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Membrane Fouling Mitigation: Membrane Cleaning

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Fouling is an inevitable hurdle limiting flux and performance of membrane processes. This paper reviewed the literature studies on physical cleaning methods and chemical cleaning and commented on the indices for cleaning efficiencies therein used in literature works.

Keywords cleaning; fouling; removability; resistance; reversibility

INTRODUCTION

Fouling exists ubiquitously in various types of membranes in filtration, including those on dense membranes, i.e., nanofiltration (NF) and reverse osmosis (RO) membranes, and on porous membranes, i.e., microfiltration (MF) and ultrafiltration (UF) membranes. The IUPAC Working Party on Membrane Nomenclatures has given "fouling" a definition as "the process resulting in loss of performance of a membrane due to deposition of suspended or dissolved substances on its external surfaces, at its pore openings, or within its pores" (1). Fouling of the membrane causes deterioration of membrane materials and decreased membrane performance (in terms of flux decline).

Jagannadh and Muralidhara (2) listed four approaches to mitigate membrane fouling and concentration polarization:

(i) boundary layer (velocity) control.
(ii) turbulence inducers/generators.
(iii) membrane modifications and materials.
(iv) combined (external) fields.

Williams and Wakeman (3) listed a few fouling alleviation techniques, such as feed pre-treatment, flow manipulation, gas sparging, rotating membranes, and others. Cleaning of fouled membranes is elucidated as "a process where material is relieved of a substance which is not an integral part of the material" (4). Cleaning is classified as physical and chemical cleaning, with the former incorporating only physical processes such as hydraulic, pneumatic, mechanic, and applied electric fields; and with the latter comprising use of numerous chemicals, like acids, bases, oxidants, and surfactants. In practice, physical cleaning followed by chemical cleaning is widely applied in membrane applications to confine the extent of membrane fouling. Zhang and Liu (5) used a four-step method, including water cleaning, acid cleaning (0.1N hydrochloric acid), second water cleaning, and caustic cleaning (1% w/w NaOH) to clean a hollow fiber PS/PDC (MWCO 30 kDa) membrane.

This paper provides an up-to-date summary of cleaning studies reported in pertinent literature, considering the indices proposed in these studies for characterizing membrane cleaning efficiencies.

PHYSICAL CLEANING

Table 1 summarizes studies considering physical cleaning on fouled membranes.

Physical cleaning of porous membranes includes hydraulic and pneumatic approaches and sonication (ultrasound). The hydraulic cleaning, which includes flushing (forward) and backwashing/backpulsing, is the most common and easiest technique for mitigating fouling (6). Regular intermittent backwash leads to the lift-off of deposited particles from the membrane surface and minimizes the extent of concentration polarization (7), which nowadays becomes a standard cleaning procedure in MBR and many other crossflow filtration systems. Forward flushing can be undertaken during the filtration cycle with a backwash to improve shear and remove particle concentration build-ups. Backpulsing (or called backshocking) is a more rapid backwash with a forward filtration step and followed by a reversed filtration step. Rapid backpulsing (<0.1 s) effectively removed the non-adhesive foulants from membranes (8.9). Yigit et al. (10) noted that a more intensive backwash can more readily reduce reversible fouling on MBR filtration.

The pneumatic cleaning of the membrane was termed as air sparging, air lifting, air scouring, and air bubbling. Air is applied for direct cleaning or to enhance flux in the filtration step such as water/air flushing (11). The pneumatic

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TABLE 1
Physical cleaning for fouling of various types of membranes. MF: microfiltration; UF: ultrafiltration; PE: polyethylene; PVDF: Polyvinylidine Fluoride; PP: polypropylene; CA: cellulose acetate; MBR: membrane bioreactor; HF: hollow fibre membrane; OF: organic fouling; BF: biofouling; IF: inorganic fouling

Type	Membrane configuration, materials and specs*	Feed (application)	Physical cleaning approaches	Fouling type(s)	Reference
MF	flat sheet, CA (0.2 μm)	washed yeast suspension (*Sacchromyces cerevisae*)	frequent forward filtration (1–40 s) and reversed filtration (backwashing, 0.5–4 s)	OF, BF	Redkar and Davis (9)
MF	MBR, immersed ZW-10 module (ZeeWeed® 500) PVDF/HF (0.2 μm)	screened raw domestic wastewater (MLSS = 6,600–6,800 mg l^{-1})	seven backwash scenarios: backwash time (s)/total filtration time (min) = 0/60, 15/60, 15/25, 5/10, 15/10, 20/10, 15/5	BF, OF, IF	Yigit et al. (10)
MF, UF	HF PE (0.1 μm) and PVDF (0.1 μm); PAN(MWCO 100 kDa)	surface water, contains NOM, metals (Al, Ca, Mg, Mn)	(1) hydraulic backwash, 60 s; (2) air scrubbing, 30 s; (3) filtration, 30 min	OF, IF	Yamamura et al. (19)
MF	MBR, HF PVDF	wastewater	backwash with DI water, sonication (42 kHz), combination with chemical cleaning (acidic and caustic)	BF, OF, IF	Lim and Bai (22)
MF	PP, HF (0.2 μm)	surface water	periodic backwash (10 steps, with/out compressed air supply by valves control)	BF, OF, IF	Chellam et al. (46)
MF	PVDF, HF (0.1 μm)	Bentonite suspension	backwash	OF	Marselina et al. (57)
MF	MBR, hollow fibre, PVDF (pore size 0.22 μm)	alginate and bentonite mixture	Backwash	BF	Le-Clech et al. (58,59)
MF	MBR, flat sheet, PVDF (0.22 μm)	wastewater (mixed liquid suspended solid, MLSS)	bubble size transformer was used to coalesce fine bubbles to coarse bubbles	BF, IF	Phattaranawik et al. (60)
MF	MBR, HF, PE (0.1 μm)	surface water containing natural or reconstituted humic acid	off-line clean by manual sponge cleaning every 11 h, periodical backwashing, 30 s (50 kPa)	coagulated surface water	Kimura et al. (61)
UF	regenerated cellulose, RegC (MWCO 10 kDa)	Cu-PEI solution and W/O emulsions dispersed in aqueous solution	ultrasonic cleaning (20 kHz)	OF	Juang and Lin (18)

(*Continued*)

TABLE 1
Continued

Type	Membrane configuration, materials and specs*	Feed (application)	Physical cleaning approaches	Fouling type(s)	Reference
UF	PES (MWCO 3, 10, 30, 100 kDa), PVDF (40 kDa), PAN (40, 50 kDa)	DI water with supporting electrolyte (HCl dissolved in 0.1 $moll^{-1}$ NaCl)	ultrasonic bath (47 kHz), total duration is two hour: (1) 5 minutes for 3 times, (2) 10 min for 3 times, (3) 15 min for 3 times, (4) finally 30 min.	none	Masselin et al. (20)
UF	flat sheet, PES (MWCO 10 kDa)	aqueous extract of *Radix astragalus*	cleaning by DI water with ultrasonic, (2)ultrasonic cleaning with/out chemical cleaning (0.1M NaOH)	OF	Cai et al. (23)
UF	PES (MWCO 50 kDa), PA (50 kDa), RegC (30 kDa), PVDF (20 kDa)	BSA	alternating electric field strength (0–80 $V \cdot cm^{-1}$), frequency (0.5–50 Hz)	OF	Zumbusch et al. (25)
UF	inorganic membrane (Carbosep® M8, MWCO 50 kDa)	proteins (BSA)	cleaning by electric field (DC = 15 V)	OF	Tarazaga et al. (26,27)
UF, MF	Commercial MF (PVDF, 0.2 μm), UF (PS, MWCO 10 kDa) and lab-made UF (PAN8 & PAN15)	peptone solution	(1) ultrasonic cleaning (frequency of 45 kHz, power of 2.73 $W \cdot cm^{-2}$), (2) water cleaning, (3) water cleaning under sonication.	OF	Chai et al. (49)
UF	two rotating membrane disks, PS (MWCO 750 kDa)	synthesized wastewater with nitrifying bacteria seeding	increasing shear-stress by speed-up the rotation disk (from 50 rpm to 400 or 500 rpm) and using mechanic cleaning by sponge cube in another run	BF, OF, IF	Kimura et al. (62)

UF	PS (MWCO 750 kDa)	NOM and polysaccharide	(1) sponge cube cleaning for reversible fouling (immersed in water, rotation at 20–70 rpm, 60 min); (2) filtration, 30 min; (3) pause, 2 min	OF	Kimura et al. (62,63)
UF	hollow-fiber, PES (MWCO 150 kDa)	humic acid and sodium alginate solution	periodic backwash (5 min backwash/1 h total filtration duration)	OF	Katsoufidou et al. (63)
UF	PS (MWCO 50 kDa)	fouling of BSA	AC electric field (800 V/m)	OF	Mameri et al. (65)
UF	MBR, mixture of CA and CN (0.1 and 0.22 μm)	biological suspended solids (MLSS 8,000 $mg \cdot L^{-1}$)	intermittent DC electric field (6 $V \cdot cm^{-1}$), switched on/off (every 90 s)	OF, BF	Akamatsu et al. (66)
UF	flat sheet, PS(MWCO 30 kDa)	reconstituted whey protein solution	ultrasonic bath (50 kHz, 300 W)	OF	Muthukumaran et al. (67)
UF	dead-end and crossflow, PS (MWCO 35 kDa)	paper mill effluent	(1) forward flushing, (2) ultrasonic cleaning, (3) (frequency of 20 kHz, power of 375 W), (4) ultrasound together with forward flushing, each for cleaning times of 20 min	OF	Li et al. (68)
NF	three spiral wounds module, ESNA2 (TFC PA layer, supported by a PS)	tap water (containing traces of iron and manganese, <0.01 mg/l)	sporadic (>1 week) and daily (1 $h \cdot d^{-1}$) air/water cleaning (ratio of water/air = 1/2 (700/1400 $L \cdot h^{-1}$), reverse flow)	BF and particulate fouling	Cornelissen et al. (13)
RO	flat sheet, PA	wastewater (containing $CaSO_4$, $FeCl_3$ and carboxyl cellulose)	ultrasonic clean (20 kHz, 2.8 $W \cdot cm^{-2}$)	BF, OF	Feng et al. (69)

cleaning process benefits in its low maintenance cost, ease to integrate in the existed system, and the use of no cleaning chemicals. However, the disadvantages of air sparging include limited effectiveness in cleaning and the high pumping cost. The combined cleaning of air sparging and hydraulic backflush (AS + BF) is commonly applied in MBR (12) and spiral wound NF to reduce biofouling (13).

Mechanical cleaning, such as sponge ball wiping, is an effective physical cleaning process (14,15). A large-diameter tubular membrane can be cleaned mechanically by using sponge balls (16).

Chen et al. (17) discussed the effects of the production interval between physical cleaning (0.5 h or 3 h), the duration of forward flush (1 min or 5 min), the duration of backwash (1 min or 5 min), the pressure during forward flush (1.72 bar or 3.45 bar), the type of water used (RO permeate or tap water), and the sequence of forward flush (F) and backwash (B) (either F + B or B + F), on cleaning efficiencies of UF membranes. Juang and Lin (18) developed a correlation for the reversible resistance for their ultrasound oil/water filtration tests.

Ultrasound irradiation (sonication) is another effective physical cleaning method, but has received comparatively lesser attention in literature. Ultrasound waves produce cavitation and induce acoustic streaming, which provide vigorous mixing to breaking concentration polarization and cake layer on the membrane surface. However, it cannot influence the intrinsic permeability of the membranes (19). The use of high energy, ultrasonic pulse in membrane cleaning can break absorbed foulants and delodge detached bacterial biofilm on the membrane surface. Effects including ultrasound frequency, power intensity, feed properties, membrane materials, crossflow velocity, temperature, and pressure are needed to be considered by using this physical cleaning (19). Lower ultrasound frequencies are preferred for membrane cleaning. Masselin et al. (20) commented that the polyethersulfone (PES) membranes are more readily damaged by the ultrasound waves compared with the polyvinylidene fluoride (PVDF) and polyacrylonitrile (PAN) membranes. Li et al. (21) used ultrasonic cleaning, forward flushing, and a combined approach of the two to clean the flat sheet nylon microfiltration membranes. Lim and Bai (22) tested the effects of sonication duration and the combination with other physical and chemical cleanings on flux recovery of hollow fiber PVDF membranes in the MBR process. Cai et al. (23) compared two types of the ultrasonic source (ultrasonic transducer plate and probe) in cleaning a UF membrane fouled by an aqueous extract of *Radix astragalus*.

Applying electric fields to alleviating membrane fouling is regarded as a physical cleaning method although it is traditionally used to enhance transport through membranes (24). Zumbusch et al. (25) applied alternating electrical fields to minimize fouling in ultrafiltration of biological suspensions. These authors also introduced a "step-change" procedure to perform crossflow filtration. Williams and Wakeman (3) examined how DC electric fields affected MF membrane fouling and rejection. Tarazaga et al. (26,27) applied electric field to clean biofouled inorganic UF membrane (i.e., Carbosep® M8, ZrO_2-TiO_2 on carbon support). The exponential flux decline was characterized by various technical parameters.

Intermittent filtration (or "membrane relaxation mode") is a "passive" physical cleaning step as no "active" cleaning methods are involved. The intermittent filtration mode effectively reduces fouling in suction-type membrane operation, e.g., submerged MBR, and normal pressure-driven type operations, e.g., tangent-flow (cross flow) and dead-end operations. Such relaxation mode during the filtration process if carried out in a shorter period could be seen as an operation scheduling or flow manipulation for fouling elimination, such as pulsating flow or periodic air/water cleaning. Cornelissen et al. (13) conducted and compared different physical and chemical cleaning in parallel in three individual spiral-wound modules, and noted that daily air/water flushing presented very effective procedure in minimizing membrane fouling.

CHEMICAL CLEANING

Chemical cleaning presents the major method to restore and maintain the "expected" permeability and selectivity in most membrane processes. Chemical cleaning can be carried out in various ways:

i. directly immersing the fouled membranes in the chemicals, i.e., "clean-in-place (CIP),"
ii. soaking in a separate tank with higher concentration cleaning agents, i.e., "clean-out-off-place (COP)."
iii. adding chemicals in the feed stream, i.e., chemical wash (CW), or
iv. cleaning in conjunction with the physical cleaning step, i.e., a chemical enhanced backwash (CEB).

Most chemical cleaning agents are commercially available and many of them were recommended by membrane manufacturers to deal with different types of foulants in the feeds. For example, acid cleaning is often used to remove precipitated salts or scalants (such as $CaCO_3$); while caustic cleaning is suitable for removing adsorbed organics. The recipes of cleaning agents should be varied depending on the applications, feed characteristics (e.g., pH, ionic strength, temperature, and other "significant" metals or compounds), and membrane materials (as some are more vulnerable). Chemical cleaning agents were normally classified into five categories:

a. alkaline.
b. acids (e.g., nitric, phosphoric, hydrochloric, sulphuric, citric acids),

c. metal chelating agents,
d. surfactants (i.e., surface-active agents, including anionic, cationic, non-ionic and amphoteric electrolytes),
e. enzymes.

In addition to the five main categories, disinfectants (O_3), oxidants (e.g., H_2O_2, $KMnO_4$), or sequestration agents (e.g., EDTA) are often used for cleaning chemicals of membranes. A blend of various cleaning agents or combination with other physical cleaning are also commonly adopted (28). Zondervan and Roffel (29) listed several common chemical cleaning agents, including caustic (NaOH, KOH, NH_4OH), acidic (HCl, HNO_3,H_2SO_4, H_3PO_4, citric, oxalic), sequestering/complexing (EDTA), detergent/surfactant (alkyl sulphate, SDS, CTAB), enzymatic (α-CT, CP-T, peroxidase), oxidative/disinfectants (NaOCl, H_2O_2, $KMnO_4$), and blend cleaning (4 Aquaclean®, Divos®, TRiclean®, Ultrasil®/Aquaclean®). In addition to the types of chemical agents, other factors affecting the chemical cleaning efficiency include cleaning time, concentration, cleaning temperature, and flux. Chen et al. (17) evaluated the effects of recirculation duration of high pH cleaning (0.5 h or 1.0 h), concentration of high pH cleaning solution (0.5% or 1.0%), the temperature of the high pH cleaning solution (25°C or 50°C), static soak (0 or 0.5 h), and forward flush (F), or backwash (B) after chemical cleanings on cleaning UF membranes.

The possible interactions of chemical agents and fouled membranes include: hydrolysis, peptization, saponification, solubilization, dispersion (suspension), and chelation. The preferred cleaning agents in relation to membrane foulants source (i.e., proteins, glucanes, pigments, minerals, hydrophobes, starch, tannins, pectin, and fat) are also addressed. In addition, they evaluated the efficiency of the cleaning agents and optimized the cleaning sequence. Weis et al. (30) illustrate that a cleaning agent can affect fouling materials presenting on membrane surface in the following three ways:

i. the foulants may be removed,
ii. the morphology of foulants may be changed (swelling, compaction) and/or
iii. the surface chemistry of the deposit may be altered, such that the hydrophobicity or charge is modified.

Chemical cleaning processes at membrane surface can be divided into six stages:

1. bulk reactions.
2. transport of detergent to interface,
3. transport of detergent into foulant layer,
4. cleaning reactions in the fouling layer,
5. transport of cleaning reaction products back to interface,
6. transport of product to bulk solution. Exposure of the membrane in the cleaning agent of too high concentration and for too long a period of time would damage membranes structure (31).

A complete chemical cleaning cycle (CC) comprised several filtration cycles (FC) and a subsequent chemical cleaning phase (C). The FC is comprised of the filtration phase (F) and the baskwash phase (B). Zondervan and Roffel (32) evaluated the effects of different chemical cleaning agents on removing UF membrane fouling. Zondervan et al. (33) modelled the dynamic decay of irreversible fouling resulted from ultrafiltration of the surface water, which can predict the states of irreversible fouling as a function of cleaning time, cleaning flux, and initial cleaning agent concentration of each cleaning cycle. However, the parameters adopted in this model are somehow oversimplified, such as characterizing the cleaning effectiveness of irreversible fouling by turbidity integral and the chemical cleaning agent concentration by pH. Moreover, the ratio of the flushing rate constant and cleaning rate constant can be used to characterize the effectiveness of cleaning irreversible fouling and is mainly dominated by either mechanical (flushing) or chemical aspects.

Popović et al. (34) evaluated the cleaning efficiency of alkali (0.2% and 1% w/w NaOH) and detergent (0.2% w/w P3-ultrasil 69 + 1% w/w P3-ultrasil 67; 1.2% w/w P3-ultrasil 69 and 0.75% w/w P3-ultrasil 67). These authors proposed a kinetic model for the two chemical cleanings of a whey-protein-fouled ceramic membrane of average pore size 200 nm (SCT, Bazet, France).

Table 2 summarizes studies considering chemical cleaning on fouled membranes.

CLEANING EFFICIENCY

Bowen and Jenner (35), Belfort et al. (36), van den Berg and Smolders (37), and Fane and Fell (38) reviewed quantitative models for membrane filtration data. The fouling of membranes in general can be interpreted by Darcy's law as follows:

$$J = \frac{dV}{A \cdot dt} = \frac{\Delta P}{\mu \cdot (R_0 + \sum R_i)} \quad (1)$$

where J, is the volumertric flux of permeate, V is the accumulated volume of permeate, A is the membrane surface area, ΔP is the pressure drop imposed across the fouling layer and membrane, μ is the permeate viscosity, R_0 is virgin membrane filtration resistance and determined by clean water flux measurement, and ΣR_i is the resistances-induced during filtration (39).

The cleaning efficiency of fouling NF membranes of surface water were evaluated (40) by the *flux recovery* and the *fouling ratio* as follows:

$$Flux\ recovery = J_c/J_0, \quad (2)$$

TABLE 2
Chemical cleaning for fouling of various types of membranes

Type	Membrane configuration, materials and specs	Feed (application)	Cleaning agents	Fouling type(s)	Reference
MF	HF, PVDF	wastewater	acidic cleaning, caustic cleaning, oxidative cleaning	BF, OF, IF	Lim and Bai (22)
MF	Monotabular ceramic (pore size 200 nm)	reconstituted whey protein	alkali cleaning (NaOH), detergent cleaning (P3-ultrasil® 69 and P3-ultrasil® 67 in various ratios)	OF	Popović et al. (34)
MF	Polycarbonate (0.80 μm)	mixture of BSA, dextran and tannic acid	enzymatic cleaning (P3 Ultrasil® 53)	OF	Zator et al. (44,45)
MF	tubular, ceramic, (0.1 μm)	reconstituted whey protein (Protarmor® PS90)	caustic cleaning (1 wt.% NaOH)	OF	Blanpain-Avet et al. (51)
MF	Tami® 150 + 4T (MWCO 400 kDa); Carbosep® M6 (MWCO 340 kDa)	reconstituted whey protein (Protarmor® PS90)	detergent cleaning (Maxatase® XL, P3-Ultrasil® 62 and NaOH for adjusting pH)	OF	Blanpain-Avet et al. (50)
MF	flat-sheet, (0.22 μm, Isopore®)	protein (green fluorescent BSA and red fluorescent ovalbumin)	caustic cleaning (NaOH), oxidative cleaning (free Cl from NaOCl), enzymatic cleaning (Ultrasil® 53)	OF	Field et al. (70)
MF	HF, submerged, PE (0.1 μm)	coagulation pretreated (PACl), surface water	acidic cleaning (2% HCl), caustic cleaning (2% NaClO)	BF, OF, IF	Mo and Huang (71)
MF	HF, PE (0.1 μm)	seawater contaminated with crude oil and fuel spillages	(1) acidic cleaning (oxalic acid), (2) caustic cleaning (caustic soda), (3) blend cleaning	BF, OF, IF	Al-Obeidani et al. (72)
MF	MBR, flat sheet, PVDF (0.1 μm)	wastewater (sludge supernatant)	(1) detergent (NaOCl, 200 ppm and 1,900 ppm Cl^-), (2) detergent (NaOH, 0.013 wt%), (H_2O_2, 0.5 wt%) (A3 Activor® A101, 1 wt%); (3) enzymes (Ultrasil® 67, 0.5 wt% + Ultrasil® 69new, 1 wt%), (Filzym® p, 1 wt% and 2 wt%), (A3 SERL®, 1 wt% and 4 wt%); (4) acid (HCl, 0.056 wt%), (citric acid, 1 wt%) (A3 Activor® A103, 1 wt%)	BF, OF	Grélot et al. (73)

UF	HF, blend of PS/PDC (MWCO 30 kDa)	wastewater from banknote printing works	(1) DI water cleaning, (2) acid cleaning (0.1N hydrochloric acid), (3) second DI water cleaning, (4) caustic cleaning (1 wt.% sodium hydroxide)	OF	Zhang and Liu (5)
UF	hollow fiber (Norit-Xiga FSU®)	Surface water (canal water)	HCl, H_2SO_4, citric acid, NaClO, P3 Ultrasil® 115, P3 Ultrasil® 70, P3 Aquaclean® Sal, 4 Aquaclean® Fer 12, Kleen® MTC 411	BF, OF, IF	Zondervan and Roffel (29)
UF	PES	sulphite liquor	Caustic cleaning, blend cleaning	OF, IF	Weis et al. (30)
UF	dead-end, PS (MWCO 30 kDa)	model tea component solution	caustic cleaning (NaOH, 0.2 wt.%)	OF	Wu and Bird (48)
UF	PS	BSA/WPC	caustic cleaning, detergent cleaning, enzymatic cleaning (CTAB, Treg-A-Zyme, α-CT)	OF	Munoz-Aguado et al. (54)
UF	capillary membranes, PS (MWCO40 kDa)	natural occurring brown water (surface water) and commercial humic acid make-up solution	(1) NH_4OH (1%) 1 h, (2) NH_4OH (1%) +$CaSO_4$(1%) 1 h, (3) NH_4OH (2%) 2 h, (4) repeat washing last solution 1 h, (5) NH_4OH (1.5%) + Triton® X100(0.1%) 1 h, (6) flusing last solution with DI water 2 h, (7) NaOH + SDS + EDTA ($1\,g \cdot L^{-1}$) 1 h, (8) NH_4OH (2%) + EDTA 1 h, (9) by Biotex® (1%) 1 h.	OF	Maartens et al. (56)
UF	PS (MWCO 750 kDa)	NOM and polysaccharide)	(1) NaCl (0.1M); (2) EDTA (20 mM); (3) HCl (pH 2); (4) oxalic acid (pH 2); (5) NaOH (pH 12); (6) NaClO (500 ppm)	OF	Kimura et al. (63,79)
UF	spiral-wound, PES (MWCO100 kDa)	whey protein, long-term fouling (14 h each run)	(1) heating 20 L DI water to 50°C, (2) dissolving and recirculating 1% (w/w) Terg-a-zyme (a protease-active detergent) for 1.5 h; (3) soaking for 1 h; (4) recirculation for 1 h; (5) rinse with tap water for 1 h	OF	Yee et al. (74)
UF	PS	wastewater	acidic cleaning, detergent cleaning, sequestering cleaning, oxidative cleaning, enzymatic cleaning, blend cleaning	BF, OF, IF	Mohammadi et al. (75)
UF	PAN	wastewater	caustic cleaning, oxidative cleaning	BF, OF, IF	Pavlova (76)
UF	spiral wound, (MWCO 20 kDa, NIROSOFT RM10–8®, 8040SW)	secondary wastewater	caustic cleaning	BF, OF	Gillerman et al. (77)

(*Continued*)

TABLE 2
Continued

Type	Membrane configuration, materials and specs	Feed (application)	Cleaning agents	Fouling type(s)	Reference
UF	dead-end,: PES with PP backing (MWCO 25 and 40 kDa), PVDF (70 kDa,), PES (5–10 kDa)	Whey protein concentrate (WPC 80)	caustic cleaning (Ultrail® 91), acid cleaning (Ultrail® 75)	OF	Lawrence et al. (78)
UF	inorganic, ZrO_2 filtering layer (Carbosep® M6, MWCO340 kDa)	reconstituted whey protein	enzymatic cleaning (Alcalase®)	OF	Argüello et al. (80)
UF	inorganic (Tami®150 + 4T, MWCO 400 kDa)	reconstituted whey protein (Protarmor PS90)	enzymatic cleaning (P3-Ultrasil® 62 and Maxatase® XL)	OF	Argüello et al. (81)
UF	inorganic, ZrO_2 filtering layer (Carbosep® M1, MWCO150 kDa)	reconstituted whey protein (Protarmor PS90)	enzymatic cleaning (P3 Ultrasil® 62), NaOCl used for conditioning and disinfection after the cleaning	OF	Argüello et al. (82)
UF	capillary membranes, PS (MWCO40 kDa)	natural occurring brown water (surface water) and commercial humic acid make-up solution	Triton® X100(0.1%) and Pluronic® F108	OF	Maartens et al. (83)
UF	PES	paper effluent	caustic cleaning, detergent cleaning, oxidative cleaning, enzymatic cleaning, blend cleaning	OF	Maartens et al. (84)
UF	flat sheet, sulphonated PES (NTR 7410®, MWCO 20 kDa)	surface water and ground water	acidic cleaning (citric acid, 0.1M), caustic cleaning (NaOH, 0.1M), surfactant cleaning (SDS, 0.001M)	OF	Lee et al. (85)
UF	PS (GR51®, MWCO 50 kDa), modified PS (HEKLA20A®, 20 kDa)	BSA and LYS	NH_4OH, MP (machine powder), sodium hypochlorite, sodium hydroxide, oxalic acid, citric acid	OF	Zhu and Nyström (86)

UF	PS (MWCO 50 and 100 kDa)	aqueous extract of soy flour	hydrochloric acid (0.5 wt%, pH 1.5), sodium hydroxide (0.5 wt%, pH 12.5), protease detergent (0.75 wt% Terg-a-zyme®), sodium hypochlorite (150 ppm)	OF	Sayed Razavi et al. (87)
UF	flat sheet, PS	abattoir effluent	enzymatic cleaning (serine protease A, lipase A, Alkazyme® and Zymex®), detergent cleaning, (SDS and Triton × 100)	OF	Maartens et al. (88)
NF	spiral wounds, ESNA2® (TFC PA layer)	tap water	copper sulphate dosing	BF and particulate fouling	Cornelissen et al. (13)
NF	NF255–400® (MWCO 200–300 Da, PPA)	surface water	citric acid, oxalic acid, HCl, $Na_2S_2O_4$, Ultrasil® 73, Ultrasil® 141, Noah, Na_4EDTA $Na_5P_3O_{10} + Na_4EDTA$, NaOH+Na_4EDTA	BF, OF, IF	Liikanen et al. (40)
NF	MPF®-34 (MWCO 200 Da) MPF®-3 (1000 Da)	textile effluent	0.2 wt.% HNO_3 and subsequently 0.5 wt.% NaOH	Dye	Sungpet et al. (96)
NF	dead-end flat sheet, NF270®, TFC	commercial humic acid	(1) alkaline solutions (pH 11); (2) metal chelating agents; (3) surfactants	OF	Li and Elimelech (97)
NF	DK®, HL®, DL® (TFC, MWCO 150–300 Da)	saline water	HCl (1M), NaOH (2M), SDS (0.1%), mix agent (0.1%) of trisodium phosphate (TSP), sodium tripoly- phosphate (STP) and EDTA, NaOH followed by HCl (2M and 1M)	OF	Al-Amoudi et al. (98)
UF RO	spiral wound PS, PA	Municipal wastewater reclamination	hydrochloric acid, sodium hydroxide, TriClean® 212 F	OF	Chen et al. (17)
RO	FT30®, PA	Whey (cheese manufacturing)	acidic cleaning (HCl, HNO_3, H_2SO_4, H_3PO_4, $C_2O_2(OH)_2$, NH_4Cl), caustic cleaning (NaOH), surfactants cleaning (SDS, CTAB), complexing agent (EDTA) ammonia, urea, surfactants (SDS, Triton®-X100, CTAB)	OF	Madaeni and Mansour-panah (47)
RO	PA(ESPA3®)	mining effluents ($2\,g \cdot l^{-1}$ calcium carbonate solution)	cleaning with water under the same pressure (20 ± 0.5 bar) and cross-flow conditions ($50 \pm 15\,ml \cdot min^{-1}$)	IF	Sanderson et al. (89)
RO	modified by oxidants	wastewater (secondary effluent)	oxidizing agents: H_2O_2 + SS, $KMnO_4$, NaClO, NaClO + SDS, NaClO + H_2SO_4,NaOH	IF	Veza and Rodriguez-Gonzalez (90)

(*Continued*)

TABLE 2
Continued

Type	Membrane configuration, materials and specs	Feed (application)	Cleaning agents	Fouling type(s)	Reference
RO	TFC (LFC-1®)	alginate and NOM	NaOH, EDTA and SDS	OF	Ang et al. (91)
RO	organic-fouled RO, PA TFC (LFC-1®)	polysaccharide and commercial NOM	salt cleaning	OF	Lee and Elimelech (92)
RO	FT30®, PA	boiler water	hydrochloric acid, nitric acid, sulphuric acid, phosphoric acid, citric acid, sulphamic acid, sodium hydroxide, potassium hydroxide, ammonium hydroxide, sodium hypochlorite, ammonium chloride, EDTA, SDS, two commercial dish washing detergents (Yekta® and Goli®)	IF	Madaeni et al. (93)
RO	PA (BW30®)	sugar (sucrose) solution	acidic cleaning (HCl, HNO_3), surfactants cleaning (SDS, CTAB), complexing agent (EDTA), ammonia	OF	Madaeni et al. (94)
NF, RO	spiral wound ESNA® 2540, RE®-2540-TN, ESPA®-2540 (PA and MWCO 200 Da)	ground water	in the order of hydraulic washing (200 ml DI water), acid cleaning (0.1N sulphuric acid), and alkaline cleaning (0.1N sodium hydroxide)	OF	Gwon et al. (95)

where J_c is the flux after the application of a certain cleaning solution or total cleaning procedure, J_0 is the flux of the virgin membrane, and

$$Fouling\ ratio = J_f/J_0 \tag{3}$$

where J_f is the flux for fouled membrane (in this case, after every week's run). The cleaning efficiency of the applying electric field was evaluated by Tarazaga et al. (26,27) using an index identical to the *Flux recovery* in Eq. (2).

Yamamura et al. (41) used an index, *decline ratio* of pure water flux (J_f/J_c), to characterize fouling on various stages and efficiency of chemical cleaning on MF membrane for surface water and ceramic UF membrane for whey protein concentrates (WPC) characterize. Factors affecting the chemical cleaning efficiency (flux recovery), such as NaOH concentration, temperature, crossflow velocity, and TMP, were investigated by Bird and Bartlett (42). An index for evaluating the cleaning efficiency, called "percent flux recovery ($\%J_r$)," was proposed with a definition similar to the flux recovery in Eq. (2).

The extent of fouling (for 40 h continuous experiment) for NF 200, NF 270, and the laboratory-modified membranes are characterized below (43):

$$Fouling\ percentage(\%FR) = 100(J_c(i-1) - J_f(i))/J_c(i-1) \tag{4}$$

where $J_c(\mathrm{i}-1)$ is the pure water flux for the membrane cleaned at cycle $\mathrm{i}-1$, and $J_c(\mathrm{i}-1)$ is the pure water flux for the membrane fouled at cycle i.

Zator et al. (44,45) used two similar indices to evaluate the cleaning efficiency of the membrane: *Pure water flux reduction* (*FRED*) is defined as follows:

$$FRED(\%) = 100(J_0 - J_f)/J_0 \tag{5}$$

Clearly, $FRED(\%) = 100$(1-*Fouling Ratio*(Eq. (3)), and *FREC* is identical as the Flux recovery defined in Eq. (2).

Chellam et al. (46) evaluated the baskwash efficiency by

$$n = 100(p_f - p_c)/(p_f - p_0), \tag{6}$$

where p_f, p_c, and p_0 are pressures applied for the fouled membrane, the cleaned membrane, and the virgin membrane, respectively. Under constant-flux test, η represents the fraction of the fouled resistance that is removed by the applied cleaning.

Madaeni and Mansourpanah (47) evaluated the chemical cleaning efficiency of fouled RO membranes by following two parameters: resistance removal ($RR\%$) and flux recovery ($FR\%$):

$$RR\% = [(R_f - R_r)/R_f]^*100 \tag{7}$$

where R_f is the resistance remaining after water flushing and R_r is the resistance remaining after chemical cleaning, and

$$FR\% = 100(J_c - J_{wf})/(J_0 - J_{wf}) \tag{8}$$

where J_{wf} is the permeate flux for the fouled membrane after simple water flushing. Restated, in case the water flushing cannot remove a significant part of the fouled materials, $FR\% = [1$-*Flux recovery* (Eq. (2))$/$*Fouling ratio* (Eq. (3))] and $FR\% = [\%FR$(Eq. (4))$/FRED\%$(Eq. (5)]* (J_c/J_0). Wu and Bird (48) also used the flux recovery ($FR\%$) and, additionally, the fouling resistance recovery (*FRR*) to evaluate the cleaning efficiency of ultrafiltration for the model tea component solution, with the latter defined as follows:

$$FRR\% = [(R_f - R_c)/(R_f - R_0]^*100 \tag{9}$$

where R_f and R_c present the pure water flux resistances for the fouled and the cleaned membrane, respectively. That is, $\mathrm{FRR}\% = \eta$ defined in Eq. (6) for constant flux tests. Chai et al. (49) used the cleaning efficiency (E_c) to compare the effectiveness of three physical cleaning approaches (i.e., ultrasonic cleaning, water cleaning, and water cleaning under sonication) in microfiltration and ultrafiltration of a peptone solution. The definition of E_c is in fact equivalent to $FRR\%$ in Eq. (9).

Chen et al. (17) evaluated the efficiency of membrane cleaning by the following three parameters:

i. clean water flux (CWF) recovery [%], which is the same as Eq. (5);
ii. wash water usage, defined as (volume of wash water used/total volume water produced); and
iii. improvement in TDS rejection, defined as: [TDS rejection before chemical cleaning]-[TDS rejection after chemical cleaning].

Blanpain-Avet et al. (50) characterized the cleaning efficiency of a tubular ceramic filtration MF membrane, which fouled with a whey protein concentration suspension. Parameters, such as "relative flux decline $(\mathrm{RFD} = [1 - (J_f/J_0)] \times 100)$," identical to *Fouling ratio* in Eq. (3), were used to evaluate the membrane performance during filtration (fouling); and the "percent flux recovery $(FR\% = (R_0/R_{ci}) \times 100)$" and "hydraulic cleanliness criterion (HCC)" were used for the cleaning effectiveness evaluation. The hydraulic cleanliness criterion $(\mathrm{HCC} = (R_{ci} - R_0)/R_0)$ representing the proportion of the residual fouling resistance left after cleaning in the i-th cycle $(R_{ci} - R_0)$ to the initial membrane resistance (R_0), identical to (1/Flux recovery (Eq. (2))-1 nuder constant pressure filtration. The cleaning approach is considered effective while HCC <0.05. A "residual fouling resistance (R_{ir})"

was proposed to characterize the sequence chemical cleaning efficiency as follows:

$$R_{ci}/R_0 = 1 + R_{ir}/R_0 \quad (12)$$

Blanpain-Avet et al. (51) further introduced another parameter, namely "percent irreversible removed fouling (*RF*)," to evaluate the hydraulic and chemical membrane cleanliness of whey protein fouled ultrafiltration membranes, as follows:

$$RF = \frac{R_f - (R_n - R_m)}{R_f} \times 100(\%) \quad (13)$$

This index lacks sound physical meaning in its definition.

The cleaning efficiency (CE), adopted by Matzinos and Alvarez (52), assessed the cleaning process by the following parameter:

$$CE = \frac{R_r - R_c}{R_r - R_m} \times 100 \quad (14)$$

where R_0 is the clean membrane resistance, R_r is the resistance of the irreversible fouling deposit, and R_c is the cleaning resistance. Restated, CE represents the fraction of fouling removed during cleaning, but based on the total fouling resistance, indicating the fraction of cleaning efforts that is effective for removing fouling. The cleaning efficiency proposed by Petrus et al. (53) was based on the same arguments. The solute resistance removal was defined by Munoz-Aguado et al. (54) to evaluate the enzymatic and detergent cleaning (i.e., CTAB, Treg-A-Zyme®, α-CT) efficiency also regarded the fraction of foulants removed on the basis of the removable and irremovable foulants.

Fabris et al. (55) proposed an index to evaluate the effectiveness of pre-treatment in fouling reduction. This definition ignored the occurrence of the chemically irreversible fouling part in filtration.

Maartens et al. (56) introduced an index called "E_4/E_6 ratio," which is the UV absorption ratio at 465 nm (E_4) and 656 nm (E_6), to quantify the efficiency for the membrane rejection for naturally occurred brown water. This ratio, together with the percentage decrease of the original pure water flux (% PWF), successfully evaluated the effectiveness for various chemical cleaning agent recipes and schemes.

Clearly from the above summary the cleaning studies principally described the cleaning efficiencies for specific fouling occurred in a few membrane/water systems via prescribed cleaning processes. Using similar terminologies, researchers are dealing with different parts of the same fouling layer, but the lack of solid ground makes the direct comparison for the used cleaning agent/procedures derived from different studies difficult. A systematical analysis for how a cleaning agent reacts with foulants and membrane is needed to provide a common platform for cleaning studies

CONCLUSIONS

Membrane fouling needs to be mitigated to maintain an acceptable flux over a long-term operation. Cleaning is classified as physical and chemical cleaning, with the former incorporating only physical processes such as hydraulic, pneumatic, mechanic, and applied electric fields; and with the latter comprising use of numerous chemicals like acids, bases, oxidants, and surfactants. This paper reviews the up-to-date studies on physical and chemical cleaning of membranes. The ways of evaluating cleaning efficiencies were discussed.

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