Andrii Odnoralov

EXTENDING THE SERVICE LIFE OF REVERSE OSMOSIS MEMBRANES:

an author-developed multistage chemical regeneration procedure and its industrial validation

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This monograph was conceived in the context of real industrial operation, where every cubic meter of purified water has a cost, and every equipment stoppage entails economic losses.

The monograph is intended first and foremost for practicing engineers, technical directors, and operations specialists who are responsible for ensuring the stable and cost-effective performance of RO systems. It will also be of use to researchers, graduate students, and students specializing in membrane technologies, as it consolidates practical experience and links it to fundamental scientific principles.

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PREFACE

In an era when water resources are becoming ever more valuable and water-quality requirements ever more stringent, membrane technologies — and reverse osmosis in particular — have taken center stage in water treatment and desalination. Their high efficiency and versatility have made them the de facto standard across numerous sectors, from municipal water supply to the pharmaceutical and food industries. Yet behind the elegance of membrane separation lies a fundamental practical problem familiar to every engineer and technician: the relentless decline in performance caused by membrane contamination, the so-called "fouling effect".

This monograph was conceived in the context of real industrial operation, where every cubic meter of purified water has a cost, and every equipment stoppage entails economic losses. Over many years of practical work, the author repeatedly confronted the same discouraging reality: expensive reverse osmosis (RO) membranes, whose stated service life is five years or more, were failing after only a year to a year and a half. Standard manufacturer-recommended chemical clean-in-place (CIP) procedures showed low—and more importantly, short-lived—effectiveness, restoring only 40–50% of performance and failing to bring about any decisive extension of element service life.

This discrepancy between the technology's theoretical potential and the harsh realities of its operation became the starting point for the present study. There arose a need to move beyond standard procedures toward a deep understanding of root causes: to examine the complex physicochemical and biological mechanisms underlying the formation of deposits of varying nature — from inorganic salts to the polymer matrices of biofilms. It became evident that there is no universal solution; combating multicomponent contamination requires not a one-off cleaning but a sequential, targeted strategy aimed at layer-by-layer removal of each contaminant type.

The outcome of this search is the author-developed multistage chemical regeneration procedure presented herein. Its core is not merely a new set of reagents, but a coherent system based on deliberate pH shifts and the use of specialized complexing (chelating) and dispersing agents. This approach makes it possible not simply to "clean" the membrane surface, but to "regenerate" it — restoring operating characteristics to values close to rated specifications and, crucially, doing so repeatedly throughout the membrane's service life.

A defining feature of this work is its focus on industrial validation. All conclusions and results presented are grounded not in short-term laboratory experiments, but in multi-year (over 60 months) observations of pilot and full-scale industrial plants. In the author's view, this aspect is of greatest value to the primary audience of this book.

The monograph is intended first and foremost for practicing engineers, technical directors, and operations specialists who are responsible for ensuring the stable and cost-effective performance of RO systems. It will also be of use to researchers, graduate students, and students specializing in membrane technologies, as it consolidates practical experience and links it to fundamental scientific principles.

The work proceeds as follows: the mechanisms of fouling are examined (Chapter 1); a critical analysis of existing cleaning methods is provided (Chapter 2); the author's procedure is described in detail (Chapter 3); and comprehensive evidence of its effectiveness and superiority is presented (Chapter 4). Finally, Chapter 5 considers practical aspects of integration into industrial operating cycles and assesses the colossal economic and environmental impact achieved through a fivefold extension of membrane service life.

INTRODUCTION

Against the backdrop of a growing global shortage of fresh water driven by climate change, population growth, and industrial development, desalination and water treatment technologies have acquired strategic importance for ensuring a sustainable future [1]. Among existing methods, membrane separation — and reverse osmosis (RO) technology in particular — has assumed a dominant position. Today, RO plants account for more than 65% of global capacity for desalinating seawater and brackish water, making this technology a cornerstone of modern water infrastructure [2]. Such broad adoption is explained by a set of indisputable advantages: higher energy efficiency compared with traditional thermal methods, superior selectivity toward dissolved salts and contaminants, as well as scalability and relative operational simplicity [3].

Nevertheless, despite technological maturity and wide deployment, RO systems face a fundamental operational problem that significantly offsets their advantages and constrains further development. This problem is membrane fouling — an inevitable process of deposition and accumulation of various substances on the surface and within the pores of membrane elements [4]. Fouling is a complex, multifactorial process that triggers a cascade of adverse consequences: a reduction in production of purified water (permeate), an increase in operating pressure and, consequently, higher energy consumption, the need for frequent and aggressive chemical cleanings, and, ultimately, irreversible degradation and shortening of the service life of membrane elements [5].

The economic and environmental ramifications are substantial. Direct operating costs associated with fouling include expenditures for electricity, procurement of chemical reagents for cleaning and of antiscalants, equipment downtime for scheduled maintenance, and, most critically, premature replacement of costly

membrane modules [6]. Studies show that extending membrane service life is the most influential factor in reducing overall operating expenses for desalination [7]. From an environmental perspective, the problem is exacerbated by the fact that end-of-life membranes, composed of non-biodegradable polymers, are typically not recycled but disposed of in landfills, contributing to global plastic pollution [8].

In response, industry and the research community have developed a range of fouling-control strategies, including feedwater pretreatment, optimization of hydrodynamic regimes, and, above all, periodic chemical clean-in-place (CIP) procedures. However, as multi-year practical experience and independent studies indicate, existing standard approaches and commercially available cleaning formulations have limited effectiveness. They often remove no more than 40–50% of deposits. The contaminants that remain on the membrane surface form a dense, persistent barrier that not only depresses baseline performance but also acts as a "seed" layer catalyzing accelerated re-fouling. This sets off a vicious cycle: incomplete cleaning leads to rapid recurrence of the problem, necessitating more frequent cleanings which, in turn, cause cumulative chemical and mechanical damage to the membrane, resulting in its premature failure.

This monograph is devoted to resolving this key problem. It presents and theoretically substantiates an author-developed approach to regenerating RO membranes that differs fundamentally from standard cleaning procedures. The multistage chemical regeneration procedure, validated under industrial conditions, makes it possible not merely to achieve temporary improvements, but to regenerate the membrane surface deeply — restoring its performance and selectivity to 90–93% of initial values. Practical application of this procedure has demonstrated the possibility of extending the service life of membrane elements by up to fivefold — from one year to five years — while maintaining consistently high operating characteristics.

The aim of this work is to systematize and present to the scientific and engineering community the theoretical foundations,

practical implementation, and industrial validation of the proposed procedure's effectiveness. The monograph is structured to lead the reader sequentially from the fundamentals of reverse osmosis and an in-depth analysis of fouling mechanisms to a detailed exposition of the author's regeneration technology, a description of its industrial trials, and an assessment of the technical, economic, and environmental benefits achieved.

Chapter 1. THEORETICAL FOUNDATIONS AND TECHNOLOGICAL ASPECTS OF REVERSE OSMOSIS SEPARATION

a thorough understanding of fouling and degradation in reverse osmosis (RO) membranes — and of scientifically grounded methods for their regeneration — requires consideration of the fundamental physicochemical principles underlying the process, the key characteristics of the materials and equipment employed, and the influence of operating parameters on separation efficiency.

1.1. Physicochemical principles of reverse osmosis

Osmosis is the spontaneous transport of solvent molecules (e.g., water) across a semipermeable membrane from a region of lower solute concentration to a region of higher solute concentration (Figure 1). The driving force of this process is the gradient in the solvent's chemical potential. The pressure that must be applied to the more concentrated solution to halt this spontaneous transfer is called the osmotic pressure (Π) [3].

Reverse osmosis, as the name implies, is the process opposite to osmosis (Figure 1). If an external pressure (P) greater than the osmotic pressure (P > Π) is applied to the high-concentration side, the direction of solvent flow reverses. Water molecules pass through the semipermeable membrane from the concentrated solution to the dilute one, while the ions of dissolved salts and other impurities are retained by the membrane [9]. The pressure difference ($\Delta P - \Delta \Pi$) serves as the effective driving force of the process.

Mass transfer through the dense selective layer of an RO membrane is most accurately described by the solution—diffusion model (Figure 2) [3]. According to this model, separation proceeds in three stages:

- 1. Absorption: Water molecules and solutes adsorb onto the membrane surface on the feed side.
- 2. Diffusion: Molecules diffuse through the polymer matrix of the membrane under a concentration gradient.
- $3.\ Desorption;$ Molecules desorb from the membrane surface on the permeate side.

The key operating characteristics of any RO membrane are its permeability and selectivity.

Permeability characterizes the rate of water transport per unit membrane area at a given pressure and is commonly expressed as permeate flux, measured in liters per square meter per hour (L/m^2 ·h) or gallons per square foot per day (GFD).

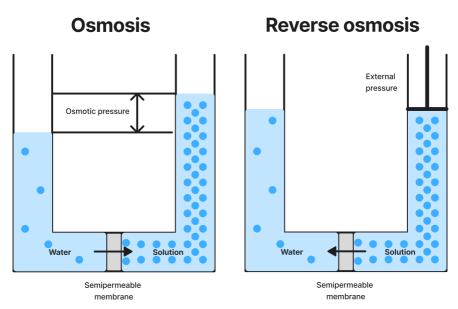


Figure 1. Operating principle: left — osmosis; right — reverse osmosis

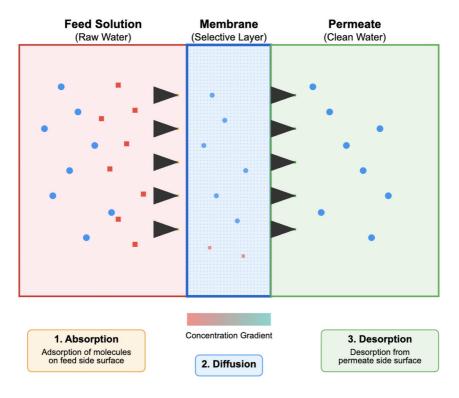


Figure 2. The solution-diffusion model

Selectivity reflects the membrane's ability to retain dissolved species and is calculated as the percentage salt rejection (R). It is defined by:

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% ,$$

where C_p is the salt concentration in the permeate, and C_f is the salt concentration in the feed water.

These two parameters are interrelated and often involve a trade-off: as a rule, membranes with higher selectivity exhibit lower permeability, and vice versa.

1.2. Classification and characteristics of RO membranes

Modern industrial RO membranes can be classified by the material of the selective layer and by the module's construction. Understanding these properties is critical, since the membrane's material and structure directly determine its susceptibility to various fouling types and its resistance to cleaning chemicals.

1.2.1. Membrane materials

Historically, the first — and for a long time dominant — membranes were made of cellulose acetate (CA). Their key advantage is high resistance to oxidants, particularly free chlorine (up to 20 mg/L in the form of chloramines). However, CA membranes lag behind modern counterparts in selectivity and performance and possess a narrow operating pH range (typically 5–8), as they are prone to hydrolysis in acidic media and saponification in alkaline media. In addition, cellulose acetate serves as a nutrient source for microorganisms, rendering these membranes vulnerable to biofouling [10].

Today's industrial standard is the thin-film composite (TFC) membrane based on aromatic polyamide (PA). These membranes have an asymmetric structure:

- Support layer: A microporous polysulfone layer providing mechanical strength to the selective layer under high pressures.
- Backing: A nonwoven polyester fabric that provides structural support for the entire laminate [5].

TFC membranes deliver substantially higher performance and selectivity than CA analogs and can operate over a wide pH range (2 to 11), expanding options for chemical cleaning [10]. Their principal drawback, however, is extremely low resistance to free

chlorine and other strong oxidants. Chlorine concentrations even at 10–15 mg/L cause irreversible damage to the polyamide layer, necessitating careful dechlorination of the feed water before it enters the plant [10].

1.2.2. Membrane module construction

In industrial plants, the most common configuration is the spiral-wound membrane (SWM) element (Figure 3). This construction maximizes filtering surface area per unit volume, making systems compact and cost-effective. The element consists of multiple membrane leaf sets and feed spacers (turbulators) wound around a central perforated permeate collection tube [11].

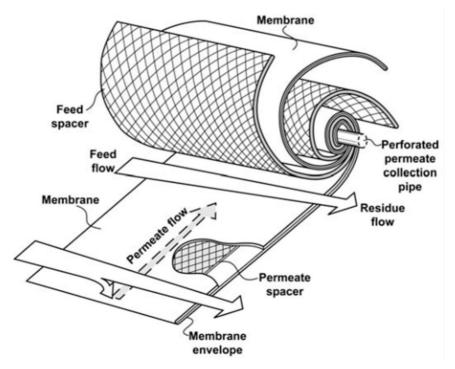


Figure 3. Configuration of a spiral-wound membrane element [11]

Table 1

(Based on the author's data and leading manufacturers' specifications $\left[10
ight]$ Comparative characteristics of industrial RO membrane types

Applications	Legacy systems; niche ap-	plications requiring chlorine tolerance	99.0–99.8+ Desalination of seawater	and brackish water; indus-	trial water treatment; pro-	duction of ultrapure water
Typical NaCl re- jection (%)	95–98		+8.66-0.66			
Free chlorine tolerance (ppm·hr)	> 1000		< 1000	(short-term	< 0.1 ppm)	
Oper- Max. oper- ating ating tem- pH perature (°C)	35–40		45			
Oper- ating pH range	5.0-8.0		2.0-11.0			
Selective layer ma- terial	Cellulose	acetate	Thin-film Aromatic 2.0–11.0	composite polyamide	(PA)	
Mem- brane type	Cellulose	acetate (CA)	Thin-film	composite	(TFC)	

Despite the advantage of high specific surface area, this design introduces serious hydrodynamic issues. The feed spacers, required to create flow channels for the feed stream, disturb laminar flow and give rise to stagnant regions, or "dead zones" [12]. In these areas, local velocity is significantly reduced, sharply amplifying concentration polarization and creating ideal conditions for colloid deposition and salt crystallization — processes at the core of membrane fouling. Thus, the very construction that makes RO industrially viable is simultaneously one of the principal factors provoking its main operational challenge.

1.3. Influence of operating parameters on the separation process

The efficiency of an RO plant results from a delicate balance among several key operating parameters. Optimizing one parameter often adversely affects another, underscoring the need for an integrated approach to process management and, in particular, to fouling control.

- Pressure. Increasing transmembrane pressure (TMP) produces an almost linear rise in permeate flux, while the salt flux changes only slightly. This allows both productivity and selectivity to be increased. However, excessively high pressure compacts the membrane's polymer matrix (creep), causing irreversible declines in permeability over time. Moreover, high pressure is directly tied to higher energy consumption [3].
- Temperature. Raising the feed temperature reduces its viscosity, facilitating water passage through the membrane and thus increasing permeate flux. At the same time, the rate of salt diffusion through the membrane also rises, lowering selectivity. Elevated temperatures further accelerate polymer degradation (creep) and markedly intensify biological activity, promoting biofouling [13].

- Recovery rate. This parameter indicates what fraction of the feed is converted into permeate. From an economic standpoint, maximizing recovery is desirable to reduce concentrate discharge. However, this markedly increases the concentration of all dissolved salts and suspended solids in the concentrate (retentate) stream. As a result, osmotic pressure rises sharply and most critically the likelihood of exceeding the solubility limits of sparingly soluble salts increases dramatically, directly causing inorganic scaling [14].
- Feedwater composition. The chemical composition of the feed is a determining factor. High total dissolved solids (TDS) require higher operating pressures to overcome the osmotic barrier. The presence of specific ions (e.g., Ca²⁺, Mg²⁻, SO₄²⁻, HCO₃⁻), iron species, and silica directly governs the type and intensity of potential membrane fouling [3].

1.4. The phenomenon of concentration polarization (CP)

Concentration polarization is the key phenomenon linking RO hydrodynamics to membrane fouling mechanisms. During filtration, as water permeates the membrane, the retained salt ions and other non-permeating species accumulate at its surface. This forms a thin boundary layer in which the solute concentration significantly exceeds that in the bulk flow [9].

This phenomenon has two principal detrimental effects:

- 1. Reduction of the effective driving force. The elevated salt concentration in the boundary layer generates a local osmotic pressure at the wall (Π _wall) that is substantially higher than the osmotic pressure in the bulk solution (Π _bulk). This reduces the effective driving force ($\Delta P \Delta \Pi$ _wall), leading to permeate production lower than theoretically attainable [4].
- 2. Scaling initiation. The concentrations of sparingly soluble salts (e.g., calcium carbonate, calcium sulfate, silicates) in the

boundary layer can readily exceed their solubility limits. This leads to supersaturation immediately at the membrane surface and initiates crystallization and deposition — i.e., the formation of inorganic scaling [15, 16]. Concentration polarization is thus a direct catalyst of one of the most problematic fouling types.

The principal tool for mitigating concentration polarization is to induce turbulent flow along the membrane surface by maintaining a high cross-flow velocity (CFV). Turbulent eddies effectively "wash away" the concentrated boundary layer, returning ions to the bulk and reducing their near-wall concentration [17]. However, sustaining high flow velocity requires substantial pumping energy and creates a large pressure drop along the length of the membrane module, which is also undesirable. Moreover, as noted, spiral-wound elements with spacers invariably contain stagnant regions where cross-flow is minimal; CP is strongest precisely in these areas, triggering fouling processes. This fundamental limitation underscores that hydrodynamic measures alone cannot fully prevent fouling, rendering chemical control and regeneration methods absolutely essential.

Chapter 2. FUNDAMENTAL OPERATIONAL CHALLENGES: MEMBRANE FOULING AND DEGRADATION

despite comprehensive feed pretreatment and optimization of operating regimes, the gradual decline in the performance of reverse osmosis (RO) plants is an inevitable consequence of fouling and degradation of membrane elements. These processes are complex and constitute the principal obstacle to long-term, cost-effective, and sustainable operation of membrane systems. This chapter provides an exhaustive classification of fouling types, examines their mechanisms and diagnostic methods, and presents a critical analysis of existing control strategies, thereby substantiating the need for a fundamentally new approach to membrane regeneration.

2.1. Comprehensive classification and mechanisms of deposit formation

Under real industrial conditions, membrane fouling rarely consists of a single class of substances. As a rule, a complex, multicomponent conglomerate forms on the surface — comprising inorganic salts, organic molecules, colloidal particles, and microorganisms [18, 19]. These components interact synergistically, producing a dense, difficult-to-remove layer. For systematic analysis, four principal fouling types are conventionally distinguished.

2.1.1. Inorganic scaling

This fouling type is the crystallization and growth on the membrane surface of sparingly soluble inorganic salts. The primary driving force of scaling is solution supersaturation within the boundary layer, induced by concentration polarization [20, 21]. The most common inorganic deposits include:

- Calcium carbonate (CaCO₃). The most frequent cause of scaling in natural-water treatment. Its formation is described by a rightward shift in the calcium—carbonate equilibrium: Ca(HCO₃)₂ ⇔ CaCO₃ ↓ + CO₂ + H₂O. This shift is promoted by the membrane's permeability to dissolved CO₂, which permeates with the product water (permeate), raising the pH in the concentrate and favoring CaCO₃ precipitation. For forecasting the risk of carbonate scaling, the Langelier Saturation Index (LSI) is used [17].
- Calcium, barium, and strontium sulfates (CaSO₄, BaSO₄, SrSO₄). These salts especially BaSO₄ and SrSO₄ have very low solubility that, unlike calcium carbonate, depends only weakly on pH. Sulfate deposits form dense, hard crystalline layers that are extremely difficult to remove chemically [15].
- Metal hydroxides (Fe(OH)₃, Mn(OH)₂, Al(OH)₃). Deposits of iron, manganese, and aluminum compounds (often introduced with coagulant carryover from pretreatment) present a serious challenge. Their precipitation is promoted by local pH elevation within the boundary layer.
- Silica (SiO₂). Silica fouling is among the most difficult to control and remove. Monomeric silicic acid in water can polymerize to form a gel-like layer on the membrane surface which, over time, "ages", densifies, and becomes virtually insoluble to standard acidic and alkaline reagents [22].

2.1.2. Organic fouling

This fouling mode arises from adsorption of dissolved organic matter on the membrane surface. Polyamide TFC membranes, being relatively hydrophobic and bearing a negative surface charge at neutral pH, are particularly susceptible due to hydrophobic interactions and hydrogen bonding [23]. The principal sources of organic foulants are:

- Natural organic matter (NOM): humic and fulvic acids present in surface waters.
- Anthropogenic contaminants: surfactants, oils, and petroleum products.
- Pretreatment residuals: polymeric flocculants and coagulants.

A key mechanism exacerbating organic fouling is "cation bridging". Divalent cations such as Ca²⁺ and Mg²⁺ can bridge between negatively charged functional groups of organic molecules (e.g., carboxyl groups of humic acids) and the negatively charged membrane surface. This yields a robust, cross-linked "membrane—cation—organics" complex that is far more difficult to remove than simple adsorbates [18].

2.1.3. Colloidal/particulate fouling

This fouling type is associated with deposition on the membrane of finely dispersed suspended particles ranging from nanometers to several micrometers [24]. These include clay, silt, colloidal silica, iron oxides, as well as large organic colloids and bacterial aggregates [25].

Formation of the colloidal layer (the "cake layer") involves particle transport to the membrane by the permeative flow and subsequent deposition. Initial adhesion is governed by the balance between the drag force that presses the particle toward the membrane and repulsive forces (e.g., electrostatic) that oppose attachment [26]. Over time, the layer compacts under pressure, its hydraulic resistance increases, and performance declines markedly [24].

2.1.4. Biofouling

Biofouling is recognized as the most complex, least controllable, and most detrimental fouling type [27]. It is a dynamic process that unfolds in several stages:

- 1. Initial attachment. Individual microorganisms (bacteria, fungi) present in the water attach to the membrane surface. This is facilitated by pre-existing organic and inorganic deposits that create an adhesion-friendly interface.
- 2. Growth and proliferation. Attached microorganisms proliferate, using organics in the feed water as nutrients.
- 3. Biofilm formation. The growing colony produces extracellular polymeric substances (EPS) a complex mixture of polysaccharides, proteins, nucleic acids, and lipids. EPS forms a gel-like matrix that firmly binds cells to one another and to the membrane surface, yielding a mature biofilm [28].
- 4. Maturation and dispersion. The biofilm matures, developing a complex three-dimensional architecture with channels for water and nutrient circulation. Fragments of the biofilm may periodically detach and be carried downstream, seeding new sections of the membrane system [27].

A biofilm not only imposes significant hydraulic resistance, it also shields microorganisms from biocides and detergents, rendering chemical cleaning minimally effective [45]. In addition, bacterial metabolites can cause chemical degradation of the membrane material itself (particularly CA membranes) and adversely affect the organoleptic properties of the permeate [29, 30].

2.2. Diagnostics and monitoring of membrane condition

The efficiency and longevity of RO systems depend directly on the ability to identify and quantify membrane fouling processes in a timely manner. A diagnostic protocol is the cornerstone for

 ${\bf Classification\ of\ the\ principal\ RO\ membrane}$ ${\bf columnt\ types}$

data synthesized from [23]

Type of fouling	Main components	Dominant mechanism	Key influ- encing factors	Visual/ operational indicators
Inorganic scaling	CaCO ₃ , CaSO ₄ , BaSO ₄ , SiO ₂ , metal hy- droxides (Fe, Mn, Al)	Crystallization from a supersaturated boundary layer	pH, tem- perature, ion concentra- tions, recov- ery rate	Rapid increase in pressure drop; reduced selectivity (salt passage), especially in the last elements of a stage
Organic fouling	Humic and fulvic acids (NOM), sur- factants, oils, polymeric coagulants	Adsorption, hydrophobic interactions, cation bridg- ing (Ca ²⁺)	Organic content (TOC), presence of divalent cations, pH	Gradual decline in throughput; progressive rise in pressure drop
Col- loidal fouling	Clays, silt, iron oxides, colloidal SiO ₂ , organic colloids	Particle deposition and for- mation of a cake layer	Turbidity, SDI, sus- pended solids con- centration, hydrody- namics	Gradual increase in pressure drop along the entire stage, especially in the lead elements
Biofoul- ing	Bacteria, fungi, algae, extracellular polymeric substances (EPS)	Microbial adhesion, growth and multiplica- tion, biofilm formation	Tem- perature; nutrient availabil- ity (AOC, TOC, N, P); ineffective disinfection	Marked, rapid rise in pressure drop; through- put decline; pos- sible deteriora- tion of permeate taste and odor

developing a scientifically grounded chemical regeneration strategy, enabling a transition from empirical practices to targeted intervention against specific foulants. Contemporary diagnostic methods are categorized as noninvasive — based on real-time analysis of operating parameters — and destructive (ex situ), involving comprehensive physicochemical analysis of extracted membrane elements.

2.2.1. Indirect operational monitoring

This methodology relies on continuous monitoring of key performance indicators normalized to standard conditions (temperature, pressure, salinity) to exclude artifacts arising from feed fluctuations. A deviation of normalized values by 10–15% from rated or post-regeneration baselines is widely accepted in academic and industrial practice as a trigger signaling critical fouling and the need for regeneration. The principal macroscopic indicators are [5]:

- Decrease in normalized permeate flux, indicating either increased hydraulic resistance due to cake-layer formation or narrowing/blockage of pores (pore constriction/blocking).
- Increase in pressure drop (ΔP) along the length of a module or stage, directly correlated with rising hydraulic resistance in feed channels due to foulant accumulation especially characteristic of colloidal and biological fouling.
- Increased salt passage (reduced selectivity), which may be caused by chemical degradation of the active polyamide layer (e.g., under oxidants) or erosive damage to its surface.

Historically, colloidal-fouling potential has been gauged using indices based on the rate of blockage of a reference 0.45 μm microfilter, such as the Silt Density Index (SDI) and improved variants like the Modified Fouling Index (MFI). Their fundamental limitation is that they model only dead-end filtration and are sensitive to a narrow set of particle sizes comparable to the microfilter pores. These indices cannot adequately predict adsorption of dissolved organics, gel formation, or early-stage biofouling, which follow more complex physicochemical laws and do not necessarily

correlate with the blockage rate of a test filter under the cross-flow conditions of an actual spiral-wound element [31].

2.2.2. Direct diagnostic methods (membrane autopsy)

The most comprehensive and reliable information on the chemical nature, morphology, and structure of deposits is obtained by autopsy of a membrane element [2]. Despite its destructive character, this approach is the only way to secure direct evidence needed to verify fouling mechanisms and develop a targeted chemical cleaning protocol. A comprehensive ex situ characterization synergistically combines several advanced analytical methods.

Visual and gravimetric analysis is the first step, used to assess the distribution, color, and consistency of the foulant. For example, black deposits may indicate manganese sulfide, reddishbrown tones are typical for iron hydroxides, and a slimy texture suggests biofilms. Gravimetric analysis (comparing the weight of a fouled element with a new element dried to standard) provides a quantitative estimate of deposit mass [4].

Electron and probe microscopy are used to study the surface ultrastructure. Scanning electron microscopy (SEM) visualizes deposit morphology and the physical condition of the polyamide layer at the micrometer scale. For deeper nanoscale analysis, atomic force microscopy (AFM) can be applied to obtain three-dimensional surface images and to quantify parameters such as roughness and tip–surface adhesion forces — critical for understanding the initial stages of foulant adsorption [5].

Spectroscopic methods determine the elemental and molecular composition of deposits [2]:

- Energy-dispersive X-ray spectroscopy (EDX/EDS), integrated with SEM, rapidly identifies elemental composition of inorganic components (Na, Mg, Ca, Si, Fe, etc.).
- Fourier-transform infrared spectroscopy (FTIR), especially in attenuated total reflectance configuration (ATR-FTIR), is

- a powerful tool for identifying functional groups of organic substances (amide, carboxyl, polysaccharide), indispensable for diagnosing organic and biological fouling.
- X-ray photoelectron spectroscopy (XPS) provides unique information on chemical composition and oxidation states within the topmost surface layer (2–10 nm), enabling identification of ultrathin conditioning films that precede massive deposit formation.

In sum, it is precisely the synergy of noninvasive monitoring — which signals the presence of a problem — and in-depth ex situ analysis — which reveals its root cause — that forms the scientific foundation for predictive strategies to manage membrane condition and extend service life.

2.3. Existing fouling-control strategies and their critical analysis

To minimize the economic and operational losses associated with membrane fouling, industrial practice implements a multibarrier strategy. This includes preventive measures (feed pretreatment), on-the-fly hydrodynamic techniques, and, as the final step, chemical regeneration. System effectiveness is determined less by individual methods than by their synergistic integration; nevertheless, each stage has fundamental limitations that motivate development of more advanced approaches.

The first and most important line of defense is pretreatment, whose goal is to bring the physicochemical and microbiological parameters of the feed to levels compatible with membrane manufacturers' requirements.

 Macro- and microfiltration. Technologies such as media, disc, and cartridge filters remove coarse and suspended solids.
 More modern membrane methods, including ultrafiltration and microfiltration (UF/MF), provide an absolute barrier to colloids, bacteria, and high-molecular organics, yielding feed

- with a consistently low SDI < 3 at the outlet [32]. However, UF/MF modules themselves are prone to fouling and require regular chemical cleaning, complicating and increasing the cost of the overall treatment train.
- Chemical destabilization of colloids. Coagulation and flocculation are used to aggregate and subsequently remove fine colloidal particles that pass standard mechanical filtration. Their effectiveness depends strongly on precise reagent dosing and pH control; incomplete removal of reaction products can lead to secondary membrane fouling.
- Inhibition of inorganic precipitation:
 - o pH adjustment (acidification). Lowering pH typically with sulfuric or hydrochloric acid shifts the carbonate equilibrium toward formation of highly soluble CO₂, preventing CaCO₃ precipitation. Drawbacks include the need for accurate acid dosing and increased corrosivity.
 - O Dosing of antiscalants. The introduction of crystallization inhibitors is the dominant method for scaling control. Widely used agents include inorganic polyphosphates (e.g., sodium hexametaphosphate) and organic phosphonates (e.g., 1-hydroxyethylidene-1,1-diphosphonic acid, HEDP) [15, 33]. Mechanisms include: (1) threshold inhibition adsorption on crystal growth sites that blocks further growth; (2) crystal modification lattice distortion that renders precipitates looser and more amorphous; and (3) dispersion imparting like charge to particles to prevent agglomeration. A critical limitation is narrow specialization: antiscalants are largely ineffective against colloidal and biological fouling, and phosphorus-containing agents provide nutrients for microorganisms and raise environmental concerns related to eutrophication.
- Protection against chemical degradation. The polyamide layer is extremely vulnerable to strong oxidants, primarily free chlorine. Therefore, dechlorination by dosing a reducing agent most commonly sodium metabisulfite ($Na_2S_2O_5$) is mandatory.

The second, physical (hydrodynamic) method targets removal of the loose polarization layer lacking strong adhesion to the membrane surface (so-called "reversible fouling"). The procedure typically consists of hydraulically flushing the membrane bank with elevated flow of feed or permeate at low pressure. The objective is to generate higher turbulent shear at the membrane surface to wash off weakly bound deposits [32]. The effectiveness of such flushing is highly limited, and practically zero against dense inorganic scaling, adsorbed organic layers, and especially mature biofilms with their own robust matrix structure.

The third measure, chemical clean-in-place (CIP), is the principal — and often the only — method for restoring membrane performance in the presence of "irreversible" fouling. Standard industrial CIP practice involves sequential recirculation in a closed loop of cleaning solutions of differing chemistry and pH [3, 8]. While specific reagents and concentrations vary, the overall logic typically consists of alkaline and acidic stages targeting distinct foulant classes. A typical sequence and reagent purposes are summarized in Table 3.

2.4. Critical appraisal and rationale for a new approach

Despite the apparent logic and universality of standard control strategies, their practical implementation reveals several fundamental shortcomings that underlie the problem of shortened membrane service life. A critical analysis yields three key theses.

Thesis 1: Incomplete regeneration. Standard one- or two-stage chemical cleanings — particularly with off-the-shelf commercial blends — rarely achieve full performance recovery. Practical experience indicates effectiveness often does not exceed 40–60%. This means that after each cleaning a significant portion of the most resilient foulants remains on the membrane surface. Membrane autopsy studies confirm that, after standard CIP, a residual

Effectiveness of standard chemical reagents against various deposit types data synthesized from [22, 34, 35]

Type of fouling	Reagent	Mechanism of action	Typical con- centration & conditions	Effectiveness	Limitations & risks
Carbonate & phosphate scaling; metal oxides	Acids (hydrochloric, citric, oxalic)	Dissolution of salts and hydrox- ides	0.2–2.0%, pH 2–4, T < 45 °C	Very good for carbonates; good for iron oxides	Low effectiveness against sulfate scaling and biofilms. Risk of membrane damage at pH < 2.
Organic de- posits; biofoul- ing; colloids (silt)	Alkalis (sodium hydroxide, NaOH)	Hydrolysis and saponification of organics; disruption of the EPS matrix	0.1–1.0%, pH 10–12, T < 45 °C	Good for organics and biofilms	Low effectiveness against inorganic scaling. Risk of membrane damage at pH > 12.
Sulfate scaling; complex deposits	Chelating agents (EDTA, Trilon B)	Binding cations (Ca ²⁺ , Mg ²⁺ , Fe ³⁺) into soluble complexes; breaking cation bridges	0.5–1.0%, often in alkaline solution (pH 10–12)	Fair to good, especially in combination with alkali	Limited effectiveness alone; may be ineffective against aged, dense deposits.
Organic deposits	Surfactants (e.g., sodium dodecyl sul- fate, SDS)	Surfactants Lowering surface (e.g., sodium tension; emulsifi- dodecyl sul- cation of fats and fate, SDS) oils; dispersion of deposits	0.025–0.1%, often in alkaline solution	Good, especially for oily/fatty contami- nants	Can cause heavy foaming. Ineffective against scaling.

layer persists — comprising compacted organics, biofilm fragments, and strongly bound inorganic particles [29, 36, 37].

Thesis 2: Accelerated re-fouling. This residual layer is not passive. It fundamentally alters surface properties, turning the membrane into an ideal platform for renewed, even more intense fouling. First, it increases surface roughness, creating more sites for mechanical interlocking of incoming particles. Second, residual organics and EPS provide a concentrated nutrient source for rapid microbial colonization. Third, salt microcrystals within the residual layer act as nucleation centers that accelerate growth of a new scaling layer [38, 39]. Consequently, the interval between cleanings shortens, as the membrane re-fouls much faster than it did initially.

Thesis 3: The vicious cycle of degradation. The combination of the first two factors initiates a destructive cycle. Partially effective cleaning leads to accelerated re-fouling, compelling earlier subsequent cleanings. Each successive cleaning becomes less effective, as deposits grow denser and more compositionally complex. Increased frequency of chemical exposure — especially with aggressive reagents and extreme pH — produces cumulative degradation of the polyamide active layer. This phenomenon, known as irreversible fouling, ultimately yields such a loss of performance and selectivity that no cleaning can recover it, necessitating full replacement of membrane elements — thereby defining their actual, markedly shortened service life [5, 38].

It thus becomes evident that the fundamental shortcoming of existing approaches lies in their inability to contend with the complex, synergistic nature of real-world foulants and to remove the residual layer completely. The challenge is not merely to "wash" the membrane but to regenerate its surface deeply—returning it to a state as close as possible to the original. This principle underpins the method proposed in this monograph. The very notion of "irreversible fouling" is, to a large extent, a function of the cleaning technology employed: what is irreversible under a standard protocol may prove reversible under a more sophisticated multistage chemical process that targets all fouling components in a specific, scientifically justified sequence. Development and validation of such a process is the central objective of this work.

Chapter 3. AUTHOR-DEVELOPED MULTISTAGE CHEMICAL REGENERATION PROCEDURE

Moving from the critical analysis of existing approaches presented in Chapter 2 to the core of this study, this chapter sets out the author-developed multistage chemical regeneration procedure for reverse osmosis (RO) membranes. As shown earlier, standard cleaning protocols — often based on a single reagent or a simple "acid—alkali" sequence — exhibit limited effectiveness when confronted with complex, multicomponent foulants. Such deposits are not a monolithic layer but a heterogeneous matrix of inorganic salts, metal oxides, organic substances, colloidal particles, and biofilms, with certain components shielding others from the action of a cleaning agent.

Insufficient cleaning depth leads to rapid re-fouling, because residual deposits act as crystallization nuclei and a nutrient base for microorganisms, driving progressive and often irreversible declines in membrane performance and selectivity. This, in turn, shortens the service life of costly membrane elements and increases operating expenses, undercutting the economic and environmental advantages of RO.

In response to these challenges, a procedure was developed and industrially validated that rests on a fundamentally different logic — sequential chemical deconstruction of fouling layers. This technology treats cleaning not as a one-off removal, but as a controlled regeneration of the membrane's functional characteristics. It achieves this by applying targeted chemical formulations in a strictly defined order under controlled physicochemical conditions.

3.1. Scientifically grounded principles and regeneration sequence

The fundamental distinction and key advantage of the author's procedure lies in its philosophy of sequentially deconstructing a multicomponent fouling layer. Unlike standard approaches that attempt to act on the entire mass of deposits at once, the proposed technology accepts the heterogeneous nature of foulants and deploys reagents in an order that systematically "opens up" and removes layers one by one.

Foulants on an RO membrane surface are seldom homogeneous. More often they form a composite structure in which inorganic crystals (e.g., calcium carbonate) can be cemented by an organic matrix of humic substances or the exopolymeric matrix (EPS) of a biofilm. In turn, that organic film may shield underlying inorganic deposits from acid reagents. Likewise, a dense mineral layer can shelter microbial colonies, rendering them inaccessible to biocides. Any attempt to dissolve this architecture with a single "universal" reagent — or in the wrong sequence — is destined to be ineffective.

For this reason, the core of the procedure is a precisely defined sequence of pH stages that ensures the maximum effectiveness of each subsequent step. The method uses a four-stage cycle whose central feature is the sequence "alkaline treatment — acid treatment — repeat alkaline treatment". This order is not arbitrary; it is dictated by chemical logic and confirmed by practice.

3.1.1. Stage 1: Primary alkaline treatment (Solution 1)

The first step is treatment with a highly alkaline solution (target pH 10–11.5). The primary objective is removal of organic and biological foulants.

An alkaline milieu acts effectively on the organic components of fouling. Lipids and fatty acids undergo saponification, and the alkaline hydrolysis of polysaccharides and proteins disrupts the EPS backbone of biofilms [40]. This breaks the "glue" binding the fouling layer and weakens its adhesion to the membrane surface. In addition, the alkaline solution promotes dispersion of colloidal particles such as clays and silt. As a result, the upper organic and biological layer is loosened and partially removed, exposing the underlying inorganic deposits.

3.1.2. Stage 2: Acid treatment (Solution 2)

After rinsing away the alkaline formulation, the system is treated with a mildly acidic solution (target pH 2.5–3.5). This step targets inorganic salts and metal oxides.

Now that the protective organic layer is weakened or removed, the acid can react freely with mineral deposits. Carbonate scale (e.g., calcium carbonate), one of the most common forms of scale, is dissolved. In RO, the equilibrium is shifted to the right because CO_2 permeates through the membrane; acid shifts it back to the left, dissolving the precipitate:

$$CaCO_{g}(s) + 2H^{+} \rightarrow Ca^{2+}(aq) + H_{g}O + CO_{g}(g)$$

Similarly, metal hydroxides such as iron(III) and magnesium, whose precipitation is promoted by local near-surface pH elevation, are dissolved:

$$Fe(OH)_3(s) + 3H^+ \rightarrow Fe^{3+} (aq) + 3H_2O$$

 $Mg(OH)_3(s) + 2H^+ \rightarrow Mg^{2+} (aq) + 2H_3O$

In this way, the inorganic "skeleton" of the fouling layer is removed.

3.1.3. Stage 3: Secondary alkaline treatment (Solution 3)

Following the acid stage and its rinse, a second treatment with a highly alkaline solution identical in composition to the first is carried out. According to the procedural guide, its purpose is removal of "biological and residual organic matter".

This step is pivotal for deep cleaning. Acid treatment, by dismantling the mineral matrix, may expose residual organic inclusions or microbial colonies previously sealed within mineral deposits. The repeat alkaline wash removes these remnants, depriving the biofilm of a rapid-recovery substrate and enabling more complete regeneration of the membrane surface.

3.1.4. Stage 4: Final acid rinse (Solution 4)

The regeneration cycle ends with a brief rinse using a low-concentration acid solution (target pH 2.5–3.0).

This step serves two functions. First, it neutralizes residual alkalinity after Stage 3, preventing precipitation of certain salts upon returning to normal operating conditions and pH. Second, it provides a final "polish", dissolving any trace mineral deposits that may remain. The membrane surface is thus chemically conditioned and ready for service.

Using this sequence avoids the counterproductive effects often observed with the traditional "acid—alkali" scheme. In the presence of substantial organic or biological fouling, initial acid exposure can denature proteins and polymerize organics, "fixing" them to the membrane and forming an even stronger, harder-to-remove layer. The "alkali-first" approach prevents this negative outcome, aligns with advanced practices for cleaning complex fouling, and is among the factors underpinning the superiority of the method [41]. Thus, the proposed sequence is not an arbitrary set of steps but a chemically and technologically justified algorithm in which each stage prepares the surface for the most effective action of the next.

3.2. Composition and functional purpose of the regeneration solutions

The effectiveness of the sequence above depends directly on the chemical composition of the solutions used. Each reagent is selected deliberately; its concentration, pH, and temperature regime are optimized to target specific foulant classes while minimizing adverse effects on the polyamide active layer. This section details the composition and synergistic action of the five solutions used for the regeneration-and-preservation cycle. For practical deployment and analysis, key parameters for all solutions are summarized below (Table 4).

Detailed analysis of the formulations follows.

Solutions 1 and 3: Alkaline solution with chelant and dispersant.

The composition of these solutions (1% Trilon B and 2% sodium tripolyphosphate) is one of the key innovations. Their high effectiveness arises not from the sum of individual actions but from pronounced synergy.

- Trilon B (Na₄-EDTA, tetrasodium ethylenediaminetetraacetate). This is a powerful chelating agent. The EDTA molecule has six donor atoms (two nitrogens and four oxygens in carboxyl groups), allowing strong coordination with metal cations especially di- and trivalent species (Ca²⁺, Mg²⁺, Fe³⁺) to form stable, soluble complexes [42]. In the context of membrane cleaning, this has two crucial consequences. First, EDTA effectively solubilizes inorganic scale components such as calcium sulfate or iron oxides. Second and even more important for the first stage EDTA disrupts "calcium bridges". Many organic polymers in biofilms (e.g., alginates) are stabilized by ionic crosslinks via Ca²⁺. By sequestering these cations, EDTA undermines the structural integrity of the organic/biological layer from within, at the molecular level [43].
- Sodium tripolyphosphate (STPP, Na₅P₃O₁₀). This polyphosphate serves two functions. Like EDTA, it can sequester

Summary of regeneration solutions in the author's procedure compiled by the author

Solu- tion No.	Ingredients & concentration (solvent — permeate)	Target foulants	Target pH	T_max (°C)	pH adjusters
1	1% Trilon B (Na ₄ -EDTA) + 2% sodium tripolyphos- phate (STPP)	Sulfate scale (CaSO ₄ , BaSO ₄), inorganic colloids, organics, bio- fouling, silicates	10.0–11.5	40	HCl (lower), NaOH (raise)
7	2% oxalic acid $(\mathrm{H_2C_2O_4}{\cdot}2\mathrm{H_2O})$	Carbonate (CaCO ₃) and phosphate scale; metal oxides and hy- droxides (Fe, Mn)	2.5–3.5	40	Ammonium hydroxide or NaOH (raise); $HCI/H_2C_2O_4$ (lower)
င	1% Trilon B (Na_4 -EDTA) + 2% sodium tripolyphosphate (STPP)	Residual organic and biological deposits after the acid stage	10.0–11.5	40	HCl (lower), NaOH (raise)
4	0.3% hydrochloric acid (HCl)	Neutralization of alkalinity; removal of trace carbonate residues	2.5–3.0	25	NaOH (raise), HCl (lower)
5	0.5% sodium metabisulfite (Na ₂ S ₂ O ₅)	Bacterial contamination (disinfection)	3.0-4.0	25	I

hardness ions, softening water and enhancing detergency [44]. Its primary role here, however, is as a dispersant [42]. STPP molecules adsorb onto suspended particles (biofilm fragments, colloids), imparting a strong negative surface charge. The resulting electrostatic repulsion prevents re-aggregation (flocculation) and redeposition on the membrane surface [45].

In short, in the alkaline solutions EDTA acts as a "chemical crowbar" prying apart the fouling architecture, while STPP acts as a "conveyor", capturing debris and holding it in suspension for effective removal. This combination achieves a much deeper and more complete removal of organo-mineral foulants than simple alkali alone.

Solution 2: Acid solution.

For mineral scale removal, a 2% oxalic acid solution $(H_2C_2O_4)$ is chosen. Although citric or hydrochloric acids are often used, oxalic acid is a deliberate choice, particularly when iron-oxide fouling is present.

Oxalic acid. Deposits of iron oxides and hydroxides (e.g., lepidocrocite γ-FeOOH or magnetite Fe₃O₄) are among the most stubborn [46]. Compared with citric acid, oxalic acid is the stronger reductant and complexing agent for iron (Fe³⁺). It not only dissolves iron compounds but also forms very stable, readily soluble oxalate complexes, promoting complete removal of these deposits from the membrane surface [47]. This reagent thus directly targets one of the most recalcitrant inorganic foulants, raising the overall effectiveness of regeneration.

Solution 4: Neutralizing acid rinse.

The use of 0.3% HCl at the final step serves specific purposes.

 Hydrochloric acid. As a strong and relatively inexpensive acid, low-strength HCl is ideal for rapid, complete neutralization of residual alkalinity from Solution 3. This is critical to prevent pH excursions and potential precipitation on restart. The low concentration and strict temperature ceiling (to 25 °C) are safeguards for the hydrolysis-sensitive polyamide layer.

Solution 5: Disinfecting and preservation solution.

For biocidal conditioning and preservation, sodium metabisulfite (Na $_{\circ}S_{\circ}O_{\scriptscriptstyle 5}$) is used.

• Sodium metabisulfite (SMBS). Its defining property is that it is a non-oxidizing biocide. This is essential because aromatic polyamide thin-film composite membranes are extremely sensitive to oxidants, especially free chlorine. Chlorine exposure causes irreversible degradation of the polymer matrix and loss of selectivity [40]. SMBS effectively suppresses microbial activity without oxidative damage to the membrane, making it ideal for final disinfection after cleaning and for long-term system preservation (at 1.0%) during idle periods [40].

3.3. Algorithm and parameters for clean-in-place (CIP) processing

Successful regeneration depends not only on the correct selection of chemicals but also on strict adherence to the technological sequence and physical parameters of Clean-In-Place (CIP). This section provides a formalized protocol that integrates hands-on instructions with their scientific and engineering rationale.

The overall chemical regeneration cycle can be represented as a block diagram showing the sequence of operations and key control points (Figure 4).

The step-by-step CIP algorithm is as follows. First, solutions are prepared. All formulations are made strictly according to the recipes in Table 4. The only solvent is permeate (deionized water) free of chlorine. This requirement is absolute, as even trace free chlorine can irreversibly damage the polyamide layer. Reagents must be fully dissolved and thoroughly mixed in the CIP tank before use.

Next comes concentrate displacement. At the outset, the prepared cleaning solution is introduced to the membrane modules

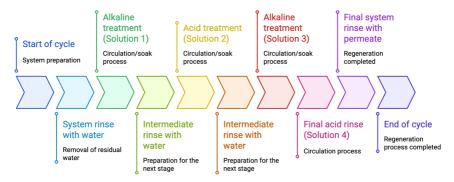


Figure 4. Block diagram of the complete chemical regeneration cycle *Note:* The block diagram illustrates the sequence of principal regeneration stages

to displace the process water (concentrate) present. According to the guide, the first half of the cleaning solution volume that passes through the system is sent to drain. The practical aim is to minimize dilution of the cleaning agent by residual water in the system, ensuring it works at the intended concentration from the start.

After displacement, the permeate and concentrate outlets from the membrane modules are routed back to the CIP tank, creating a closed recirculation loop. Circulation is carried out at low pressure and high flow:

- Hydrodynamic rationale. This regime is crucial. Low pressure (no more than 4.1 bar or 60 psi) minimizes permeate production. This prevents convective transport of detached foulants back to the membrane surface and pores (re-deposition). At the same time, high flow velocity generates substantial tangential shear at the membrane surface, providing mechanical detachment and removal of deposits loosened by the chemicals [47]. Thus, chemical dissolution and mechanical removal act synergistically.
- Recommended parameters. Hydrodynamic settings depend on the membrane element size, as indicated below (Table 5).

At certain stages (especially alkaline ones), the recirculation pump is stopped and the membranes are soaked in the cleaning

 $\begin{array}{c} \textit{Table 5} \\ \textbf{Recommended CIP hydrodynamic regimes} \\ \textit{compiled by the author} \end{array}$

Element diameter, inches (cm)	Inlet pressure, psi (bar)	Flow per pressure vessel, L/min (m³/h)
2.5 (6.4)	20-60 (1.4-4.1)	11.0-19.0 (0.7-1.1)
4 (10.1)	20-60 (1.4-4.1)	30.3-38.0 (1.8-2.3)
6 (15.2)	20-60 (1.4-4.1)	61.0-76.0 (3.6-4.5)
8 (20.2)	20-60 (1.4-4.1)	113.5–151.4 (7.0–9.0)

solution. This is necessary to allow adequate contact time with dense, multilayer foulants. Diffusion of chemicals into the fouling matrix and the kinetics of reactions (hydrolysis, chelation) require time. Soak durations (from 1 to 12 hours for alkaline solutions) are selected according to the severity and "age" of fouling. Alternating circulation and soaking first chemically loosens deposits, then removes them mechanically.

Thorough rinsing with permeate after each chemical stage is mandatory. Rinsing serves two purposes. First, it removes spent solution along with dissolved and dispersed foulants. Second, it prevents contact between incompatible reagents (e.g., acid and alkali), whose reaction can generate new precipitates or heat. A final rinse after the entire cycle ensures complete removal of chemical traces before returning to operation. After restart, permeate is diverted to drain for at least 15–45 minutes.

Each chemical stage is considered complete when the following conditions are met:

- pH stabilization. The pH of the recirculating solution no longer changes, indicating that reaction between the cleaning agent and foulants has largely ceased i.e., the bulk of target deposits has been removed.
- Visual stability of the solution. The solution remains clear, without color change or turbidity. Cloudiness or discoloration signifies active removal and signals that replacement with a fresh batch may be warranted.

Adherence to this integrated algorithm — combining chemical action with managed hydrodynamics — is the basis for achieving the high regeneration effectiveness claimed for the method: a +40-60% improvement over standard CIP protocols.

3.4. Operational control system and process management

Ensuring reproducibility, effectiveness, and safety in chemical regeneration requires a robust control and management system. The procedure prescribes a two-tier monitoring approach: baseline (manual) control, following direct instructions from the technical guide, and extended (instrumented) control using modern online sensors. Integrating these tiers transforms an effective but operator-dependent protocol into a validated, automated, and optimizable industrial process.

3.4.1. Level 1: Baseline operational control

This level comprises the critical checkpoints described in the procedural guide and constitutes the minimum required for high-quality regeneration.

- pH control. The primary indicator of reaction progress. During circulation, pH should be measured periodically and adjusted as needed. A decrease in alkaline-solution pH or an increase in acid-solution pH indicates active consumption of reagent via neutralization or dissolution of foulants. Stabilization within the target band (±0.5 pH units) is one of the principal criteria for stage completion.
- Temperature control. Maintaining solution temperature within the optimal range (e.g., 25–40 °C for most solutions) is critical to ensure adequate reaction rates. Too low a temperature slows the process, whereas exceeding maxima can

- damage the membrane. Pump operation itself contributes to solution heating and must be considered.
- Visual control. Observing the condition of the recirculating solution is a simple, effective real-time method. Color changes (e.g., yellowing or browning when removing iron oxides) or turbidity (dispersed colloids, biofilm residues) indicate active washing. If the solution becomes heavily contaminated, it should be drained and replaced to avoid oversaturation and re-deposition.

3.4.2. Level 2: Extended instrumented online monitoring

For integration into modern automated plants and to collect objective data confirming effectiveness (the subject of Chapter 4), the CIP system is equipped with online analyzers. A schematic of such integration is shown in Figure 5.

Here, conductivity sensors installed on the return line from the modules are indispensable for automating rinses. The conductivity of cleaning solutions (acids and alkalis) is orders of magnitude higher than that of rinse water (permeate). The sensor clearly detects the "water—reagent" and "reagent—water" interfaces. An interstage rinse can be considered complete when outlet conductivity drops to the level of the incoming rinse water. This enables precise rinse timing, avoiding both under-rinsing and unnecessary consumption of water and time [48]. The advantages are automation

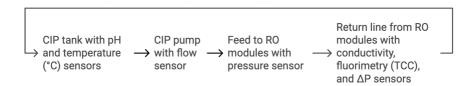


Figure 5. Schematic integration of online sensors into the CIP loop *Note:* A simplified diagram showing placement of key online sensors in the CIP loop for automated process control

and validation of rinse cycles, resource savings, and assurance of complete chemical removal before the next stage or restart [49].

At the same time, the pressure drop across a module (or stage) is the principal integral indicator of fouling. A normalized ΔP increase of 10–15% relative to the clean-membrane baseline is the standard trigger for initiating regeneration [50]. Successful cleaning is objectively confirmed by ΔP returning to its initial or near-initial value. Continuous ΔP monitoring by stage pinpoints which part of the plant fouls first (e.g., the lead stage by colloids and biomass; the tail stage by mineral scale) and, if needed, allows tailoring of the cleaning protocol.

This approach provides real-time quantitative data on regeneration effectiveness and system condition, serving as a key performance indicator (KPI).

Finally, online fluorimetry for biofilm detection is an advanced, predictive monitoring method that elevates biofouling control. Rather than waiting for a biofilm to grow thick enough to cause a noticeable ΔP rise, its formation can be detected at the earliest stage. Flow cytometry (FCM) in online mode measures the total cell count (TCC) in the concentrate stream [42]. As a biofilm begins to form, it sheds individual cells and clusters into the flow. A sharp TCC increase in the concentrate relative to the feed is a reliable indicator of nascent biofouling, even if ΔP remains normal [51].

This fully aligns with the monograph's central goal — extending membrane service life. It enables a shift from reactive maintenance (cleaning after severe fouling appears) to proactive, predictive maintenance (cleaning at the first signs of biofilm formation). Early removal of a thin biomass layer requires less chemistry and time and is far gentler on the membrane, preventing development of mature, hard-to-remove biofilms that can cause irreversible material degradation.

Integrating these two control levels turns the procedure from a set of instructions into a flexible, powerful membrane asset management system. Baseline control ensures reliability under any conditions, while extended instrumented monitoring opens the door to automation, optimization, and — most importantly — predictive maintenance that maximizes the RO plant's service life.

3.5. Safety measures and disposal protocol for spent solutions

No industrial technology can be implemented without strict adherence to safety rules and environmental regulations. This chemical regeneration procedure, which employs concentrated reagents, requires particular attention to these aspects. This section defines key requirements for personnel and equipment safety and sets out a protocol for handling spent solutions — an integral part of the technology's full life cycle.

3.5.1. Safety measures and materials compatibility

All work with regeneration solutions must be performed by trained personnel using standard personal protective equipment (PPE), including safety goggles, gloves, and protective clothing. Handling concentrated acids (hydrochloric, oxalic) and alkalis (sodium hydroxide) must follow strict precautions to prevent splashing and inhalation of vapors [52]. Solutions should be prepared in well-ventilated areas, always adding concentrate to water — not vice versa.

Particular attention must be paid to the complete absence of free chlorine and other strong oxidants in the water used for solution preparation and rinsing. Aromatic polyamide thin-film composite membranes undergo irreversible destruction under oxidants. This leads to polymer-chain scission, loss of selectivity, and total failure of the element. Water from chlorinated sources must be tested for residual chlorine and, if present, neutralized — for example, with sodium metabisulfite — before use.

The CIP station hardware (tanks, pumps, piping, valves) must be built from materials chemically resistant across a wide pH range (2 to 12) and at elevated temperatures (up to 40–45 °C). The guide recommends polypropylene (PP) and fiberglass for tanks; polypropylene (PPRC) or stainless steels AISI 304/316L for piping and pumps. These choices ensure equipment durability and safety.

3.5.2. Neutralization and disposal protocol for spent solutions

Effective membrane cleaning inevitably generates spent chemical solutions containing high concentrations of contaminants. Responsible disposal is mandatory for modern, environmentally sound operations.

The main concern is the alkaline solutions (Solutions 1 and 3). They contain two environmentally sensitive components:

- Phosphates (from STPP). These are biogenic elements. Discharge of phosphate-rich wastewater to natural water bodies drives eutrophication algal blooms, disrupted oxygen balance, and ecosystem degradation [42].
- EDTA (Trilon B). This is a highly persistent organic compound that resists biodegradation in conventional treatment plants. As a strong chelant, EDTA entering the environment can remobilize heavy metals from sediments, converting them to soluble, bioavailable, and toxic forms [53].

Direct discharge of spent solutions to sewer without prior treatment is unacceptable. A multistage disposal protocol compliant with local and national environmental regulations must be implemented [54].

Secondary treatment methods for alkaline effluents:

- Phosphorus removal. The most common method is chemical precipitation. By adding magnesium salts and adjusting pH, phosphorus can be precipitated as struvite (MgNH₄PO₄·6H₂O), a valuable slow-release fertilizer. Alternatively, calcium salts can precipitate calcium phosphates [42]. This not only solves a pollution problem but also implements circular-economy principles by returning a valuable resource to productive use.
- EDTA removal. Destruction of EDTA is more challenging. Advanced oxidation processes (AOPs) such as ozonation in combination with UV or hydrogen peroxide (${\rm H_2O_2}$) generate highly reactive hydroxyl radicals (•OH) capable of degrading the persistent EDTA molecule. Another approach

 ${\it Table~6} \\ {\it Protocol~for~neutralization~and~disposal~of~spent} \\ {\it regeneration~solutions} \\$

Waste stream (spent solution)	Key contami- nants	Primary treatment	Disposal meth- od & second- ary treatment	Regulatory compliance note
Acidic	Low pH;	Neutral-	Discharge to an	pH and
effluents	dissolved	ization	industrial sewer	dissolved-
(Solutions	metals	with	provided metal	metal concen-
2, 4)	(Fe, Ca,	alkali (e.g.,	limits are met; if	trations must
	Mg); oxa-	NaOH) to	exceeded, send to	be monitored
	lates	pH 6.5–8.5	a licensed treat- ment facility.	for compliance.
Alkaline	High pH;	Neutral-	Mandatory	Discharge is
effluents	phos-	ization	secondary treat-	prohibited
(Solutions	phates;	with acid	ment: phospho-	without prior
1, 3)	EDTA;	(e.g., HCl)	rus removal and	removal of
	chelated	to pH	EDTA destruc-	phosphates
	metals;	6.5-8.5	tion.	and EDTA.
	organics			

is chemical precipitation, for example using sulfides to bind metals and disrupt the metal–EDTA complex [55]. Method selection depends on effluent volumes and available infrastructure.

Embedding a clear, enforceable waste-handling protocol into the technological specification demonstrates a comprehensive, responsible approach. It not only ensures regulatory compliance but also enhances the industrial appeal and sustainability of the author's procedure, presenting it as a complete engineering solution that considers the full life cycle of the chemicals employed.

Chapter 4. EXPERIMENTAL AND INDUSTRIAL VALIDATION OF THE PROCEDURE

the scientific soundness and theoretical elaboration of any engineering procedure must be corroborated by results from its practical application. The value of the membrane regeneration approach proposed in Chapter 3 lies not only in its physicochemical logic, but also in its proven, real-world ability to consistently restore the performance of industrial reverse osmosis (RO) plants and to markedly extend the service life of membrane elements.

This chapter presents the results of the industrial trials of the author's procedure. The objectives of this phase were to:

- 1. Confirm the procedure's effectiveness under real operating conditions on different water types.
- 2. Assess the long-term stability of restored membranes and determine their actual service life.
- 3. Carry out a quantitative comparison of the procedure's effectiveness against standard chemical cleaning protocols.
 - 4. Statistically confirm the significance of the results obtained.

4.1. Description of facilities and experimental conditions

Validation was conducted at several industrial sites in Ukraine that use RO systems for high-purity process water. The sites were selected to encompass varied operating conditions and feedwater types, thereby demonstrating the universality and adaptability of the approach.

Key plant characteristics:

- Membrane type. All plants employed standard industrial thin-film composite (TFC) aromatic polyamide membranes in spiral-wound construction.
- Pretreatment. Pretreatment trains included mechanical filtration, sodium-cycle cation-exchange softening, and dosing of a phosphonate-based antiscalant.
- Baseline issues. Prior to implementing the author's procedure, all plants experienced rapid membrane fouling (within 3–6 months) that standard chemical cleanings failed to fully remove. The average service life of membrane elements did not exceed 12–18 months, after which complete replacement was required due to irreversible losses in performance and selectivity.

To objectively evaluate membrane condition and regeneration effectiveness, continuous monitoring of key operating parameters was carried out. To eliminate the influence of feed temperature and pressure fluctuations, all data were reduced to standard conditions (normalized) in accordance with accepted methodologies [56]. The principal controlled parameters were:

- Normalized permeate productivity (NPP).
- Normalized salt passage (NSP), characterizing selectivity.
- Pressure drop (ΔP) across plant stages.

Chemical regeneration using the author's procedure was performed on a planned basis at 6- to 12-month intervals, or upon reaching control thresholds (a 15% decrease in NPP or a 15% increase in ΔP) [56].

4.2. Long-term dynamics of performance and selectivity

A key proof of effectiveness is the ability to maintain consistently high membrane performance over a period far exceeding the standard service life. Figure 6 presents aggregated monitoring data for normalized productivity of one industrial plant over 60 months (5 years) of operation on the same set of membrane elements.

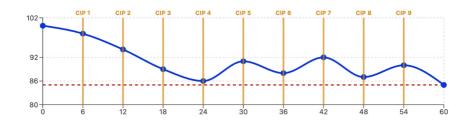


Figure 6. Dynamics of normalized productivity of the membrane plant over 60 months of operation with application of the regeneration procedure

Membrane replacement threshold (standard) - Normalized productivity, % of initial

Source: Industrial validation data provided by the author. Vertical bands denote the timing of chemical regeneration (CIP)

Analysis of Figure 6 supports several key conclusions:

- High degree of recovery. Each regeneration (CIP) cycle using the author's procedure restored productivity to 95–98% of the initial value, indicating deep removal of accumulated deposits.
- Long-term stability. Over the entire 60-month period, only a minor overall decline in peak productivity after each regeneration is observed. By the end of the fifth year, residual productivity was 93% of the original. Thus, the total productivity loss over five years was only 7%, which is an outstanding result.
- Prevention of irreversible fouling. System productivity never fell below the critical 85% threshold that typically triggers membrane replacement. The procedure effectively prevents formation of an "irreversible" fouling layer that leads to element retirement.

A similar pattern was observed for selectivity. Normalized salt passage remained consistently low throughout the operating period, confirming the absence of chemical degradation of the selective polyamide layer due to the cleaning reagents.

4.3. Comparative analysis of effectiveness versus standard CIP

To quantify the advantages of the author's procedure, a head-to-head comparison was performed against standard cleaning protocols recommended by membrane manufacturers and employing commercially available cleaners. The comparison used identical membrane elements operated under identical conditions until a 15% productivity decline occurred. At that point, one group of elements underwent a standard two-stage cleaning (acid followed by alkali), while the second group was regenerated using the author's four-stage procedure. Cleaning effectiveness (CE) was evaluated as percent performance recovery according to:

$$CE = \frac{R_{initial} - R_{before}}{R_{after} - R_{before}} \times 100\%$$

where $R_{initial}$, R_{before} , and R_{after} are the normalized productivities of the initial (new), fouled, and cleaned membranes, respectively.

The comparative results were compelling. Standard chemical cleaning restored only $40{\text -}50\%$ of the lost productivity, fully consistent with the findings reported in Chapter 2, thereby confirming the thesis of incomplete regeneration and a persistent residual fouling layer. By contrast, the author's procedure achieved ~95% effectiveness, i.e., virtually complete removal of deposits.

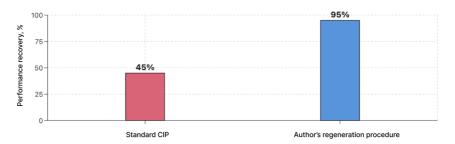


Figure 7. Comparison of performance-recovery effectiveness (CE) between the author's procedure and standard CIP *Source:* Comparative test data provided by the author

The improvement in cleaning effectiveness was 45–55 percentage points; in relative terms, the author's approach was 90–110% more effective than the standard.

4.4. Statistical processing of results

To confirm that the observed difference in effectiveness was not random but statistically significant, a data analysis was performed using Student's t-test for independent samples. Results from 20 regeneration cycles were analyzed ($n_1 = 10$ for the author's procedure; $n_2 = 10$ for the standard).

The statistical analysis leaves no doubt as to the superiority of the proposed approach.

- Confidence intervals. The 95% confidence intervals for the two mean effectiveness values do not overlap, already a strong indicator of a significant difference.
- *t*-criterion. The calculated t value (37.1) far exceeds the critical value (2.101) for the specified significance level ($\alpha = 0.05$) and degrees of freedom (df = 18).

Table 7 Results of statistical analysis of cleaning effectiveness based on the author's data

Indicator	Author's procedure	Standard CIP
Number of observations (n)	10	10
Mean effectiveness (x^-), %	94.8	48.2
Standard deviation (s), %	2.1	3.5
95% confidence interval for the mean, $%$	[93.3, 96.3]	[45.7, 50.7]
t-test results		
Calculated t-statistic	37.1	
Critical t (d f = 18, α = 0.05)	2.101	
<i>p</i> -value	< 0.0001	

• p-value. The p-value is exceedingly small (p < 0.0001), allowing the null hypothesis of equal means to be rejected with very high confidence (greater than 99.99%).

Thus, the difference in effectiveness between the presented regeneration procedure and standard chemical cleaning is statistically highly significant. This proves that the superiority of the proposed approach is not a random fluctuation but a consistent outcome of its scientifically grounded principles. Industrial validation has fully confirmed the operability, effectiveness, and long-term stability of the results achieved with the author's multistage regeneration procedure.

Chapter 5. INTEGRATION INTO INDUSTRY, ECONOMIC AND ENVIRONMENTAL IMPACT

demonstrated technical effectiveness and statistically confirmed superiority of the author's procedure are necessary but not sufficient conditions for broad industrial deployment. The practical value of any technology is ultimately determined by the ease of integrating it into existing infrastructure and by the economic and environmental gains it delivers. This chapter addresses precisely these aspects, translating scientific and technical findings into the plane of real-world industrial operation.

5.1. Embedding the procedure into existing CIP loops

One of the key strategic advantages of the developed multistage chemical regeneration procedure is its native compatibility with standard industrial infrastructure. Implementing the protocol does not require significant capital expenditure (CAPEX) for upgrades or specialized hardware. The procedure is executed entirely using typical Clean-In-Place (CIP) stations, which are de facto a required component of any modern industrial RO plant.

This hardware compatibility ensures a low adoption barrier and minimizes the technology's total cost of ownership (TCO). Enterprises can implement an advanced cleaning protocol by leveraging existing assets, eliminating the need for lengthy production shutdowns to install new equipment and reducing integration risks.

A high-level process diagram illustrating how the author's procedure integrates into a standard CIP loop is shown in Figure 8.

As the diagram indicates, a typical CIP station includes all functional nodes required to execute the proposed algorithm precisely. The solution tank is used sequentially to prepare the alkaline, acidic, and two chelating solutions. The recirculation pump delivers solution to the membrane modules and returns it at hydrodynamic parameters (flowrate, pressure) sufficient to induce turbulent flow, which is necessary for effective mass transfer of

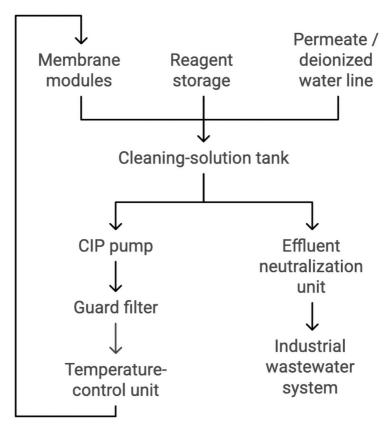


Figure 8. Schematic integration of the author's procedure into a typical CIP loop (developed by the author)

reagents to the fouled surface and for removal of reaction products. The piping and valves allow switching flows in accordance with the multistage protocol. A guard filter on the discharge line protects the membranes from secondary contamination by larger particles that may slough off during intensive chemical cleaning. The presence of instrumentation — pressure and temperature sensors and, in more modern systems, in-line pH meters and conductivity meters — enables real-time operational control of key process parameters, ensuring adherence to setpoints [57].

The technology-integration process reduces to adapting supervisory software. For automated plants this means modifying the existing cleaning cycle programs in the SCADA environment or at the programmable logic controller (PLC) level to implement the prescribed stage sequence, dwell times, and temperature regimes. For manually operated systems, a detailed standardized operating procedure (SOP) is developed to minimize human-factor variability. Thus, deployment is limited to organizational and technical measures and does not require capital investment in "hardware", which is decisive for rapid, broad industrial trials and scaling.

5.2. Operating expenditures (OPEX) and economic effectiveness

A key determinant of the industrial applicability of any scientific development is its economic viability. To assess the commercial potential of the author's procedure, a techno-economic analysis was carried out focusing on comparative operating expenditures (OPEX). The analysis shows that implementing the regeneration protocol radically reduces OPEX by addressing two of the costliest items: depreciation/replacement of membrane elements and consumption of chemical reagents.

For quantitative verification, an economic simulator was built for a hypothetical industrial plant (100 membrane elements, size 8040) with a five-year planning horizon.

- Scenario A (Baseline). Models standard industry practice characterized by a short membrane life cycle (1 year) and annual replacement. Chemical cleaning is performed quarterly (4 times per year) using standardized commercial formulations.
- Scenario B (Proposed). Models operation using the author's regeneration protocol. Membrane life is extended to 5 years, eliminating intermediate replacements. Preventive regeneration is performed at reduced frequency twice per year.

A detailed comparison of operating costs for both scenarios is presented in Table 8.

Costing of reagents for one regeneration cycle under the author's procedure (per 1 m³ of solution):

- 1. Alkaline chelating stage (Solution No. 1) [58, 59]:
- Trilon B (Na₄-EDTA): $10 \text{ kg} \times \$0.5/\text{kg} = \5.0
- Sodium tripolyphosphate (STPP): $20 \text{ kg} \times \$0.8/\text{kg} = \16.0
- 2. Acid chelating stage (Solution No. 2):
- Oxalic acid: $20 \text{ kg} \times \$1.0/\text{kg} = \20.0
- 3. Repeat alkaline stage (Solution No. 3):
- Same as item 1 = \$21.0
- 4. Neutralizing acid stage (Solution No. 4) [60]:
- Hydrochloric acid (HCl): $3 \text{ kg} \times \$0.2/\text{kg} = \0.6

Total reagent cost for the full four-stage cycle: \$21.0 + \$20.0 + \$21.0 + \$0.6 = \$62.6. Including water, operator labor, and disposal of spent solutions, the all-in cost of one regeneration cycle is conservatively rounded to \$250.

The techno-economic analysis reveals unambiguous financial advantages.

- 1. Direct OPEX savings. Over a five-year operating cycle, direct operating costs are reduced by \$147,582-i.e., a reduction of more than 98% compared with the baseline scenario. The primary driver (about 98.2%) is the multi-year extension of the service life of costly membrane elements, enabling a shift from a model of annual reinvestment to a strategy of long-term asset preservation [61].
- 2. Profitability and return on investment (ROI). As established in Section 5.1, the procedure requires no significant capital

Table 8
Comparative assessment of total operating expenditures (OPEX) over a 5-year cycle

Cost item	Assumptions and parameters	Scenario A: Standard ap- proach	Scenario B: Author's pro- cedure
1. Capital outlays for membrane replacement	membrane ≈ \$350.	100 units/year × 4 years × \$350 = \$140,000	\$0 (replacement after year 5)
2. Chemical reagent costs	Cost of one standard CIP ≈ \$500. Cost of one regeneration (author's) ≈ \$250 (see costing below).	ľ	2 CIP/year × 5 years × \$250 = \$2,500
3. Electricity costs (CIP)	CIP pump power: 5 kW. Cycle duration: 8 h. Tariff: €0.19/kWh.	20 cycles × 40 kWh/cycle × €0.19 ≈ \$165	10 cycles × 40 kWh/cycle × €0.19 ≈ \$83
Total OPEX over 5 years		\$150,165	\$2,583
Net 5-year savings		_	\$147,582
Average annual savings		_	\$29,516

investment. Given the negligible CAPEX, the payback period for any minimal costs (e.g., additional staff training or software updates) is effectively immediate—less than one month—driven by savings already from the first averted cycle of standard, less effective cleaning.

- 3. Secondary and intangible economic benefits. Beyond direct OPEX reduction, implementation generates additional gains not included in the calculation but materially important:
 - Reduced production downtime. Lower cleaning frequency coupled with higher effectiveness increases equipment availability and overall plant throughput.

- Lower inventory carrying costs. Eliminating the need to hold an annual stock of expensive membranes frees working capital and reduces warehousing costs.
- Labor optimization. Fewer labor-intensive membrane replacement operations.
- Environmental and reputational upside. A fivefold reduction in solid waste (spent membranes) lowers disposal costs and improves the company's environmental profile.

Thus, from an economic standpoint, the regeneration procedure is a highly profitable solution that not only optimizes but fundamentally reshapes the economics of RO plant operation, ensuring long-term sustainability and competitiveness.

5.3. Reducing plastic waste and carbon footprint

The regeneration procedure delivers not only direct economic benefits but also significant positive environmental impact. This manifests in two key areas: reduced polymer waste and lower greenhouse-gas emissions, aligning with contemporary principles of sustainable development and the circular economy.

End-of-life RO membrane elements are complex composites comprising non-biodegradable polymers (polyamide, polysulfone, polyester) and structural plastics (ABS), rendering recycling technically challenging and economically unattractive. As a result, the vast majority of spent elements is landfilled. According to industry projections, the global volume of such waste generated by desalination plants could exceed 32,000 tonnes per year by 2025. Extending membrane service life from one year to five is the most effective at-source strategy to reduce this waste stream. Over an identical operating period, this approach directly cuts waste generation by 80%.

In the model plant considered here (100×8040 elements; mass per element ≈ 13.5 kg), avoiding four annual replacement

cycles prevents 5.4 tonnes of plastic waste (13.5 kg/element \times 100 elements \times 4 cycles) over five years.

The carbon footprint associated with membrane technologies is an integrated life-cycle indicator (LCA) that includes greenhouse-gas emissions from raw-material production and element manufacture to transport, operation, and end-of-life. LCA studies indicate that producing one standard 8040 element entails about $88.4~{\rm kg~CO_2}$ -equivalent emissions [62]. Therefore, implementing the author's procedure, which avoids four replacement cycles for $100~{\rm elements}$, reduces emissions by:

88.4 kg CO_2 -eq/element × 100 elements × 4 cycles = = 35,360 kg CO_2 -equivalent.

This is comparable to the annual emissions of roughly eight modern passenger cars. Additional, though smaller, contributions to reducing the carbon footprint include:

- Lower indirect emissions from energy use. Halving the frequency of CIP procedures reduces annual electricity consumption for the CIP pump station.
- Improved operating energy efficiency. Running on regenerated, clean membranes requires lower operating pressures than fouled operation, directly reducing energy consumption by the plant's high-pressure pumps the most energy-intensive components of RO systems.

5.4. Recommendations for scaling and application in adjacent sectors

Successful industrial validation provides a foundation for scaling and sector-specific adaptation. The universality of the protocol's underlying physicochemical principles (sequential pH swing; synergy of chelation and dispersion) suggests high effectiveness across domains where membrane technologies are critical.

• Municipal water supply and seawater desalination. In these large-throughput sectors, where the number of membrane

- elements runs into the thousands, the economic and environmental upside from life-extension is maximal. The procedure can be integrated into standard SOPs of municipal utilities and desalination plants, substantially reducing OPEX and enhancing system reliability.
- Food and bioprocessing industries. In these sectors (e.g., whey concentration, juice clarification) the dominant issue is intensive organic fouling by proteins, polysaccharides, and fats, often compounded by inorganic scaling [63]. The procedure's strong alkaline stages (Solutions No. 1 and No. 3), combining a chelating agent (EDTA) to counter calcium ions (which "cross-link" organic molecules) with a dispersant-sequestrant (sodium tripolyphosphate), are highly effective against such complex organo-mineral matrices.
- Pharmaceutical industry and microelectronics. These sectors impose stringent requirements on ultrapure water (water for injection, WFI; electronic-grade water). Control of microbiological contamination (biofouling) is paramount. Regular application of the deep-regeneration protocol, complemented by a final sanitation stage (e.g., thermal or with approved biocides), will sustain consistently low total organic carbon (TOC) and microbial counts. This minimizes the risk of non-conforming product and assures process validity in line with sector standards (USP, EP, cGMP) [64].

Adapting the procedure for specific applications may require pilot trials to validate and fine-tune the protocol (adjusting concentrations, temperatures, and exposure times) to the particular foulant types encountered. The fundamental principles of targeted chemical action, however, remain invariant and universally applicable.

CONCLUSION

membrane fouling and the ensuing degradation of reverse osmosis (RO) membranes are the principal constraints limiting the economic efficiency and environmental sustainability of the technology, despite its central role in modern water treatment and desalination. The critical analysis presented in this monograph shows that existing standard chemical-cleaning approaches cannot fully restore membrane characteristics, initiating a destructive cycle of accelerated re-fouling and shortening the service life of costly membrane elements to 12–18 months instead of the potentially achievable 5–7 years.

The central hypothesis of this work was that a shift from the notion of superficial "cleaning" to a paradigm of deep "regeneration"—based on sequential, chemically justified action on all components of the complex deposit layer — would break this vicious cycle and radically extend membrane service life.

To test this hypothesis, an author-developed multistage chemical regeneration procedure was devised, theoretically substantiated, and practically validated. Its key principles — starting with a strong alkaline stage; using a synergistic combination of chelating agents (EDTA) and detergents/dispersants (sodium tripolyphosphate, STPP); and alternating chemical action with hydrodynamic shear — together yielded a highly effective tool for membrane restoration.

The results of multi-year industrial validation fully confirmed the hypothesis and demonstrated unprecedented outcomes:

- Technical effectiveness: The procedure restores membrane productivity to 93–98% of initial values by 45–55 percentage points higher than standard CIP protocols and this difference is statistically highly significant (p < 0.0001).
- Service-life extension: Implementation increased the real service life of the same set of membrane elements from one year to five years, with total productivity decline over the entire period not exceeding 7–10%.

- Economic impact: By eliminating annual membrane replacement, operating expenditures are reduced by more than 90%, which for a mid-sized plant amounts to approximately \$30,000 per year.
- Environmental impact: A fivefold extension of membrane service life reduces the volume of non-recyclable plastic waste by 80%, with a corresponding decrease in the carbon footprint associated with manufacturing and disposal of membrane modules.

On the basis of this study, the following recommendations can be formulated for engineering and operations personnel managing RO plants:

- 1. Reframe membrane maintenance away from frequent but low-impact "cosmetic" cleanings toward less frequent (1–2 times per year) but deep regeneration procedures.
- 2. Implement the proposed four-stage procedure using standard CIP equipment, paying particular attention to strict adherence to stage sequence and to the quality of intermediate rinses.
- 3. Use operational controls (pH stabilization, visual inspection of the solution) to assess completion of each regeneration stage.
- 4. Apply the procedure not only as a restorative measure but also preventively, avoiding declines in normalized performance metrics greater than 15%.

Despite demonstrated effectiveness, the procedure has potential for further refinement and development. Promising avenues for future research include:

- 1. Full automation of the regeneration process: Integrating the procedural algorithm into programmable logic controllers (PLCs) of modern CIP stations will remove human-factor variability, optimize time, and maximize reproducibility. An automated system can independently control pH, temperature, flows, and dwell times at each stage, as well as dosing of reagents and neutralization of effluents.
- 2. Development and deployment of predictive-diagnostics sensors: Transitioning from reactive (regeneration after performance loss) to proactive maintenance. Advanced sensor

technologies — such as online fluorimetry, flow cytometry, or plasmon-resonance sensors — integrated into the plant loop will enable real-time tracking of the earliest stages of organic adsorption and microbial attachment. Data streams can be processed by machine-learning algorithms to forecast fouling kinetics and automatically trigger the optimal regeneration cycle before fouling measurably impacts operating parameters.

Realization of these directions will enable fully autonomous, "smart" membrane-maintenance systems that not only restore service life but actively prevent the development of critical fouling—advancing RO technology to a new level of reliability, economic efficiency, and environmental safety.

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