



Review Paper

Recent Developments in Chemical Techniques for Foulant Removal from NF Membranes

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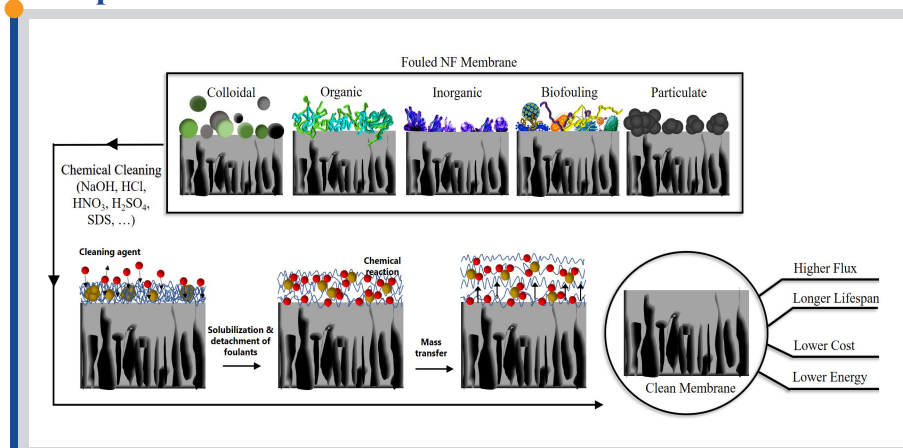
Keywords

Nanofiltration
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Highlights

- Recent chemical techniques for foulant removal from NF membranes are discussed.
- Various organic and inorganic fouling types have been investigated.
- Several factors affecting NF membrane fouling are addressed.
- Various aspects influencing the efficacy of chemical cleaning are reviewed in detail.

Graphical abstract



Abstract

Nanofiltration (NF) membranes find extensive use in many fields such as food processing, water treatment, and resource recovery due to their exceptional capacity to selectively separate small solutes and exhibit high permeability to water. Nonetheless, the occurrence of membrane fouling is an unavoidable consequence over extended periods of operation, hence imposing constraints on the widespread use of NF technology at a larger scale. Chemical cleaning is widely acknowledged as the most efficient method for mitigating fouling, subsequently restoring membrane permeability. The repeated use of chemical cleaning methods may induce both reversible and permanent alterations in the chemical and physical characteristics of NF membranes. These changes can lead to membrane degradation and a decline in separation efficiency. Additionally, the NF membrane, characterized by its high selectivity and relatively low degree of crosslinking, exhibits greater sensitivity to chemical cleaning procedures. Despite the extensive work reported for chemical cleaning of NF membrane, a systematic discussion and analyses of these approaches have not been performed. This review offers an overview of NF selectivity and separation fundamentals, exploring various factors affecting fouling in NF membranes. It provides a thorough examination of multiple aspects pertaining to the chemical cleansing of membranes. The examination encompasses the comprehension of the goals that direct this cleaning procedure, the calculated methods utilized, the complex mechanisms in operation, and the methodical processes that are fundamental to the overarching methodology. An in-depth exploration of these facets is intended to yield a nuanced comprehension of membrane chemical cleansing. In addition to the comprehensive discussion on elements that influence the efficacy of chemical cleaning, such as suitable cleaning agents, cleaning duration, concentration of cleaning agents, pressure, pH levels, and cleaning temperature, they were thoroughly investigated. The purpose of this review is to enhance comprehension of the membrane cleaning procedure to optimize the efficiency of membrane separation processes.

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Contents

1. Introduction.....	2
2. NF Membrane Selectivity and Separation Fundamentals.....	3
2.1. Size sieving.....	3
2.2. Donnan exclusion.....	3
2.3. Dielectric exclusion.....	3
3. Fouling of NF membrane.....	4
4. Characteristics of NF membranes.....	5
5. Chemical agents and mechanism of operation.....	5
5.1. Alkaline.....	6
5.2. Oxidants.....	7
5.3. Acids.....	7
5.4. Surfactant.....	7
5.5. Others.....	8
6. Factors influencing the efficacy of chemical cleaning.....	8
6.1. Effects of concentration.....	8
6.2. Effects of pH.....	9
6.3. Effects of ionic strength.....	10
6.4. Effects of temperature.....	10
6.5. Effects of Cleaning Time.....	10
6.6. Effects of pressure.....	10
7. Evaluating the ecological impact of cleaning agents.....	10
8. Perspective and Conclusion.....	12
9. References.....	13

1. Introduction

NF is a membrane separation technique that operates under pressure and is positioned between ultrafiltration (UF) and reverse osmosis (RO) in terms of its filtration capabilities. The primary technique used to produce commercially accessible NF membranes is the interfacial polymerization process, which results in the formation of thin film composite (TFC) membranes. The membranes possess a unique structure consisting of a thin polyamide (PA) layer placed on top of a polysulfone (PS) ultrafiltration (UF) base membrane. The process of interfacial polymerization is crucial in determining the characteristics and performance of these membranes. As a consequence, the TFC architecture that is formed shows improved abilities for selective separation and filtering. Polysulfone UF is used as the primary material for the membrane, which offers a strong basis. The membrane's performance in accomplishing accurate separations and filtering processes in nanofiltration is enhanced by the ultra-thin polyamide skin layer. Depending on the pH of the solution, the membrane's surface charge can be either positive or negative due to PA's dissociable amine and carboxyl groups [1]. Consequently, size exclusion, Donnan effect, and dielectric exclusion effects are the major factors that regulate the solute separation process using NF membranes [2]. Due to its exceptional capacity for separating microscopic particles and its effective permeability to water, NF has found extensive use in the fields of water treatment and food processing, as shown by several studies. Nevertheless, the occurrence of membrane fouling is an unavoidable phenomenon over extended periods of operation. This phenomenon not only leads to a decrease in the permeate flow but also negatively affects the separation selectivity, hence imposing significant constraints on the widespread use of NF in industrial applications [3]. Reducing membrane fouling has been successfully achieved by feed pre-treatments, surface modification of membranes, and process optimization. Nevertheless, in order to avoid worsening and quickly regain membrane permeability, membrane cleaning continues to be the most effective method. The process of membrane cleaning is often categorized into two main

approaches: chemical and physical cleaning. The utilization of chemical cleaning is frequently considered the most practical and effective method for restoring membrane function. This preference arises from the potential risk of TFC membrane delamination caused by back-flushing operations and the ineffectiveness of forward flushing in eliminating interior fouling [4]. The primary classifications of chemical cleaning agents are Alkaline agents, acids, complexing agents, detergents (surfactants), solvents, and disinfectants. Table 1 provides a brief overview of some of the chemical components often found in commercial cleaning products.

The composite cleaning agents provide a synergistic enhancing effect via the combination of several chemical constituents. As a result, they are capable of efficiently eliminating complex foulants present on or inside membranes in the majority of instances. Although NF membranes exhibit excellent cleaning efficiency, the regular use of chemical cleaning methods may lead to both reversible and permanent alterations in the chemical and physical characteristics of these membranes [11]. Consequently, this can result in membrane breakdown and a decline in their ability to effectively separate substances. A comprehensive knowledge of the chemical-membrane interactions is imperative in order to grasp the potential detrimental effects that chemical cleansing may have on NF membranes, including the PA epidermis layer. It is imperative to examine these dynamics to evaluate the intricacies linked to the impacts of chemical cleansing on NF membranes. Nevertheless, the majority of the research has primarily concentrated on examining the impact of individual cleaning agents on membrane characteristics and performance [12,13]. Consequently, there is a dearth of comprehensive analyses that systematically summarize the procedures and the impact of chemical cleaning on the fouling of membranes. The importance of this topic becomes evident in the continuous effort to create cleaning solutions that are both efficient and resistant to chemicals, enhance cleaning methods, and advance the technology of chemically-resistant NF membranes.

Table 1
Major chemical constituents of cleansing agents.

Agent classification	Examples	Application	Negative health effects	Ref.
Acids	Phosphoric (H_3PO_4), acetic (CH_3COOH), citric ($C_6H_8O_7$), sulfuric (H_2SO_4), hydrochloric (HCl)	Dissolve inorganic substances	Strongly corrosive to all body tissue, especially eyes and skin	[5]
Alkaline agents	Silicates (SiO_4^{4-}), carbonates (CO_3^{2-}), hydroxide (OH ⁻), ammonia (NH ₃)	Dissolve fatty contaminants, disinfection, and prevent metal surface corrosion	Skin, eyes, and mucous membrane irritation	[6]
Complexing agents (water softeners)	Ethylenediaminetetraacetic acid (EDTA), Tripolyphosphates ($Na_3P_3O_{10}$)	Dissolving calcium and other cations; controlling the pH	Skin, eyes, and mucous membrane irritation	[7]
Detergents (surfactants)	Fatty acid salts (soap), Organic sulphonates (RSO-3)	Lower water surface tension	Intensive respiratory or gastrointestinal distress, ocular irritation, and potentially fatal coma	[8]
Solvents	Alcohols ($C_nH_{2n+1}OH$), glycol ethers	Dissolve organic substances	Irritation of the eyes, lungs, and skin, headaches, nausea, dizziness, or light-headedness	[9]
Disinfectants	Hypochlorite (HOCl), aldehydes (RCHO), quaternary ammonium compounds	Eliminate bacteria and other microorganisms	Irritating to the skin, eyes, and respiratory system	[10]

The study examines how cleaning chemicals impact the performance of polyamide NF membranes while also considering the potential for synergistic or inhibitory interactions resulting from these interactions. In addition, it provides an in-depth review of several categories of chemical cleaners. A comprehensive analysis of the objectives, tactics, mechanisms, and procedures related to membrane chemical cleaning is also included in this paper. Each one was carefully examined in addition to the extensive discussion of factors that affect chemical cleaning's effectiveness, including appropriate cleaning chemicals, cleaning times, concentrations of cleaning agents, pressure, pH levels, and temperatures. Moreover, it provides a comprehensive exploration of recent research that delves into the interaction between chemical cleaning agents and the resulting impacts they generate. The findings of this research are very significant in furthering the progress of creating efficient cleaning substances, improving cleaning procedures, and facilitating the enhancement of nanofiltration membranes that can withstand chemical impacts.

2. NF Membrane Selectivity and Separation Fundamentals

The ion-selective behavior that occurs during the separation of ions is determined not only by the micro-hydrodynamic features of the solution but also by the interactions that occur between the ions and the membrane. The ion selectivity at the nanoscale is mostly dependent on the interplay of many factors. The inherent free volume elements present in the polymeric networks of NF membranes, including both intermolecular and intramolecular gaps, can selectively permit the passage of some ions while effectively obstructing others. This phenomenon is attributed to the physical selectivity based on the size of the ions. Ion selectivity is achieved via the interactions between ions and the membrane, which include several factors such as dielectric properties, electrostatic forces, affinity, hydrophobicity, hydrophilicity, and coordination interactions. In this section, we provide a comprehensive overview of the many methods used by NF membranes to effectively differentiate ions. These mechanisms, primarily depicted in Fig. 1, encompass size sieving, the process of separating ions based on their size; Donnan exclusion, which occurs when the presence of charged species influences ion distribution; and dielectric exclusion, where the dielectric properties of the solution impact ion separation.

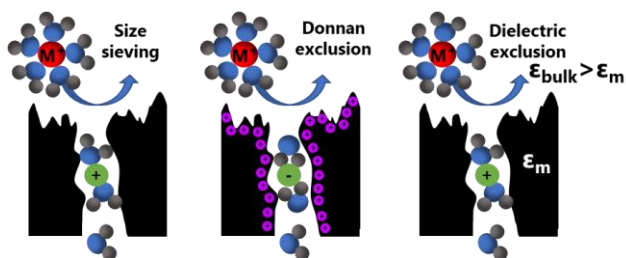


Fig. 1. Three Fundamental Mechanisms of the TFC NF Membrane.

2.1. Size sieving

The steric effects occurring when a hydrated ion comes into contact with a membrane pore, known as the size-sieving effect, constitute a critical step in species filtration separation technologies. This phenomenon is well-recognized in the field. The presence of solvation shells around hydrated ions in an aqueous solution allows for the size-selectivity of polymeric membranes. This selectivity is often accomplished by exploiting the disparity between the inherent effective pore size of the membrane and the size of the hydrated particles. Experiments have shown that altering or removing the hydration shell before transporting hydrated ions over the membrane pore barrier may dramatically lower the ions' effective size [14]. The process of ion dehydration becomes necessary in cases when ion transport occurs via a nanopore that has a diameter smaller than the dimensions of the solvation shell. Within the narrow space of the pore, charged groups like hydroxyl groups, carboxyl groups, sulfonic acid groups, and amino groups present on the pore walls can develop electrostatic contacts with dehydrated ions. This helps to reduce the energy loss caused by dehydration [15]. However, this phenomenon does not promote rapid ion mobility inside the pore. Ionic dehydration can additionally take place in cases when the size of the nanopore exceeds that of the solvation shell. This phenomenon arises due to the intrinsically low water permittivity present in the pore. The restricted environment causes water molecules to be less polarizable, which subsequently leads to a reduction in the solvation energy of ions. As a result, this series of events precipitate a decline in the ions' capacity to hydrate [16]. It is a well-known fact that confinement effects can significantly impact ion transport by causing low dielectric constants in polyamide membranes. The

hydration or dehydration behavior of ions is closely linked to their valence, as shown by the larger hydrated size of high-valent ions and the higher energy barrier associated with their dehydration compared to low-valent ions. By taking into account the different sizes of hydrated ions, it is possible to design polymeric membranes with precise pore diameters. This allows for efficient differentiation between ions with a single charge and ions with multiple charges.

2.2. Donnan exclusion

The Donnan effect was first suggested in 1911 and has since become a widely accepted classical theory for explaining the distribution of ions on either side of a semipermeable membrane [17]. The objective of developing the Donnan exclusion mechanism is to enhance our understanding of the ion-selective behaviors that occur in electrically powered ion-exchange membranes and pressure-driven NF membranes. This expansion largely focused on studying the dynamic interaction between charges and the membrane, revealing the complex processes that affect ion selectivity in various membrane-driven situations [18]. The selectivity of polymeric membranes that include ionizable charged groups, such as $-\text{SO}_3^-$, $-\text{COO}^-$, and $-\text{NH}_4^+$, is attributed to the electrostatic interactions between the membrane surface and ions. The Donnan potential, also known as the surface electric potential, is generated by dissociated charged groups inside the polymer network, as postulated by the classic electric double-layer theory [19]. This causes the co-ions to interact electrostatically with the charged polymer, which often results in Donnan exclusion of the co-ions from the side of the membrane [20]. The surface potential is of great importance in influencing the distribution of ion concentrations in the double electric layer [21]. Additionally, it functions in the regulation of ion distribution across the membrane, thus exerting control over the internal concentration of ions and impacting their subsequent passage through the apertures of the membrane.

2.3. Dielectric exclusion

Dielectric exclusion is a popular way to describe the phenomena when charged solutes interact with the membrane matrix in a repellent way. This repulsion occurs due to the presence of ions and the image charges they induce at the interface. The origin of this phenomenon lies in the discrepancy in dielectric constants between the polymeric membrane matrix (ϵ_M), with a value of less than 10, and the aqueous solution (ϵ_S), which has a value of approximately 80 [22]. The presence of a wide range of dielectric constants in the membrane matrix leads to the creation of polarization charges [23]. This is a result of the electrostatic field generated by the ions, resulting in electrostatic repulsion between the bound polarisation charges and the ions. The phenomenon of polarized interaction, which arises from disparities in dielectric characteristics, can be alternatively understood as image forces [24]. Drawing from the explanation provided above, one can deduce that the dielectric exclusion effect encountered by ions is not dependent on the polarity of their charge, but rather on the value of their valence. Consequently, both positively and negatively charged ions are impeded. Hence, the interaction serves as a supplementary mechanism for enhancing the rejection efficiency of NF by mitigating the adverse effects of excessive ion repulsion.

The ion transfer behavior is influenced not only by the ionic exclusion resulting from dielectric exclusion at the interface between the polymeric matrix and solvent but also by the alteration of the solvent's dielectric constant inside a confined region [25]. Numerous investigations and theoretical simulations have provided evidence that the dielectric properties of the solvent undergo substantial fluctuations upon its entry into the membrane pore (ϵ_P) from the bulk solution (ϵ_B) [26]. These fluctuations arise from the conformational changes experienced by solvent clusters under the nanoconfined conditions. In the case of inserting a water molecule into a polyamide nanopore channel with a diameter of 1 nm, it was shown that only a small number of water molecules were able to pass through the nanopore channel, given the water molecule size of 0.276 nm [27]. Hence, the structural arrangement of water molecules inside nanoconfined spaces exhibits a higher degree of organization compared to that in bulk solutions. As a consequence of this phenomenon, the dielectric constant of water shows a notable decrease when it is constrained to the nanoscale range. The reduction in solvent permittivity has a notable impact on the solvation energy of ions and the ionization behavior of charged groups. As a result, it modifies the transport of ions and the selectivity behavior inside the pores. The investigation of property alterations in water and ions within a nanoconfined environment is crucial due to the obvious effect of unusual shifts in dielectric characteristics on the ion selectivity. This research endeavor aims to enhance our comprehension of the fundamental mechanism underlying ion selectivity. Nevertheless, the study of these features remains difficult owing to the absence of sophisticated characterization methods capable of giving details at the sub-nanometer range.

Due to the combined impacts of the two processes, the polymeric membrane exhibits remarkable ion selectivity when ions pass through its nanopores, which are limited in size due to pore size constraints and affected by electrochemical interactions [28]. One notable example is the synergistic impact resulting from the combined effects of three types of exclusion: dielectric, size-based, and Donnan. This combination often confers a greater repulsion of bivalent ions compared to univalent ions in polyamide NF membranes [29]. Nevertheless, due to the intricate interplay and competing nature of several pathways, it presents a challenge to succinctly isolate and manipulate a solitary element in order to showcase its specific role in ion selectivity. Improving ion selectivity necessitates careful optimization in both the choice of ion-selective membrane materials and the membrane fabrication process, therefore maximizing the synergistic impact of these processes. To construct high-performance ion-selective membranes, it is crucial to focus on the selection of multi-parameters tunable materials and the development of diverse membrane production techniques. These measures are necessary to effectively boost the underlying processes involved in membrane performance.

3. Fouling of NF membrane

The physicochemical interactions between the feed solution and membrane surface have been demonstrated to influence the permeability to water and the rejection of solutes, which are affected by membrane fouling and concentration polarization [30]. Membrane fouling is a phenomenon that can be attributed to many causes, including cake and gel development, adsorption, pore constriction, and obstruction [31]. The elimination of reversible fouling, such as the production of cake and gel, can be achieved by the use of physical cleaning techniques [32]. Irreversible fouling, such as adsorption and pore blockage, can only be removed by the use of chemical cleaning methods [33]. The phenomenon of reduced water flow can be attributed to pore obstruction, whereby the presence of particles leads to the blockage of pore openings. Furthermore, as little particles infiltrate the pores, the pores undergo a contraction. Under optimal hydrodynamic circumstances, the particles exhibit diffusion towards the walls of the pore or rapidly migrate to that location. The prevention of tiny particles from infiltrating the pores leads to an enhancement in water flux due to the reduction in pore constriction. However, the deposition of big particles on the surface of the membrane and subsequent formation of a filter cake results in the cake layer serving as a hydraulic resistance, impeding water transport and causing a reduction in flow [34].

Fouling phenomena can occur in membrane filtration systems, depending on the nature of the foulant species present in the raw water and on the membrane surface. These foulant species may include colloidal, organic, inorganic, biological, and particle contaminants [35]. The types of foulants are shown in Fig. 2 along with illustrative examples of each category. Colloids are defined by some sources as particles of tiny dimensions, often ranging from 1 nm to 1 μm [36]. However, other sources use a more inclusive approach by extending the definition to include particles with larger sizes. Due to the size range of colloids, they are inherently susceptible to membrane fouling. By means of molecular diffusion, particles with a lower molecular size may easily spread out from the surface of the membrane. In contrast, bigger particles may be eliminated by shear-induced diffusion or lateral migration. Some examples of colloids found in aquatic environments include colloidal silica, iron oxides, manganese oxides, organic colloids, suspended matter, clay minerals, calcium carbonate precipitates, as well as microorganisms like bacteria and viruses [37]. In the context of membrane surface water treatment and wastewater reclamation, the primary substances that cause fouling are organic macromolecules, including polysaccharides, proteins, and natural organic compounds [38]. The aforementioned macromolecules exhibit colloidal properties and have several resemblances with their inorganic counterparts concerning membrane fouling. Moreover, microorganism cells and cell detritus can be classified as bio-colloids [39].

Organic fouling is caused by the deposition of non-biological organic contaminants such as humic acid, sugar proteins, oil, and cationic surfactants [40]. The susceptibility of the NF membrane to organic fouling arises from the presence of organic contaminants, such as natural organic matter (NOM), pesticides, polysaccharides, and polycyclic aromatic hydrocarbons, in water sources. The presence of organic debris can lead to two possible outcomes: deposition on the pores of the membrane or adsorption onto the surface of the membrane [41]. These processes contribute to the occurrence of organic fouling and subsequently reduce the flow of the membrane. The formation process is influenced by several elements, including the chemical composition, interaction with membrane components, and organic structure. Foulants are more difficult to develop when organic elements are present.

Scaling, also known as inorganic fouling, refers to the process of in situ formation of hard sediments by the action of inorganic chemicals. Inorganic fouling can inflict physical damage on the membrane [42]. The prevalent

fouling substances include silicon dioxide (SiO_2), barium sulfate (BaSO_4), strontium sulfate (SrSO_4), calcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$), iron hydroxide ($\text{Fe}(\text{OH})_3$), and aluminum hydroxide ($\text{Al}(\text{OH})_3$). Another frequent inorganic fouling on NF membranes is heavy metals [43]. The impact of heavy metals on membrane fouling can vary from that of conventional metals due to their dual influence on both membrane damage and substantial alteration of sludge properties. To decrease the flow, inorganic foulants that need nucleation and crystallization can either obstruct the surface or create a filter cake [44]. The formation of a permeable layer on the surface of the membrane, which gradually increases in thickness and ultimately obstructs the surface owing to the development of crystals in a lateral manner, leads to the production of a filter cake. To reduce inorganic fouling, scale inhibitors are often used to slow the nucleation rate [45].

The term "biological fouling" encompasses the presence of bioactive organisms that contribute to fouling. Biological fouling is active, whereas organic and inorganic fouling are deposited passively [46]. On the membrane's surface, bacterial colonies grow and mix with glycocalyx. Once the colonies reach a particular size, the permeability of the membranes starts to drop [47]. Biofouling can be reduced by enabling fungicides access to biofilm cells, where they can damage the glycocalyx. To remove the glycocalyx, it is necessary to use detergents or compounds that possess chelating properties toward metals [48]. Since oxidants are ineffective in this process and certain scale inhibitors provide growth substrates for bacteria, they should be avoided [49]. Frequent replacement of detergent is necessary in order to mitigate the development of microbiological resistance during chemical cleaning procedures.

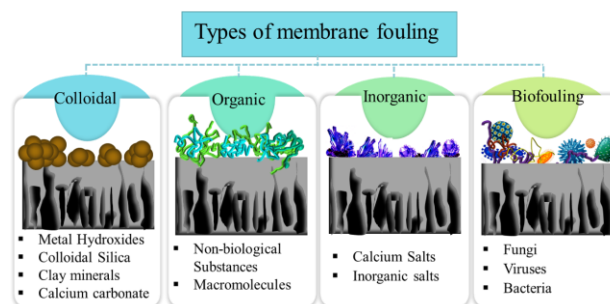


Fig. 2. Examples of many types of foulants.

The fouling rate is influenced by several aspects, including the composition and concentration of solvents and solutes, the properties of membrane substances and surfaces, the distribution and dimensions of pores, and the hydrodynamics of the membrane modules [50-55]. The phenomenon of fouling has been often noted to exhibit greater severity in environments characterized by low pH or high ionic strength during the process of NF involving macromolecules, including disaccharides, nucleic acids, polysaccharides, proteins, starch, lipids, cellulose and NOM [56-58]. This can be described by the Derjaguin-Landau-Verwey-Overbeek (DLVO) hypothesis with regard to particle suspension stability. The decrease in pH levels results in a decrease in the deprotonation of acidic chemical groups like $-\text{COOH}$ and $-\text{SO}_3\text{H}$ present in macromolecules, including lipids, proteins, carbohydrates, and HAs. By reducing the electrostatic repulsion between molecules, the barriers to their interaction are decreased. Anticipated, this decrease in repulsion will facilitate the development of a gel—a three-dimensional arrangement of interrelated molecules. Nevertheless, this promotion of gel formation has a possible drawback, since it is also linked to a higher probability of fouling [59,60]. An elevated ionic strength has a compressive effect on the electrical double layer (EDL) by reducing the spatial extent of oppositely charged ions. As a result of this occurrence, the energy barrier of repulsion that was previously present between particles is removed, making it possible for attractive Van der Waals interactions to exert their preponderant impact. Consequently, colloidal instability is induced, leading to heightened aggregation and the formation of more substantial fouling cakes. Increased ionic strength has the potential to reduce the hydrodynamic radius of molecules, such as HA, hence facilitating their passage into pores and leading to enhanced pore adsorption [61].

Protein fouling is known to be particularly severe at pH values close to the molecules' isoelectric points and on water-repellent membranes [62]. At the point of isoelectricity, proteins exhibit enhanced hydrophobicity due to a reduction in electrostatic repulsion. Consequently, the hydrophobic interactions among additional contaminants and the membrane, as well as among the contaminants themselves, become heightened [63]. The hydrophobic interaction may become less prevalent when the pH is raised or the ionic strength is lowered, leading to an increase in electrostatic repulsion.

The occurrence of membrane fouling is often intensified when divalent cations, such as Fe^{2+} , Ca^{2+} , Mg^{2+} , and Zn^{2+} , which are often found in surface water like lakes, rivers, and oceans, are present. This phenomenon has been extensively documented in scholarly publications [64–66]. Ca^{2+} 's capacity for interaction with substances that cause fouling (such as NOM) and membrane surfaces, resulting in a shift of surface chemistry in either scenario, may be responsible for its detrimental influence on permeate flow [67]. Complexes can be formed between the negatively charged carboxylate groups and the calcium ion, which involves the deprotonation of carboxylic groups found in macromolecules. This interaction leads to a partial shielding of the charges on these macromolecules, especially when the pH level is neutral. Due to its divalent nature, Ca^{2+} can facilitate the connection between two functional groups bearing negative charges. Inside the domain of molecular interactions, complexation occurs intramolecularly when distinct groups coexist inside a single molecule. This phenomenon arises when both of these groups, likely referring to certain functional groups or atoms, are present inside the same molecular entity. The intramolecular complexation results in the conversion of the molecule HA into a condensed and tightly wound structure. The complicated and folded organization of the molecule is formed as a result of the internal interactions between the relevant groups. This arrangement might have important consequences for the molecule's behavior, stability, or functioning in different chemical and biological situations. [68]. In other cases, this bridging effect occurs when two molecules join together [69]. As a result, the gel layer composed of macromolecules undergoes increased compaction and cohesion due to the cross-linking influence of Ca^{2+} [70]. Most NF membranes on the market today are constructed from thermoplastic polymers like cellulose acetate (CA), polyethylene (PE), polyether sulfone (PES), Polypropylene (PP), Polyvinyl chloride (PVC), Polystyrene (PS), Polyethylene terephthalate (PET), poly(methyl methacrylate) (PMMA), polyacrylonitrile (PAN) or polyvinylidene fluoride (PVDF) [71]. Every one of these polymers exhibits distinct characteristics in terms of surface charge, water-repellency, chemical and heat resistance, mechanical robustness, flexibility, and other relevant features [72,73]. Both these features and the processes that cause fouling and cleaning can have some influence on each other. The following section provides an in-depth overview of a set of NF membrane attributes that may aid in the selection of a more suitable detergent for achieving optimal stability.

4. Characteristics of NF membranes

The mechanical durability, heat resistance, and resistance to chemicals of polymeric NF membranes are contingent upon the specific material used in their production. Nevertheless, while a membrane may be stable against one performance, it doesn't guarantee it will perform optimally against all others. Table 2 provides a comprehensive listing of the mechanical, thermal, and chemical characteristics associated with these polymeric materials.

CA membranes possess inherent hydrophilicity and exhibit little protein binding [74]. However, it is important to note that these membranes have a restricted operating pH range. A decrease in pH may lead to the degradation of the β -glucosidic linkages present in the cellulose polymer's backbone. This degradation process results in a reduction of the polymer's effective molecular weight and, ultimately, results in the degradation of its structural stability. At elevated pH levels, the membranes of CA will undergo deacetylation [75]. Since chlorine is a powerful oxidizing agent, it poses a greater danger of oxidation and damage to the membrane if its concentration goes over 1 ppm over the long run or 50 ppm over the short term [76].

Ethanol, methanol, acetone, and dimethyl sulfoxide are just a few of the organic solvents to which PAN membranes exhibit high resistance, making them well-suited for the remediation of industrial wastewater [77]. PES and PS membranes' phenylene ring-sulfonyl (SO_2) group-connected architectures provide them with superior stiffness and chemical resistance. The membranes have a broad operating range, with temperatures capable of reaching up to 70 °C and an operational pH that spans from 1 to 12 [78]. The membranes exhibit significant resistance to alkaline compounds; yet, it is important to acknowledge that PS and PES membranes are vulnerable to oxidizing agents such as chlorine, potassium Permanganate, hydrogen peroxide (H_2O_2), or ozone. [79]. For short-term uses, the maximum permitted chlorine concentration is 190 ppm, while for long-term utilizations, it is 40 ppm [80].

PVDF membranes have superior stability compared to PES and polystyrene (PS) membranes. These materials often exhibit exceptional chemical resistance and can undergo several autoclaving cycles, making them well-suited for use in the food and pharmaceutical industries [81]. Nevertheless, it has been shown that in alkaline streams characterized by a pH above 12, PVDF can undergo dehydrofluorination [82].

Polymeric additives, such as polyvinylpyrrolidone (PVP), polyvinyl alcohol (PVA), and polyethylene glycol (PEG), are often mixed with or grafted

onto the polymeric backbone matrix because the increased hydrophilicity of a membrane is beneficial in many processes [83]. The basic characteristics of the membrane are greatly influenced by the chemical makeup of the parent polymer, even in the case of minor modifications. The picture is further complicated by the inclusion of polymeric additives, which may improve or change the properties of the membrane. Furthermore, the structural and functional characteristics of the membrane are greatly influenced by the particular methods used during the casting process, including temperature and pressure settings. Any further post-production changes made to the membrane may also be very important. When combined, these factors may result in significant variations in the membrane's mechanical and chemical stability, highlighting the need for accuracy and control in the production process to provide the appropriate membrane performance for a range of applications [84]. Therefore, it is advisable to get comprehensive data on the operating limitations of various membranes directly from the manufacturer.

The cleaning processes should adhere to the prescribed limitations of these qualities. In practical use, it is necessary to conduct frequent monitoring of membranes in order to verify their integrity. Nevertheless, an overabundance of cleaning might expedite the deterioration or harm of the membrane structure, leading to an increase in pore sizes, cracked surfaces, or compromised and perhaps broken fibers [85]. This suggests that over time, the membrane experiences a steady decline in its usefulness as a result of the cleaning process. Observable signs such as a decrease in the number and quality of the filtrate products indicate the declining usefulness. The membrane is stressed by the frequent cleaning processes, which might result in mechanical issues, especially the embrittlement of the fibers that make up the membrane's structure. Overall, this means that over the membrane's working life, its performance is negatively impacted, which shows up as reduced filtering efficiency and structural integrity [86]. There are several causes of membrane integrity failure in a membrane factory. The build-up of air in the system, for instance, might generate shocks, which in turn can cause physical damage [87]. One crucial aspect to consider is that the interaction between chemicals and membranes during the cleaning process may lead to the degradation of membrane materials. This degradation occurs as a result of chemical modifications affecting the functional groups within the polymeric chains [88].

Table 2

Resistances of commercial NF membrane materials to mechanical, thermal, and chemical stress.

Polymeric NF membrane	Mechanical strength	Thermal limit (°C)	pH	Oxidant tolerance	Ultrasonic irradiation	Ref.
CA	Excellent	30	4-8	Average	Average	[89]
PAN	Excellent	40	2-11	Average	Average	[90]
PES	Excellent	78	2-11	Excellent	Below average	[91]
PS	Excellent	75	1-13	Excellent	Excellent	[92]
PVDF	Excellent	40	2-10.5	Superior	Excellent	[93]

5. Chemical agents and mechanism of operation

There are five stages involved in the process of membrane chemical washing, which can be regarded as a technique, as shown in Fig. 3. Initially, the cleaning chemical is conveyed to the surface of the membrane. Subsequently, the cleaning agent undergoes a process of transiting through layers of foulant in order to reach the surface of the membrane. Afterward, the cleaning processes facilitate the solubilization and detachment of foulants. Following that, the waste cleansing agent containing suspended foulants is transferred to the interface. Lastly, the transfer of waste material from the membrane's retentate side to the bulk solution.

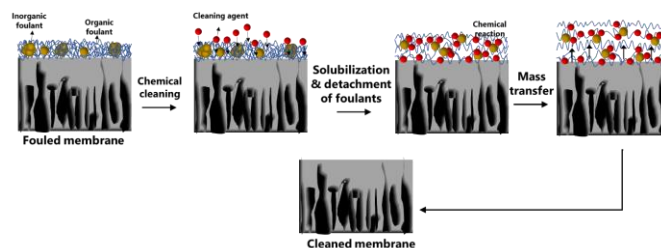


Fig. 3. Conceptual model of electrostatic equilibrium for cleaning NF membranes [94,95].

Liu et al. [96] introduced a widely acknowledged conceptual framework for the phenomena of cleaning and fouling, which establishes a connection between the primary forces responsible for fouling and cleaning. The interaction between the foulant and membrane is mostly driven by hydrophobic attraction, whereas the primary process for cleaning is generally based on electrostatic repulsion [97]. The selection of cleaning chemicals can be facilitated by a comprehension of the chemical properties of foulants. This is because the impact of chemicals on fouling is widely recognized. For instance, fouling caused by potable waters tends to be more pronounced when foulants have higher molecular weights and charge ratios [98]. Additionally, the presence of divalent cations in the water and the hydrophobicity of the membrane also contribute to fouling [99]. The enhancement of the electrostatic potential of the cleaning medium, achieved by manipulating charge density, polarity, or pH, effectively diminishes the attractive forces and hence enhances the effectiveness of the cleaning process [100].

Cleaning is regularly performed using either a clean-in-place (CIP) or a chemically enhanced backflush (CEB) method. It focuses on the procedure for introducing a chemical reagent into the permeate region of a membrane, with a particular emphasis on two membrane configurations: out-in hollow fiber (HF) and in-out capillary tube (CT). When considering an in-out capillary tube, the introduction of the chemical reagent occurs through the lumens (interior channels) of the tube. In the case of the out-in hollow fiber membrane, the module shell functions as the pathway through which the chemical reagent is introduced into the permeate side [101]. In the context of a CEB process, the introduction of chemicals occurs via a regular backflush procedure. Scheduled on a daily or weekly basis automatically with no human interaction, CEB is used to stabilize/maintain the permeability by reducing foulant build-up (and is commonly referred to as "maintenance" cleaning). In contrast to CEBs, CIPs aim to restore flux by using longer soak times, higher cleaning agent concentrations, and often ambient application temperatures. A CEB is characterized by a moderate application temperature, a brief soak duration, and a low reagent concentration. According to the comparison, CEB is more concerned with efficiency, cleaning more quickly at moderate temperatures with lower chemical concentrations, as opposed to CIP's more thorough and time-consuming cleaning method, the main goal of which is to regain membrane flow. During an unexpected CIP process, multiple cleaning sessions using wide-ranging reagents (commonly alkaline, reductive, and acidic) may be required to achieve the restoration of optimal permeability. Table 3 provides a comprehensive list of general chemical cleaning agents together with their typical interactions.

The degree of pre-treatment, backwash cycle duration, duration of the chemical cleaning cycle, washing temperature, and, most importantly, planned flow rate all have a role in the cleaning performance of the full-scale potable water NF systems [108,109]. It is now widely acknowledged that the lower washing period, improved membrane efficiency, and longer membrane life more than balance the benefit of capital costs given by higher-flux processes. In the following sections, all of the factors are considered via a review of current research studies.

5.1. Alkaline

Through a process of electrostatic repulsion, alkaline cleaning chemicals are vital in reducing organic fouling. As part of this procedure, the fouling layer solutes and the membrane are subjected to an electric charge enhancement. Repulsion between negatively charged things is caused by the electrostatic barrier that is created by the alkaline composition of the cleaning chemical. The organic fouling is efficiently removed from the membrane surface by this repulsive force, which stops it from adhering and building up. The cleaning method is more successful in eliminating organic fouling in membrane systems because the alkaline cleaning chemical augments negative charges [110]. Additionally, these agents may induce membrane pore expansion, protein hydrolysis, and foulant dissolution [111]. Moreover, alkaline cleaning agents have the capability to solubilize silicon salts that exhibit insolubility in acidic solutions. The prevailing consensus is that the polyamide nanofiltration (PA-

NF) membrane exhibits notable resilience to alkaline conditions. Furthermore, it is observed that the functional groups present on the epidermal layer of the membrane remain unaltered after alkaline cleaning procedures conducted at ambient temperatures [112]. The study conducted by Wadekar and colleagues revealed a reduction in the retention of divalent cations after alkaline washing. This drop was attributed to membrane swelling, with the extent of swelling being influenced by the carboxyl content present in the epidermal layer [113]. According to the findings of Kallioinen et al., alkaline washing had a clear impact on increasing permeability and decreasing glucose/magnesium sulfate rejections. Furthermore, it was noted that extending the duration of the cleaning process would result in further alterations in permeability and rejection [114]. Additionally, it was discovered that the expansion of membrane pores resulting from alkaline cleaning exhibited reversibility, with the membrane pore dimensions gradually returning to their original state during regular filtering operations. Nevertheless, in the event that the foulants infiltrate the engorged pores of the membrane during the filtering process, the following reduction in pore size might lead to significant and permanent fouling of the membrane. Hence, it is recommended to do acid cleaning subsequent to alkaline cleaning in order to expedite the restoration of the pore morphology inside the PA coat, hence preventing pore blockage.

The functional groups like carboxyl, amine, amide, and hydroxyl groups on the PA layer NF membrane may potentially ionize as a result of alkaline washing, which might impact the NF membrane's chargeability. Bai et al. observed that the charge on the NF270 membrane's surface exhibited a decrease, indicating a more negative charge, following alkaline cleaning [115]. Conversely, Simon et al. reported that the NF270 membrane's electrical charge distribution remained unaltered, while its hydrophobicity increased after cleaning. However, they also noted an increase in permeability and a decrease in solute retention in their specific case [116]. There is a possibility that the amount of undissociated carboxyl groups in PA affects how alkaline washing affects the surface charge of the material [117]. In cases where the isoelectric point of the NF membrane is low, indicating that the functional groups, particularly the carboxyl groups, are predominantly dissociated, washing with an alkaline solution does not alter the surface charge distribution [118]. In theory, the use of alkaline cleaning methods might potentially result in the ionization of carboxyl groups, hence leading to an improvement of the membrane's hydrophilic characteristics. Nevertheless, a consensus among the researchers about the impact of alkaline washing on the PA NF membrane's hydrophilicity and surface charge was not achieved, indicating the need for more investigations. Based on the preceding discourse, it is evident that although there is an observable reversible alteration in the physical structure of the membrane, namely the dissociation of carboxyl groups and enlargement of pores, the conventional alkaline cleaning process does not result in the rupture of chemical bonds inside the polyamide (PA) layer. This explains the common commercial use of alkaline cleaning solutions by industries.

Solutions of caustic soda, also known as NaOH, are primarily used at pH levels ranging from 11 to 12 [119]. However, in situations where membrane chemical resistance is a concern, such as with polyvinylidene difluoride (PVDF), the pH levels may be adjusted to a lower value [120]. Hydroxide ions facilitate the dissolution of organic matter that exhibits mild acidity, particularly those containing carboxylic and phenolic functional groups [121]. Additionally, hydroxide ions stimulate the breakdown of polysaccharides and proteins, leading to the formation of smaller sugar molecules and amides. The presence of hydroxide ions facilitates the expansion of NOM molecules, hence promoting improved general movement of the washing solution in the direction of the membrane's surface [122]. The use of this method may also demonstrate efficacy in the elimination of mineral colloids and silicon compounds, whereby the electrostatic repulsion and solubility are enhanced by charge effects and ionic strength. The recovery of permeability generally exhibits an upward trend as the concentration of NaOH increases, reaching a threshold value that varies depending on the specific foulants and membrane materials, as well as the application and extent of fouling [123].

Table 3
Effects of cleaning solutions and interactions on foulants.

Cleaning agent	Examples	Overall capabilities	Ref.
Acids	Citric Acid (C ₆ H ₈ O ₇), HCl, Sulphuric Acid (H ₂ SO ₄), Nitric Acid (HNO ₃), H ₃ PO ₄ ,	Acidic hydrolysis of certain macromolecules, pH regulation, and the dissolving of inorganic precipitates	[102]
Alkalis	Potassium or Sodium Hydroxide (KOH, NaOH), Ammonia, Sodium carbonate (Na ₂ CO ₃), Trisodium Phosphate	The ability to control pH, modify surface charges, catalyze the alkaline breakdown of proteins, and soapify lipids.	[103]
Oxidants	HOCl, H ₂ O ₂	Disinfection; oxidation of organic compounds	[104]
Surfactants	Sodium dodecyl sulfate (SDS), Cetyltrimethylammonium bromide (CTAB), Polysorbate 20 (Tween 20), Dodecyltrimethylammonium chloride (DTAC)	Coating dispersion and suspension	[105]
Chelants	EDTA, DTPA, Nitrioltriacetic Acid	Metal-complexion, mineral-deposit removal	[106]
Enzymes	Lipases, Proteases, Amylase, Cellulase	Catalyzing the decomposition of certain substrates, such as proteins and lipids.	[107]

5.2. Oxidants

Sodium hypochlorite (NaOCl) and H_2O_2 are two examples of oxidants. Nevertheless, the use of HOCl is not globally widespread due to two main reasons [124]. Firstly, some polypropylene (PP), PS and CA membranes are not resistant to chlorine, as stated in reference [125]. Secondly, the usage of reagents with chlorine may result in the generation of chlorinated organics, which can have adverse effects on both human health and the environment, hence imposing restrictions on their usage [126]. The process of oxidation leads to the degradation of functional groups in NOM into carboxyl, ketonic, and aldehyde groups [127]. This transformation renders them more prone to hydrolysis, particularly under high pH conditions. The empirical evidence suggests that the use of oxidant cleaning agents in conjunction with alkaline cleaning agents is more efficacious compared to the use of oxidant cleaning agents alone, particularly in situations where organic foulants are prevalent [128].

A comparative investigation was conducted to assess the effectiveness of NaOCl and alkaline H_2O_2 in cleaning PES UF membranes contaminated with NOM. The results indicated that NaOCl exhibited superior performance in eliminating organic matter by oxidizing aromatic humic compounds at higher pH values [129]. The repetition of this process was not seen for peroxide. It is well-known that one common mechanism for the degradation of organic colloids is the halogenation of aromatic rings [130]. Moreover, Brodfuehrer et al. [131] indicated that the hypochlorite's oxidative qualities might be more important than the hydroxide's catalytic role in hydrolysis. The study carried out by Liu et al. [132] suggests the utilization of caustic facilitates the formation of a fouling layer that is more permeable, hence enabling chlorine to effectively reach the surface of the membrane. This observation may provide a plausible explanation for the enhanced effectiveness of HOCl at elevated pH values. The study conducted by Huang et al. [133] demonstrated the impact of free chlorine on the oxidation process of organic colloids, with the diffusion of these colloids from the membrane surface being regulated by the duration of soaking. Some research suggests that chlorine aids the transport of the detergent to the foulant on the membrane surface by causing the membrane to expand [134].

Since high concentrations of HOCl are undesired due to expense and membrane integrity loss, it is evident that determining the ideal NaOH concentration and HOCl for cleaning membranes is useful. The C-S bond chlorination in PES has been reported at concentrations over 150 mg L⁻¹ and pH levels below neutral, while PVDF membranes' alkaline tolerance is restricted to pH levels below 11 [135]. The conclusion drawn from a current examination of plants in the UK is that cleaning effectiveness cannot be determined based merely on the foulant's, cleaner's, and membrane's chemical composition because of the difference in permeability recovery from these investigations [136].

5.3. Acids

Acid cleaning is performed to get rid of hardness salts and metal hydroxides, both of which are multivalent cationic species. Mineral acids, such as HCl, H_2SO_4 , oxalic acid ($C_2H_2O_4$), $C_6H_8O_7$, and H_3PO_4 are often used in many applications because of their affordability and efficacy in both CEB and CIP processes [137]. These acids may effectively operate at pH values as low as 1.0 for PES and PVDF materials [138]. However, it is important to note that using pH levels below this threshold may lead to potential integrity issues. In addition to facilitating acid hydrolysis, acids have a modest oxidative effect on NOM, resulting in the formation of acids and soluble aromatic aldehydes at specific groups within NOM [139]. However, their primary use is often focused on the removal of mineral scaling.

Although foulants may be solubilized by powerful mineral acids, the use of organic acids, such as oxalic and citric acids, proves to be more efficient in terms of generating and transporting organometallic foulants, like tributyltin, from the PA layer of the membrane to the substrate [140]. Citric acid exhibits buffering properties and has strong binding capabilities, making it a highly efficient and user-friendly alternative to mineral acids (H_2SO_4 , HCl, HNO_3) with a reduced likelihood of causing pH-related harm [141]. It is said to be the most popular chemical cleanser for NF membranes, along with NaOCl [142]. Citric acid can interact with the production of biofilms by eliminating minerals from foulant layers and interfering with the enzymes that enable the biofilms to form. Iron quickly forms complexes; however, the restricted solubility of these complexes hinders their transportation away from the surface of the membrane [143]. The efficacy of combining citric and oxalic acid has been demonstrated in practical applications, wherein the formation of multiple organo-metallic complex species has been observed [144]. This finding aligns with previous studies on cleaning effectiveness, which have shown that using a mixture of cleaning solutions in applications that include many stages leads to improved effectiveness and increased permeability recovery, even in cases where foulant removal is not fully achieved.

H_3PO_4 acid is a chelating ligand that has moderate efficacy in removing inorganic scale [145]. However, similar to other chelates, it demonstrates limited effectiveness in removing colloidal silicates and elemental sulfur. Similar to citric acid, the buffering capacity and sequestering properties of this substance render it potentially advantageous for regular mechanized cleaning processes or even in the context of Clean-in-Place (CIP) systems, where the likelihood of excessive dosage and subsequent harm is reduced [146]. This method is usually reserved for CA-RO membranes that have been polluted with metal oxides and a small number of proprietary industrial membranes, with its usage being mentioned in only a few NF cleaning publications [147,148]. It has comparatively lower efficacy in comparison to other cleaning agents when used against NOM.

This interfacial polymerization of monomers determines the resistance against acidic environments with regard to the PA NF membrane. The hydrolysis process of the PIP-TMC NF membrane has a lower energy barrier, resulting in its occurrence at pH levels below 2 [149]. Consequently, the PIP-TMC membrane shows a comparatively lesser resistance to acidic conditions when compared to the membrane composed of m-phenylenediamine and trimesoyl chloride (MPD-TMC). [150]. According to Jun et al., it was said that this NF membrane exhibited a favorable ability to withstand acids. It was observed that at a pH of 0, the skin layer of the membrane experienced compaction mostly as a result of hydrogen bond distortion [151]. This compaction subsequently led to a drop-in flux. The PIP-TMC membrane underwent hydrolysis at a pH of 0.25, leading to an augmentation of the surface's negative charge and an increase in permeate flow. Consequently, the skin layer gradually detached [152]. Pre-treatment of the PIP-TMC NF membrane with highly reactive acid, as described by both Tanninen et al. [153] and He et al. [154], increased pore size and decreased salt rejection. Thankfully, the PA membrane's acid resistance in moderate acid environments is satisfactory, meaning that acidic cleaning solutions are accessible for membrane cleaning. According to Al-Amoudi et al., the pH 3 cleaning agent had little impact on the permeate flow [155]. After cleaning the PA NF membrane with diluted HCl (pH 2), Simon et al. discovered that the membrane separation performance and surface charge characteristics were almost constant [156]. Hence, it can be inferred that the presence of strong acids has a detrimental effect on the mechanical integrity of the top layer of the membrane, leading to a decline in membrane separation efficiency. Upon comparing the MPD-TMC NF membrane with the PIP-TMC NF membrane, it is clear that the former demonstrates increased resistance to the harmful effects of strong acids. This suggests that the MPD-TMC membrane is better able to preserve its structural integrity and functioning when exposed to severe circumstances including very acidic environments. The exceptional resilience highlights the capacity of the MPD-TMC NF membrane to withstand and function efficiently in environments where exposure to corrosive strong acids is a major factor, hence strengthening its dependability and durability in real-world applications. To mitigate the adverse impact of acidic solvents on the NF membrane, it is essential to monitor and maintain the pH of the cleaning solution at a level beyond a certain threshold, such as 3.

5.4. Surfactant

Organic macromolecules known as surfactants are often used in polymeric membrane systems as detergents [157]. The efficacy of surface-active agents is attributed to the phenomenon of electrostatic repulsion or lipophilic/hydrophilic interactions [158]. Therefore, it may be inferred that the use of surfactant cleaning agents may result in a lower degree of damage to membrane integrity as compared to the use of traditional acidic or caustic agents. Furthermore, some surfactants' biodegradability is beneficial for the environment. Membrane properties (such as roughness, electrical charge at the surface, and hydrophobicity), foulant properties, and the physical/chemical interaction between foulants and membranes all have a role in determining the most effective surfactant [159]. To effectively separate oil at the membrane surface, it is essential for the surfactant to possess the capacity to diffuse into the fouling layer and dislodge the oil from the membrane surface. In addition, the measurement of cleaning effectiveness is contingent upon many essential factors, including cleaning duration, surfactant concentration, temperature, and pH of the solution [160].

According to Zhao et al. who evaluated the effectiveness of DTAC, $C_6H_8O_7$, EDTA-4Na, Sodium dodecylbenzene sulphonate (SDBS), CTAC, BS-12, and Diamite™ BFT [161], DTAC showed the maximum permeate recovery, with a recorded Sf value of 2.24. Additionally, DTAC exhibited the least variability in terms of desalination efficiency when applied to the contaminated NF membranes. The DTAC-recommended cleaning method, illustrated in Fig. 4, entails the mitigation of intermolecular and intramolecular electrostatic repulsions among anionic polyacrylamide (APAM) molecules. This decrease is facilitated by the binding of counterions, which is caused by the NF facility's feeding water having a high salinity. Micelles were created at

the hydrophobic ends of the APAM molecule chains as a result of increased DTAC concentrations causing a higher integration of DTAC monomers into the fouling layer and amplifying the electrostatic repulsions between APAM molecules. Concurrently, the electrostatic interactions that took place between the DTAC micelles and the APAM carboxylic acid functional group played a significant role in the adhesion of the DTAC micelles to the APAM chains. The electrostatic repulsions exhibited sufficient strength to cause the disruption of the fouling layer's structural integrity. The APAM molecules, accompanied by the associated components such as crude oil, Ca, Si, Ba, Al, and Fe were then discharged into the bulk solution as seen in Fig. 4b.

5.5. Others

One powerful chelating agent is EDTA, which may avoid unwanted interactions between metal ions in cleaning solutions by creating stable complexes with them. Scaling and mineral deposition are both prevented by EDTA's binding to metals, particularly calcium, which makes their removal easier. Surface and membrane cleanliness and performance are maintained by this additional chelation capacity, which guarantees a successful and comprehensive cleaning procedure. Cleaning formulas that include EDTA highlight the importance of this ingredient in removing metal pollutants and deposits of minerals [162]. EDTA is often used in commercially available cleaning products as an enzyme inhibitor, for instance, in the case of P3 Ultrasil 11. Research has shown evidence of its ability to impede the reconstruction of biofilms [163]. Some US-based factories that use PP membranes have had enzyme cleaning agents applied, and these agents have been demonstrated to be effective at temperatures as high as 45 °C [164]. Nevertheless, the widespread use of these technologies in the treatment of drinking water is limited due to financial considerations and regulatory requirements pertaining to potable water.

Ammonium bi-fluoride (NH_4HF_2) has been used as a facilitator in the elimination of silica deposits [165]. However, silica continues to pose significant challenges as a fouling agent due to its tendency to form inert silicate colloids characterized by strong surface attraction forces. Given its high insoluble nature, silica is difficult to hydrolyze [166]. Fluoride is the only destabilizing agent, capable of displacing the counter ion, but with potentially detrimental effects on some membranes. Ammonium bi-fluoride is periodically administered in order to disintegrate silica formations. Pre-treatment is a favorable approach for the removal of silica [167]. Table 4 summarizes current findings pertaining to the chemical cleaning of polymeric NF membranes.

6. Factors influencing the efficacy of chemical cleaning

The method of cleaning primarily entails the dissolving of material from the surface of the membrane, and several aspects might influence the chemical cleaning procedure. Multiple essential variables must be considered in membrane processes. These factors encompass temperature (to maintain an ideal operational range), pH level (to achieve a balanced acidity or alkalinity for membrane stability), detergent concentration (to adjust it to an effective level), the duration of chemical exposure to the membrane, and crucial operational factors such as pressure and cross-flow velocity. Every one of these characteristics is essential in guaranteeing the effectiveness and durability of procedures that rely on membranes. All parameters are thoroughly discussed in the following section.

6.1. Effects of concentration

The process of membrane cleaning relies on chemical interactions that occur between cleaning agents and fouling substances. Both reaction equilibrium and reaction rate can be influenced by the concentration of cleansing compounds. The precise concentration of cleaning chemicals is essential in ensuring the attainment of the optimal response rate. Nevertheless, it also has a significant influence on mitigating the mass transfer impediments posed by fouling layers. In practical applications, it is essential to ensure that the chemical concentration is sufficiently elevated to facilitate the attainment of the appropriate reaction rate. The process of mass transfer plays a crucial role in determining the minimum concentration required for effective cleaning. Madaeni et al. conducted an investigation on the utilization of chemical cleaning techniques for PVDF microfiltration membranes to eliminate whey proteins [179]. It was established that in the case of both acidic and alkaline solutions, an elevated concentration of cleaner leads to enhanced cleaning efficacy. It was asserted that this principle is also applicable to surfactants. In a study by Xing and colleagues [180], they looked at how a special filter cleans water in a city. They wanted to make the cleaning process better, so they tested different amounts of a cleaning solution. For groups treated with a little bit of the solution (0.2% NaClO), the water filter still worked pretty well, with 4% left blocked. But when they used a bit more solution (0.3% NaClO), 6% was still blocked. Surprisingly, after cleaning with a special solution for 15 minutes, the filters were much better. For the group with less solution, only 50% was still blocked, and for the group with more solution, only 35% was blocked. So, using a lower amount of the special cleaning solution worked better than a higher concentration.

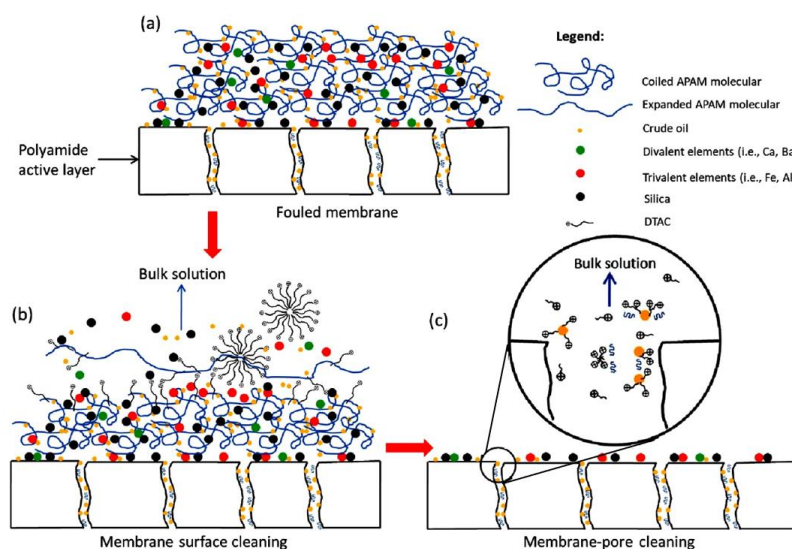


Fig. 4. Structure demonstrating the suggested DTAC cleaning procedure for NF membranes that have been polluted by generated effluent. (a) APAM molecules display coiled conformations within the fouling layer, (b) APAM molecules, along with associated substances including Al, Fe, Ca, Ba, Si, and crude oil, are subsequently released into the bulk solution and (c) DTAC monomers diffuse into membrane pores, adhering to crude oil droplets via their hydrophobic tails [161].

Table 4
Comparison of different reagents for washing the membrane and their efficiency in different foulant.

Polymeric Membrane	Fouling types	Cleaning agents	Best Cleaning agent	Ref.
NF270	Inorganic	NaOH, HCl	HCl	[113]
NF-1 8040	Anionic polyacrylamide and crude oil	C ₆ H ₅ O ₇ , EDTA-4Na, DTAC, CTAC	DTAC	[161]
NF90	Inorganic and organic foulants	NaOH, NaOCl, C ₆ H ₅ O ₇	Low-concentration C ₆ H ₅ O ₇	[168]
PVDF hollow fiber	Concentrated emulsions of oil	Sodium dodecyl sulfate (SDS), SDBS	SDBS	[169]
NF255	Organic foulants	C ₆ H ₅ O ₇ , NaOH, Na4-EDTA, Na-SDS	Na4-EDTA, Na-SDS	[170]
NF10	Organic foulants	The glucose oxidase enzyme	Immobilizing GOD on the aminated Fe ₃ O ₄ nanoparticles	[171]
XLE-2540	Humic acid (HA) Bovine serum albumin (BSA) Sodium alginate (SA)	IF, 1-ethyl-3-methylimidazolium acetate	IF in NaOH solution considerably improved irreversible membrane fouling	[172]
NF90 and NF270	Organic foulants	NaOH/EDTA solution, Methanol (20%, 50%, and ≥99.6%)	Pure methanol	[173]
NF90 and NF270	Organic foulants	Urea/HCl, NaOH/HCl	Urea/HCl	[174]
PVDF hollow fiber	Organic foulants	NaClO, EDTA, DTAC	Mix EDTA, NaClO and DTAC	[175]
PE-10 poly(ether sulfone)	Organic foulants	NaOH, NaOCl, SDS, EDTA	NaOCl	[176]
Cellulose triacetate membrane	Mix inorganic and organic foulants	Ethanol (70 w/w.%), Free nitrous acid (FNA) C ₆ H ₅ O ₇ ,	FAN	[177]
NF270	Inorganic foulants	HCl, Nitric, H ₃ PO ₄ , H ₂ SO ₄	HCl at a concentration of 0.20% w/w	[178]

According to Garmsiri et al. [181], a low concentration allowed for greater flux recovery. Upon using a specialized cleaning solution containing 5 mM of SDS + EDTA + NaOH, it was discovered that the water flow saw a significant improvement. Specifically, the flow increased by around 36% after the first cleaning and almost 49% after the subsequent cleaning. However, when a more concentrated solution of 10 mM was used, the flow rate was not as satisfactory. The first cleaning resulted in a modest improvement of 14%, whereas the subsequent cleaning yielded a somewhat higher improvement of around 19%. Therefore, the more potent solution proved to be less effective compared to the less potent one. Ahmad et al.'s [182] research on chemical cleaning of microfiltration membranes made of CA that had been clogged with microscopic algal accumulation. Additionally, concentrations of 0.1-1.0% were evaluated in order to determine the optimal cleaning agent concentration for chemical cleansing. It was noted that increasing the concentration of NaOCl leads to an increase in Jp, however, this effect was only significant at values below 0.75%. Kim et al. [183] investigated how different chemicals affect making the filter work better again. It was observed that the flux value exhibited a rise when the concentration of NaOH increased from 1% to 3%, reaching its highest recovery between 3% and 4%. The usage of 3% NaOH resulted in the maximum flux recovery. This represents 92% of the original water flow. They claimed that at concentrations greater than 5% NaOH, flux recovery declined.

Lee et al. showed that the presence of sodium dodecyl sulfate (SDS) at a concentration of 1 mM (millimole) had a negligible impact on the recovered flow [184]. The findings of this study suggest that the cleaning efficacy of SDS at this particular concentration is inadequate when attempting to remove fouling caused by NOM present in surface or ground water. When the concentration of SDS reached around 10 mM or more, the cleaning process resulted in the full restoration of the original water flow of the cleaned membrane. In contrast, Bartlett and colleagues [185] noted that, within sintered stainless steel and UF ceramic membrane systems, an elevation in NaOH concentration beyond the ideal concentration did not contribute to an improvement in the cleaning process; on the contrary, it led to a diminishes in cleaning efficacy. According to the findings of Li et al., it was observed that the augmentation of surfactant concentration, namely CTAB, ranging from 0.1-0.5wt.%, had a detrimental impact on the washing process. This negative effect could be attributed to the adsorption of surplus surfactants onto the surface of the membrane during the cleaning procedure [186]. In a research carried out by Ang et al. [187], it was observed that the cleaning effectiveness of EDTA and

SDS cleaning solutions exhibited an upward trend as the concentration of the cleaning agents increased. The researchers concluded that the use of SDS over its CMC is a crucial element in achieving effective cleaning with SDS. This is due to the fact that it enables an appropriate chemical interaction between SDS and the fouling substance, leading to the degradation of an alginate gel network. Hence, to have a more comprehensive understanding of the cleaning processes, it is necessary to take into account the "stoichiometry" that exists between the concentration of the detergent and the quantity of foulant present on the surface layer of the membrane.

6.2. Effects of pH

The recovered permeate flow of a humid acid-fouled membrane is influenced by the pH of the cleaning solution [188]. The research conducted by Simon et al. shows that the effectiveness of EDTA in cleaning is highly influenced by the pH level of the solution, which can be attributed to the deprotonation of functional groups [189]. Increasing the pH to 11 corresponds to intensifying the alkalinity. At this stage, all the carboxylic groups of a molecule undergo deprotonation, a process in which they lose a proton. By reducing the pH, we effectively decrease the alkalinity of the substances. Only a select number of carboxylic groups inside the molecule undergo deprotonation, either two or four in total. According to Ang et al. [187], the efficacy of EDTA, a cleaning agent, varies significantly based on the acidity or alkalinity of the cleaning solution. Modulating the pH level of the cleaning solution significantly impacts the efficacy of EDTA in the cleaning process. The experimental results demonstrate a significant enhancement in cleaning effectiveness, as shown by an increase from 25% to 44% when the pH is raised from 4.9 to 11.0 in the presence of EDTA. Conversely, a different cleaning agent known as SDS is not very concerned about the pH level of the cleaning solution, since its cleaning efficacy remains largely unaffected by it. The reason behind this is that SDS remains mostly in its active, cleansing form at both normal and elevated pH levels, owing to the pKa value of the sulfate functional group of SDS, which is 2.12. In summary, the pH of the cleaning solution is crucial for the effectiveness of EDTA but has less impact on SDS when it comes to removing dirt and deposits.

6.3. Effects of ionic strength

The efficacy of the chemical cleaning system is impacted by the existence of elevated ionic strength, whether it is in a solution containing just NaCl or in conjunction with a chemical agent. Lee's study revealed that a solution with a high concentration of salt (0.1 M NaCl) proved to be efficient in purifying a membrane that was contaminated by hydrophilic NOM (non-biological organic matter having an affinity for water) [184]. However, the use of SDS surfactant, caustic, and citric acid did not prove to be helpful in cleaning the hydrophilic NOM [190]. This phenomenon occurs due to the reduction of charges on the surface of the membrane and the decrease in acidity of the NOM. This leads to the pressing together of the double layer and conceals the charges. Moreover, it is conceivable that chloride ions (Cl⁻) are displacing undesirable chemicals from the water on the Horsetooth surface (HT-SW) that are linked to hydrophilic NOM. Put simply, the observed alterations occur due to a reduction in the charges on the membrane and NOM, perhaps accompanied by the replacement of undesirable substances in the water by chloride ions [191]. The effectiveness of removing hydrophobic NOM from soiled membranes was enhanced by employing a combination of high salinity and a caustic solution, such as NaOH or KOH. The efficacy of this mixture surpassed that of acid or SDS solutions. As reported by Brink [192], the incremental advantages resulting from increased cross-flow velocity and extended cleaning time were found to be insignificant in the context of high ionic strength cleaning. They found that greater ionic strengths resulted in more effective cleaning.

6.4. Effects of temperature

The cleansing process of membranes can be influenced by temperature in three primary ways. It has the ability to accelerate or decelerate the reactions that occur during the cleansing procedure. Second, it can alter the solubility of particles and grime that have been formed during the cleansing process in the solution. Ultimately, it has the potential to influence the equilibrium of the chemical reactions in progress. The literature review indicates that the chemical cleansing process is more effective at higher temperatures [193-195]. Nevertheless, the use of excessively high temperatures is sometimes hindered by the limited tolerance of membrane materials to heat. In general, it is recommended by membrane manufacturers that chemical cleaning procedures be conducted at temperatures below 45 °C [196]. Above the optimal temperature, chemical cleaning efficacy drops for both the sintered stainless-steel membrane and the ceramic membrane, as shown by Bartlett et al. [185]. Corbatón-Báguena et al. [197] evidenced that raising the temperature of the cleaning solution to around 42 °C resulted in a notable enhancement in water flux recovery, in contrast to the effects seen at 25 °C. The findings indicated that employing elevated temperatures throughout the cleaning process enhanced its efficacy in eliminating the particles on the membrane. The cleaning effectiveness of RO membranes was found to rise considerably from 20 to 40 °C, as shown by Ang et al. [187]. With the temperature rise, two significant events occurred. Initially, the chemical interactions between EDTA (a cleansing agent) and the debris on the membrane were accelerated. Furthermore, there was an augmentation in the transfer of foulant from the layer situated on the membrane to the adjacent cleaning solution. According to the findings of Goode et al. [198], an increase in temperature significantly impacts two critical factors. First, it significantly affects the amount of time required to sanitize, which has a substantial financial impact. Furthermore, it has an impact on the amount of water that can once again run continuously after being cleaned. In essence, elevated temperatures have a substantial influence on the rate and effectiveness of the cleansing process, which carries substantial ramifications for water flow recovery and financial investment. Additionally, it was shown that there is a significant correlation between cleaning speed and temperature, particularly at lower temperatures, notably when the temperature is below 40 °C. At a greater temperature of 70 °C, compared to a lower temperature of 22 °C, the cleaning rate, shown by the FR stat, was roughly 20% higher. Hoang et al. [199] investigated how flow recovery changed as a function of temperature during chemical cleaning. According to their findings, the flow was higher at 60 °C than at 25 °C. It was noticed that the flow exhibited a twofold increase at a temperature of 60 °C. That means that higher temperatures significantly impact flux. They also noted that high-temperature cleaning is completely safe.

Bartlett et al. [185] investigated the impact of temperature on the restoration of flow within a temperature range of 30-70 °C. The optimal concentration of NaOH (a cleansing agent) for a sintered stainless-steel membrane was determined to be 0.2% by weight. Conversely, in the case of a ceramic membrane, the most favorable concentration was found to be 0.4% by weight. The research demonstrated that the optimal NaOH concentration for successful flow recovery is dependent on the specific membrane type. Stainless steel membranes exhibited superior performance with a lower concentration, whereas ceramic membranes required a greater concentration. The ideal

temperature for both cases was found to be 50 °C. Additionally, it was shown that additional elevation of temperature results in a reduction in flux recovery. Moreover, it has been shown that an elevation in temperature leads to a reduction in the maximum flux recovery time. According to the findings, it was observed that the duration required for the sintered stainless-steel membrane to reach a certain condition fell from 8 minutes at 40 °C to 30 seconds when the temperature was raised to 70 °C.

6.5. Effects of Cleaning Time

The length of hydraulic cleaning conditions is a significant factor that can affect the recovery of flux in operational facilities. It was discovered that NOM foulants on membrane surfaces might be removed more effectively by washing for longer periods at lower velocities [200]. In some applications, a cleaning duration of 15 minutes has been shown to be sufficient [191]. However, in other membrane applications, a longer cleaning time of about 1 hour or even more can be required to provide the maximum cleaning impact and restore optimal membrane performance [201]. In a research conducted by Li et al. [202], it was observed that extending the cleaning duration from 10 to 20 minutes for surfactants such as SDS and CTAB resulted in a more pronounced enhancement in cleaning effectiveness. In the case of RO membranes, Ang et al. [187] found that the extension of the cleaning period from 15 to 60 minutes significantly improved the effectiveness of EDTA by providing a greater opportunity for the cleaning agent to engage with and disintegrate the deposits or fouling substances on the surface. The extended duration of contact allows EDTA to dissolve and eliminate recalcitrant impurities more efficiently, leading to a more comprehensive and efficient cleaning procedure. Nevertheless, the cleaning efficacy of SDS at low concentrations, with either 15 or 60 minutes of cleaning time, was shown to be unsatisfactory likely because the concentration of SDS used was insufficient to effectively break down and remove the fouling materials. Put simply, increasing the duration did not significantly enhance the efficacy of SDS cleaning. The projected reduction in the contact between the compounds responsible for fouling was expected to be insignificant unless there was a beneficial chemical reaction occurring between the fouling substances and the cleaning agents present in the fouling layer [203]. The literature lacks an in-depth discussion of the duration of chemical cleaning under various operational settings. Nevertheless, it has been indicated that shorter filtering cycles, which need more frequent but longer cleaning processes, provide advantages. This is because fouling layers tend to grow more compact with time, making them more challenging to remove [204,205].

6.6. Effects of pressure

The maintenance of operating pressure is crucial in ensuring the proper functioning of a system since it is closely linked to its hydrodynamics. The cleaning process should be conducted with little pressure to prevent the foulant layer from being forced into the surface, thus increasing its adhesion. In contrast, Bartlett et al. [185] found that the presence of surface deposits hinders the achievement of optimal cleaning efficiency when any applied pressure is used during the cleaning process. However, Bartlett et al. [185] noted that these findings show that, as long as surface deposits are present, using pressure during cleaning would not maximize cleaning effectiveness. Sayed et al. reported a similar pattern [206]. Insufficient coverage of this issue in the existing literature necessitates more experimentation to ascertain the correlation between operating pressure and the efficacy of cleaning. In our investigation of the cleaning process for NF membrane systems, Fig. 5 illustrates the key factors influencing chemical cleaning effectiveness. The comprehensive analysis presented in this figure contributes valuable insights into optimizing membrane performance through strategic cleaning protocols.

7. Evaluating the ecological impact of cleaning agents

The ecological ramifications of cleaning agents are of utmost importance, particularly within the framework of sustainable industrial methodologies. This factor is crucial as it directly impacts the ecological equilibrium, preservation of resources, and the general dedication to reducing the carbon emissions linked with industrial operations. As industries aim to achieve sustainability, it is crucial to assess and minimize the ecological impacts of cleaning chemicals as part of responsible and environmentally friendly operations. Table 5 demonstrates the environmental impact of the chemical agent when introduced into the natural environment.

To mitigate the environmental consequences linked to traditional cleaning chemicals, it is advisable to embrace eco-friendly chemical substitutes. By using ecologically mindful cleaning methods, we can reduce the negative impact on ecosystems and actively support the development of sustainable, environmentally friendly solutions. Adopting environmentally friendly

chemical agents not only demonstrates responsible environmental management but also signifies a dedication to protecting the fragile equilibrium of our ecosystems for future generations. Suggested in Fig. 6 are examples of eco-friendly chemical agents, proposed as a strategy to mitigate chemical agent pollution and promote a more harmonious equilibrium in nature and the environment.

The process of chemically cleaning the fouled membranes is highly efficient in eliminating the majority of the foulants and restoring the membrane's performance to nearly its original state. However, this procedure causes degradation of the membranes, resulting in a significant reduction in their expected lifespan. Consequently, frequent replacement and substitution

become necessary, leading to increased operating expenses. Moreover, the proper disposal of the used chemicals is a significant obstacle and poses a risk to the surrounding ecosystem. Hence, there is a pressing need for rigorous endeavors to create alternative technologies that are not only efficient and feasible in practical scenarios, but also environmentally sustainable. Various technologies that have attracted considerable research attention for mitigating fouling are summarized in Fig. 7, along with an indication of their level of advancement. [213-215]. These technologies possess the capability to effectively and sustainably eradicate contamination from membranes, thereby overcoming the current obstacles and challenges.

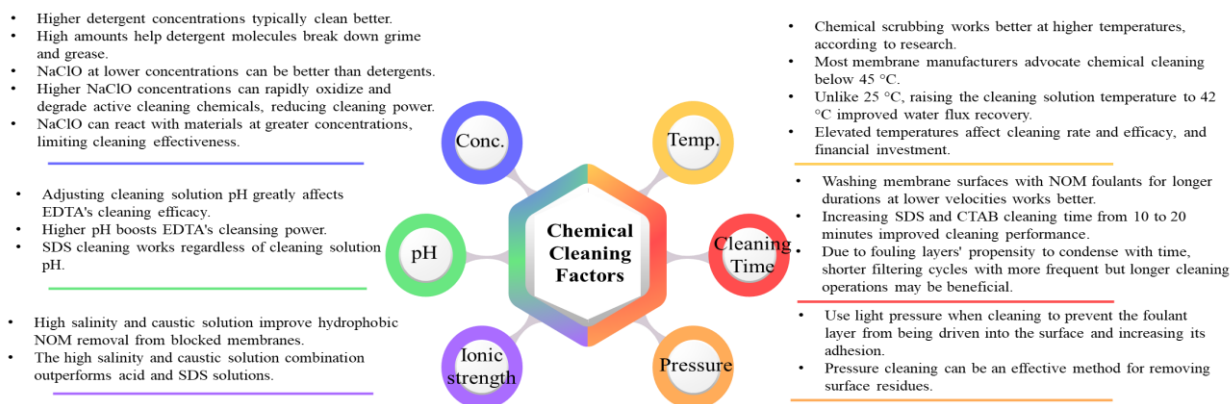


Fig. 5. Key factors affecting chemical cleaning effectiveness in NF membrane systems.

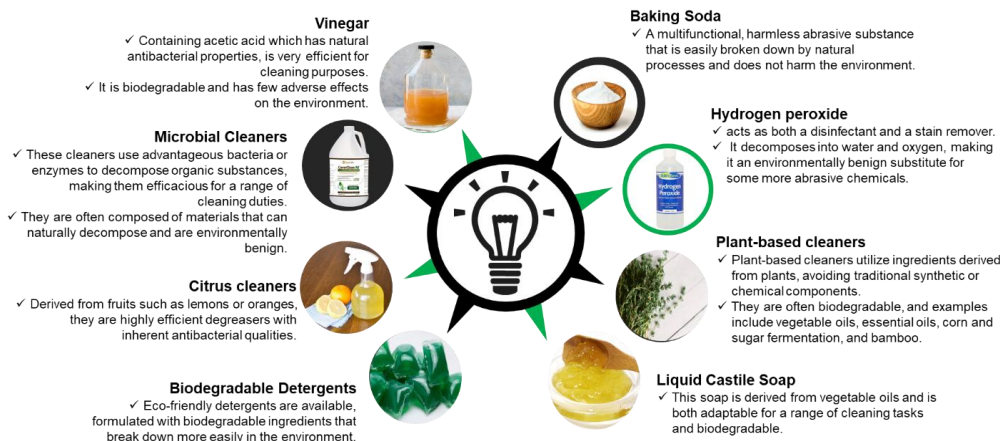


Fig. 6. Illustrating Eco-Friendly Chemical Agents and Their Properties.

Table 5
Environmental impact of introducing chemical agents into the natural environment.

Chemical Agent	Effects of cleaning agents on the environment	Ref.
Acids	<ul style="list-style-type: none"> Acids change soil and water pH, affecting nutrient availability and aquatic organisms. Acidic industrial cleaning treatments degrade infrastructure. Respiratory and skin discomfort may result from volatile acidic chemicals in the air. 	[207]
Alkaline agents	<ul style="list-style-type: none"> Alkaline chemicals modify the pH of soil and water, which in turn influences the availability of nutrients and has the potential to disrupt aquatic ecosystems. Alkaline cleaning solutions have the potential to cause corrosion in metals, which may be hazardous. Certain substances contain ammonia, which may contribute to air pollution if it is discharged. 	[208]
Complexing agents (water softeners)	<ul style="list-style-type: none"> Complexing agents form stable complexes with metal ions, strengthening metal mobility and thus complicating wastewater treatment. 	[209]
Detergents (surfactants)	<ul style="list-style-type: none"> Certain compounds, especially phosphorus-containing ones, enhance nutrients, causing eutrophication in water. Detergent surfactants injure aquatic creatures and cause foam in water bodies, which slows water treatment and degrades water quality. Runoff from detergent-cleaned surfaces may transfer surfactants into soil. This affects soil structure and microbiology. Some surfactants are persistent organic pollutants, posing long-term environmental risks. 	[210]
Solvents	<ul style="list-style-type: none"> Air pollution from solvent-volatile organic compounds is considerable. Accidental spills or improper disposal may damage water and soil. Among other things, chlorinated solvents deplete the ozone layer. Solvent exposure may harm respiratory and brain systems, causing health risks. 	[211]
Disinfectants	<ul style="list-style-type: none"> Harmful disinfection by-products may be formed by residual disinfectants present in water. Disinfectants have detrimental effects on aquatic habitats and may contribute to the development of bacterial resistance. Their introduction may have an effect on soil, sediment, and contribute to the pollution of indoor air. 	[212]

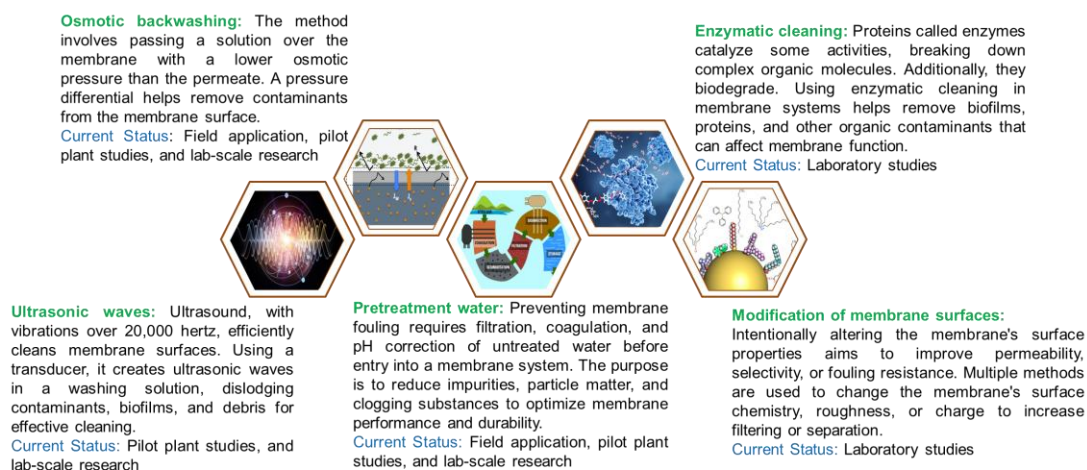


Fig. 7. The present status of environmentally conscious fouling control in NF membrane systems and their degree of progress.

8. Perspective and Conclusion

The cleaning process parameters include the selection of appropriate cleaning agents and their respective concentrations, the effects of the ionic strength, the sequencing, and length of cleaning processes, pressure, pH levels, and temperature. The aforementioned parameters influence the result of the cleaning and disinfection operation, necessitating a comprehensive examination to determine the most effective cleaning and disinfection technique and minimize membrane damage. Nevertheless, despite the existing understanding and widespread knowledge of the fundamental principles that determine the optimal needs for membrane cleaning and disinfection, ongoing research is currently being conducted. Undoubtedly, with the advancement of membrane detergent and materials, the emergence of novel processing applications, and the influence of environmental and cost factors on the industry, there persists an ongoing necessity to conduct research aimed at comprehending the mechanisms underlying cleaning and disinfection. Therefore, it is crucial for the integration of these mechanisms into both qualitative and quantitative models pertaining to cleaning and disinfection. To guarantee that the results of the research activities best meet the objectives of industry practitioners, it is also crucial that these studies be carried out in environments that closely resemble the industrial process, with sufficiently lengthy run lengths and repeated intervals. The sorts of membrane fouling processes that cause plant performance to decline and how those processes are then recovered by membrane chemical cleaning are the main topics of this review. This study has explored many forms of both organic and inorganic fouling. The occurrence of fouling issues gives rise to elevated operational expenses, increased energy requirements, diminished membrane lifespan, and a heightened need for cleaning interventions. Our research also demonstrates that chemical cleaning procedures for NF membranes are often used to restore membrane function, despite the existence of some restrictions associated with these cleaning approaches. Chemical cleaning methods' effectiveness depends on a number of factors, such as the type of fouling material (organic, inorganic, biological, particulate, or chemical), the type of detergents used (acids, alkalis, enzymatic cleaners, solvents, disinfectants, oxidizing or chelating agents), the temperature, cleaning chemical concentration, the length of time the chemical solution is in contact with the membrane, pH levels, and operating parameters like pressure. Since the results of the cleaning technique are affected by all of these factors, it needs to be thoroughly explored to find the best cleaning strategy. In order to improve and analyze the effectiveness of cleaning methods, the traditional method of flux measurement has been utilized for decades.

Obtaining defined solutions in membrane design and fouling prevention is a significant challenge. However, some approaches are available for preventing membrane fouling, including modifications to the membrane itself, adjusting the system's function parameters, and pre-treatment. Until now, the implemented preventative measures have not achieved a full cessation of fouling on the surface of the membrane. Nevertheless, these substances are effective in preventing or slowing down the accumulation of unwanted deposits or contaminants on the membrane's surfaces, hence resulting in a decrease in both the frequency and length of cleaning procedures. Each of these consequences has an important impact on the manufacturing and lifetime of the membrane.

Directly achieving the creation of highly efficient systems with decreased fouling and excellent cleaning capabilities can be accomplished by

implementing precise modifications. These modifications include optimizing the properties of the membrane surface in accordance with the chemical and physical attributes of the water being treated, as well as the operational parameters of the system. Comprehensive research efforts are necessary to examine and develop novel concepts and methodologies within the realm of membrane cleaning and repair. The NF membrane sector greatly benefits from recognizing the importance of dedicating additional effort and time to understanding contamination mechanisms. This will aid in the development of economical, practical cleaning and recovery techniques tailored to each case of fouling, significantly enhancing the industry's effectiveness. Additionally, self-cleaning or fouling-resistant membranes need to be created to reduce cleaning time and expenses. Nevertheless, there is still uncertainty about the scalability of the majority of procedures and their efficacy and reliability when expanded to the level of a factory. Another issue to consider is the durability of the material over extended periods of time when subjected to high pressure, fluctuating temperatures, and constant contact with extremely salty water containing a variety of contaminants. Furthermore, scientific efforts should be focused on developing environmentally friendly chemical agents to promote a more sustainable approach to cleaning operations.

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Data availability

No data was used for the research described in the article.

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 N. Hashim: Formal analysis
 N. D. A. P. Kerisnan: Methodology
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 R. B. R. Mamat: Resources; Validation

Declaration of Competing Interest

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

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