Chen et al. [63] found that the fouling resistance of Al<sub>2</sub>O<sub>3</sub> membrane can be improved after deposition with a thin layer of SiC for O/W emulsion separation. The deposited SiC layer increased the negative charge of the membrane and, in this way, enhanced the electrostatic repulsion between the membrane surface and oil droplets. Furthermore, some organic polymers can also be used for ceramic membrane modification [195,196]. For example, Chen et al. [197] grafted highly flexible polydimethylsiloxane (PDMS) brushes onto membranes. The grafting layer improved the permeate flux and fouling resistance towards highly viscous O/W separation. In addition, the stability of the grafted polymers was found to be thermally and chemically stable since the hybrid ceramic membranes have widely been studied for nanofiltration of organic solvents [198]. Other novel ceramic membranes include photocatalytic and electrified ceramic membranes [199,200]. These membranes are typically modified by coating a semiconductive or an electrical conductive layer on the membrane surface. Although these modifications have demonstrated improvements in fouling resistance and rejection rates, the practical application of these membranes remains a subject of further study [182].

Despite that membrane fouling can be reduced after modification, there are still some challenges in relation to the modified membranes. For example, the durability and thermal stability of the coated nanoparticles or grafted polymers on the membrane surface may be a problem for practical applications. The nanostructure and properties of the coated layer could be damaged during frequent chemical or physical cleaning. In addition, the permeance of the membrane is generally lower after modification due to additional resistance of the layer formed after modification, leading to lower water production in constant pressure filtration. Furthermore, the modified layer may be effective to oil fouling, but cannot always address the fouling caused by other components (e.g. particulate fraction, dissolved organic matter and solids) in produced water [138]. In a pilot study, Miller et al. [201] compared the fouling of hollow fiber polymeric UF membranes with and without polydopamine and poly(ethylene glycol) modification for the treatment of flowback water from hydraulic shale fracturing. It was found that those modified membranes maintained a higher flux, a lower transmembrane pressure difference, and an improved cleaning efficiency relative to the unmodified modules. Regarding the application of the modified ceramic membranes for real oily wastewater, there is still a lack of relevant studies.

## 5. Conclusion and outlook

As one of the most promising technologies for oily wastewater separation, ceramic membranes have a better permeate quality, fewer chemicals additions, a longer service life and a more compact design compared to conventional methods. In terms of the type of ceramic membranes, MF and UF membranes are the most frequently used. They have shown a high oil removal efficacy for most of the oily wastewaters except for the produced water. Ceramic NF should be considered to improve oil, dissolved organics and salt rejection in produced water treatment. Developing low-cost ceramic membranes with clay or solid-state wastes has also been studied to reduce the costs of traditional ceramic membranes, long-term investigations of these membranes for oily wastewater treatment should be studied as a proof-of-concept.

Membrane fouling is one of the largest challenges for oily wastewater

treatment. The selection of proper membranes can alleviate membrane fouling. Ceramic membranes with a higher surface hydrophilicity and charge, such as SiC membranes, are considered to have less fouling. However, there is not a consistent conclusion about the effect of membrane pores on fouling. To prevent the formation of complete pore blocking, applying the membranes with a larger pore size may mitigate the fouling. However, internal fouling is also easier to be formed in membranes with a larger pore size, leading to less permeate flux recovery. To achieve better oil filtration performance, the pore size, surface hydrophilicity and charge of the ceramic membranes should therefore be optimized together. For instance, selecting a membrane with a pore size slightly larger than the oil droplets allows for modifications using state-of-the-art thin film deposition techniques, such as Atomic Layer Deposition (ALD). This technique enables the precise tuning of the membrane's pore size and controlled modification of its surface chemistry [202]. As fouling can be initiated and accelerated by surface defects of the selective layer, the integrity of ceramic membranes should be maintained via careful control of the parameters of the membrane fabrication process.

Crossflow filtration with ceramic membranes for oily wastewater treatment has been preferred, as oil accumulation on the membrane surface can be suppressed with shear forces. More attention should be paid to the research on the permeate flux effect on ceramic membrane fouling as, in practical applications, constant flux filtration is preferred, but is rarely studied for oily wastewater treatment. If constant pressure filtration is applied, TMP should be below the critical pressure to prevent the penetration of oil droplets into the pores.

Pretreatment, backpulsing/backwashing and chemical cleaning are effective to reduce the fouling, recovering the permeance and extend the lifetime of the ceramic membranes. Pretreatment including coagulation/flocculation, electrocoagulation and adsorption can increase the membrane permeate flux, but overdosing may have an adverse effect. Backpulsing/backwashing has been recommended to maintain a long filtration run, but it's not effective to remove irreversible fouling. Chemical cleaning is necessary when the accumulation of irreversible fouling is high. To fully recover the ceramic membrane performance after the treatment of real oily wastewater, a combination of the use of chemical agents (base and acid) at high temperatures should be considered. In addition, steam regeneration, ultrasonic and heating could be effective for the regeneration of ceramic membrane and to minimize the waste production caused by chemical cleaning.

Membrane modification could improve ceramic membrane performance (higher permeate flux and oil rejection) and mitigate membrane fouling during the separation process. The aim of the modification is to precoat or deposit a thin layer of protective materials on the membrane surface and/or in the pores. In this way, the negative interaction between the foulants (e.g., oil droplets) and the membrane surface can be minimized. However, few studies have been focused on the stability of the coated layer in cleaning operation, which is commonly applied in practical applications. Therefore, the durability of nanoparticles and the long-term stability of the modified membranes is still to be studied. In addition, to minimize the loss of water flux, a membrane with higher permeance should be considered for modification to improve their fouling resistance. Furthermore, in treating oily wastewater with high salinity, the membrane's charge effect on fouling is diminished. Therefore, consideration should be given to a membrane designed with surface patterns that enable a super-hydrophilic property, thereby improving its antifouling capabilities against oil droplets.

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## CRedit authorship contribution statement

**Mingliang Chen:** Writing – review & editing, Writing – original draft, Visualization, Investigation, Conceptualization. **Sebastian G.J. Heijman:** Writing – review & editing, Supervision, Conceptualization.

**Luuk C. Rietveld:** Writing – review & editing, Supervision, Conceptualization.

### 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.

### Data availability

Data will be made available on request.

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