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BIO-INSPIRED SELF-HEALING POLYELECTROLYTE MEMBRANES FOR SUSTAINABLE DESALINATION AND ION SEPARATION: A COMPREHENSIVE REVIEW

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Abstract:
Advancements in smart separation systems have resulted in long-lasting membranes for desalination and water treatment. Unfortunately, polymeric membranes are still susceptible to gradual physical and chemical degradation, which can compromise their separation performance. In nature, organisms have developed strategies to repair damage and ensure survival. The discovery of bio-inspired self-healing polymers has impacted diverse research fields, including polymer science, nanotechnology and biomedicine. This novel approach enables polymeric membranes to autonomously recover and restore lost functionality and therefore offers a viable pathway to improving sustainability in water treatment applications [1,2]. Fundamental research can be carried out to ascertain whether several intelligent self-healing mechanisms can be controlled to produce membranes for water treatment that can recover ion selectivity as well as their inherent mechanical properties after severe physical and chemical degradation. Employing bio-inspired self-healing mechanisms in polyelectrolyte separation membranes can effectively:

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1. Provide strong water and salt repellent properties
2. Induce, maintain and repair their structure and functionality
3. Avoid deactivation and excessive hysteresis during polymer swelling-shrinking cycles

In bio-inspired self-healing membranes, the integration of dynamic covalent bond-forming and -breaking mechanisms (e.g., microcapsule-based, dynamic covalent, supramolecular coordination) is typically adapted from catechol-metal coordination chemistry in mussel adhesive proteins. Several robust fabrications approaches (layer-by-layer assembly, etc.) can easily be employed to develop membranes with repairable functional chemistries. [10,12,18]

The self-healing membranes recover their original performance with healing efficiency >94% for ion selectivity and ion transport mechanisms (i.e., Donnan exclusion, steric hindrance, dielectric exclusion). These mechanisms provide a complementary platform to exercise tight control over key separation performance parameters. However, limitations (e.g., leaching-off of metal ions (repairing units), weak stability constraints) that detract from their physical and functional longevity.The current design guidelines toward enhancement of the new generations of self-healing membranes for scalable industrial application are motivating in the analysis of their post-fabrication corrosion as well as the development of new chemical strategies to engineer sophisticated multifunctional and autonomous separation membranes. [18,23,24]

Keywords: Self-healing membranes; Polyelectrolyte multilayers; Catechol chemistry; Layer-by-layer assembly; Ion separation; Nanofiltration; Review.

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1. Introduction

Membrane-based separation processes, particularly nanofiltration and reverse osmosis, are critical components of the global strategies to mitigate increasing freshwater scarcity. While thin-film composite polyamide membranes have been the workhorse of membrane technology for decades due to their excellent initial separation performance, they are highly susceptible to physical degradation manifested in defect creation via scratch propagation and delamination as well as chemical degradation from chlorine attacks under manufacturing, cleaning, and long-term operation conditions at high pressure. Such degradation results in the formation of non-selective defects that dramatically compromise separation performance and reduce membrane lifetime. [1,2,40,41]

The concept of self-healing materials has transitioned from structural engineering to water treatment membranes as a means to reverse fouling and structural damage. Nature has very good designs for automatic repair, especially through mussel glue proteins that contain 3,4-dihydroxyphenylalanine (DOPA). The application of this bio-inspiration in membrane science using catechol-metal coordination and layer-by-layer assembly has created new paths for membrane development with controllable structures and built-in self-repair abilities. This paper critically discusses recent developments in bio-inspired SH-PEMs by looking at molecular healing mechanisms, architectural tuning, ion transport phenomena, and industrial application pathways. [6,7,10]

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2. Fundamental Mechanisms of Membrane Degradation

As compared to conventional membranes, alternative membranes operate under either different conditions or according to different principles. However, they are still composed of synthetic, polymeric materials, thereby exposing them to the same range of failure modes that conventional membrane systems face. Given the complexity of these systems and their exposure to often harsh environmental conditions, there is a pressing need to implement systems conducive to self-healing of membranes.

The degradation of membranes can be classified into categories based on the causal factors. The first general failure mode is physical degradation, which can manifest as a number of different mechanical actions, leading to the loss of effectiveness in membrane separation. Mechanical scratches, such as those induced by operational conditions like turbulence, creep and fatigue cracking lead to degradation in the functionality of membranes. The second failure mode is chemical degradation. Chemical degradation can exhibit itself in several forms but often results from a poor resistance to oxidation by free chlorine. This also often leads to a decrease in the salt rejection ability of the membrane. The last general failure mode is biological and organic fouling. Membranes can be fouled through the adsorption of organic materials, including proteins and humic acids, leading to permeate quality reduction. The accumulation of these organics may result in the fouling of membranes, where biofilms form on the surface of the membrane, causing a drop in permeate flux, which is exacerbated when the back-washing of membranes is undesirable. [43,44,45,46]

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In all the aforementioned cases, the degradation of membranes, and their properties, is accelerated by conventional strategies that aim to mitigate the impact of the previously mentioned factors. The need for self-healing systems is further underscored by the need to replace an entire module or an entire bank of modules in the case of one failure. As such the development of membrane systems that are impervious to, or even responsive to these types of membrane degradation will be a significant leap forward in membrane development.

3. Evolution of Self-Healing Strategies in Polymeric Membranes

Self-healing strategies in polymeric membranes are broadly classified into extrinsic and intrinsic approaches.

3.1 Extrinsic Healing (Microencapsulation and Vascular Networks)

In polymer science, the concept of self-healing materials has made great strides. Today, materials scientists can impersonate biological tissues with synthetic materials, including automobiles, paint, and plastics. Initially, materials scientists used microcapsules with polymerizable agents and reactive compounds to form crosslinks. The most well-known example is the use of microcapsules filled with Dicyclopentadiene (DCPD) to heal material cracks. However, materials have to massively expend energy for polymerization and release of agents in early methods. Also, these methods are single-use: once a crack occurs, the healing agent is released and the structure cannot self-heal again. [8,48]

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More importantly, self-healing agents can leak out along with moisture in the environment. For example, if water is present when a self-healing material is designed with the microencapsulation method, DCPD can react with water to form waxy deposits. Such deposits can build up and create structural defects as they move as a liquid.

3.2 Intrinsic Healing via Dynamic Covalent Bonds

Dynamic covalent chemistry, involving highly reversible covalent bonds, offers effective dynamic systems for synthesising polymer networks with intrinsic self-healing properties. However, the chemistry must be carefully selected, as dynamic covalent bonds must be sufficiently reversible to allow efficient healing, while maintaining sufficient bond strength to allow the desired mechanical and thermal properties of the material. Diels-Alder reactions, which are reversible under elevated temperature or specific pH changes, and disulfide exchange have both been successful in producing self-healing polymer networks. However, these require either external stimulus, are expected to be impractical for room temperature systems or are not suited to continuous operation in water, preventing widespread commercial use. [49,51]

New methods, such as boronic ester bonds, offer advantages over traditional methods in that they bond purely through hydrogen bonding, making them available at room temperature and whilst submerged in water. However, how effective these are at creating bonds strong enough to gain the needed mechanical strength of the material, yield new discoveries. Understanding the bonding will allow tighter control over the mechanical strength, thermal properties and self-healing efficiency.

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3.3 Intrinsic Healing via Supramolecular Interactions

Supramolecular chemistry is a branch of chemistry that gathers the study of non-covalent interactions. This is done for the purpose of designing advanced chemical systems. The key focus of this section is the study of the healing efficiencies of different non-covalent interactions. The method of hydrogen bonding offers low healing efficiency (60-65%) and is not stable in highly aqueous media. The method of hydrophobic interactions offers good healing efficiency, nevertheless, it suffers from slow healing kinetics in aqueous media. The method of metal-ligand coordination offers excellent mechanical strength and is an autonomous healing system where the metal-ligand bonds are reversibly dissociated and re-associated under stress. The key focus of this section is to highlight and emphasize the major themes of supramolecular chemistry to study, analyze, and develop advanced chemical systems that exhibit desirable characteristics like efficient healing characteristics, self-repair, reusability, and higher efficiency and effectiveness. [52,53,57]

4. Bio-Inspired Catechol-Fe^{3+} Coordination Chemistry

The catechol-Fe^{3+} complex is the most studied metal-ligand system for the preparation of a biodegradable adhesive for application in aqueous environments. The catechol-Fe^{3+}-chelation is a model of the natural cross-linking mechanism of the mussel byssal threads, which serve as super adhesive to substrates in wet conditions. [10,18,58,59]

4.1 Fundamentals of Mussel Adhesion

Natural mussel-derived DOPA-rich proteins provide inspiration for the design of synthetic materials as the catechol side chains of these proteins are known to oxidize and chelate metal ions providing this class of protein with unique

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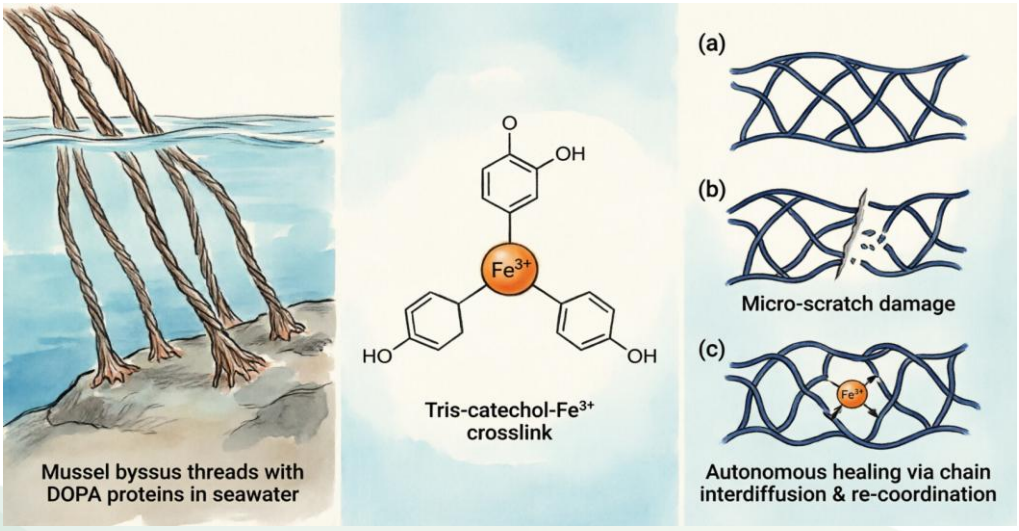
properties enabling functions such as underwater adhesion, complex material assembly, and self-healing. Using Fe^{3+} ions, synthetic polymers such as dopamine-modified polyethylenimine form tough self-healing plaques forming specific crosslinks preventing the materials from being inherently brittle enabling the material to receive damage, annihilate and recover from said damage. Alternative synthesis strategies may also be employed such as utilizing dopamine-modified PDADMAC to aid in substrate adhesion, however, the cost of such polymers limits their commercial appeal. The use of DOPA and catechol based polymers remains an area of growing research potential with possibility of functional fibre synthesis if cost effectively achievable in the future. Another area of potential exploitation is the potential dynamic cross-linking provided by catechol containing polymers.



[11,12,34]

Figure 1. Bio-inspired catechol-Fe^{3+} crosslinking mechanism based on mussel byssus chemistry and autonomous re-coordination after micro-scratch damage.

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4.2 Molecular Dynamics of Catechol-Fe^{3+} Networks

The developed binding capacity of catechol groups (1,2-dihydroxybenzenes) is due to multiple mono-, bis- and tris-catechol-Fe3+ complexes between catechol ligands and the metal ion (Fe3+)[58, 59]. Tris catechol-Fe3+-complexes are present only in high catechol concentrations and are characterized by distinctively high binding constant. Low pH (i.e. <4) and, thus, protonation of catechol groups results in dissociation of ligand functional groups and loss of strong catechol-Fe3+ bonds. This leads to physiochemical bulk material changes associated with the pH-induced causation. Pockets of tris-catechol-Fe3+ complexes are pH-independent as they contribute a strain-storing mechanism to ensure material integrity and surface structure under perturbations of environmental parameters. Bond reformation is spontaneous in the pH range of 4-7, where no protonation occurs and functional groups are able to bond link. [18,60,63]

4.3 Stress-Induced Autonomous Healing in Aqueous Environments

Localized mechanical stress, similar to the tensile strain found in drying processes, may induce transient bond dissociation in coordination equilibria. If observed with an optical microscope, such transient pH changes are not detectable at the bulk level, i.e., no bulk pH change is observed. However, localized mechanical stress can lead to temporary bond dissociation between tris-catechol-Fe^{3+} crosslinks and polyelectrolyte chains, leading to structural instability. The facilitated chain mobility leads to entropic mixing to counterbalance this instability. Although the structural stability at the micro level is compromised, this can counteract the pH change on a more macro scale.

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The individual polyspecifically-coordinating catechol groups are mobile and thus enable the polyelectrolyte chains to interdiffuse and re-coordinates in dynamic equilibrium with the external Fe^{3+} tracer from the habitat liquid upon exerting mechanical stress. Thus, the structural stability is guaranteed at zero cost for the interfacial selectivity of the polyelectrolyte.

5. Tunable Selective Layers via Layer-by-Layer (LbL) Assembly

The thickness of the selective layer, charge density, and pore structure are regarded as the most essential factors that govern NF membrane performance. LbL assembly offers unparalleled control over these parameters at the nanoscale level**.**

5.1 Principles of LbL Fabrication

The multilayer films are prepared by sequential deposition of anionic sulfonated poly(ether ether ketone) (SPEEK) and cationic PDADMAC-Cat types, with the aim of creating a multilayer polyelectrolyte structure on porous support Polyethersulfone (PES) by electrostatic interactions (Figure 1). After that, the stabilization of multilayer films is achieved by treatment with $FeCl_3$ solution, which allow performing the cross-linking via catechol coordination between the stabilizing agent and the functional groups of surfacez on the multilayer film. The layers of the obtained SPEEK/PDADMAC-Cat films obtained by the multilayer technique can work as a selective barrier against metal ions such as Cu^{2+}, Zn^{2+}, and Ag^{+}.

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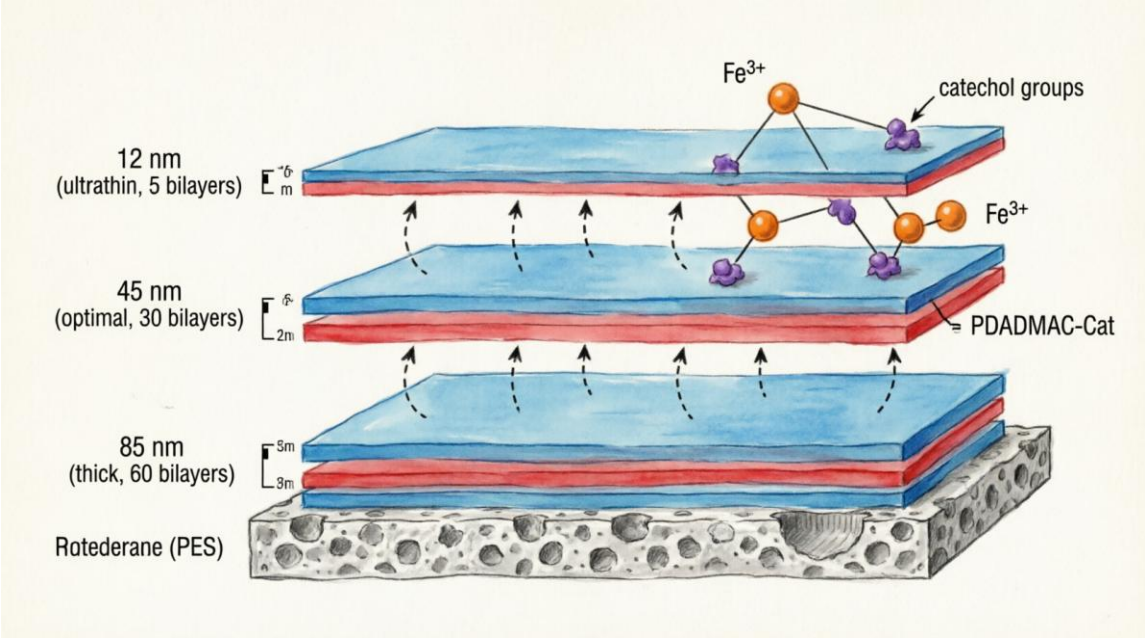

[3,4,65,68]

Figure 2. Layer-by-layer self-healing PEM architecture on a porous PES support, showing tunable selective-layer thickness and catechol-Fe^{3+} coordination sites.

5.2 Nanoscale Thickness Control and the Permeability-Selectivity Trade-off

Both ultrathin and thick LBL membranes are synthesized to show the practical water filtration performance of these materials. The linear thickness growth (~2.1 nm per bilayer) leads to the ultrathin layers with high water permeance (>60 LMH/bar) and moderate salt rejection (~84% for Na_2SO_4) as well as thick layers that provide near-complete rejection (>99%) but decreased permeance (<9 LMH/bar). Interestingly, the best performance was achieved at the membrane thickness of ~45 nm (30 bilayers), where the permeance was

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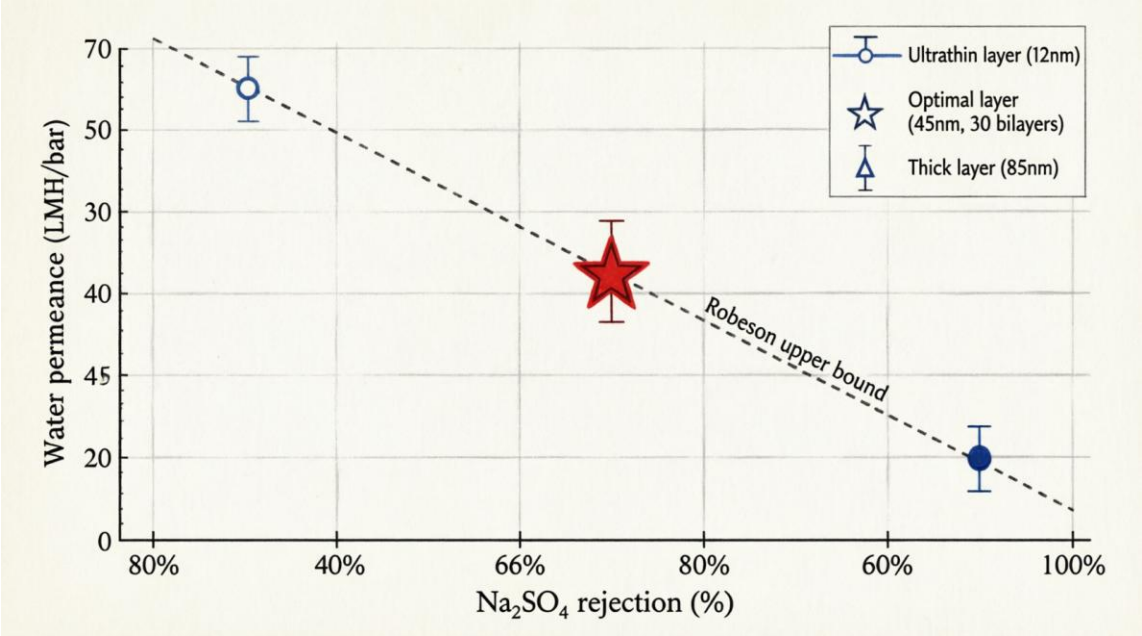
sufficiently high (28.4 LMH/bar) while the rejection was exceptional (98.3% for Na_2SO_4). This high performance surpassed the standard polyamide NF membranes that have been commercialized today and showed that the LBL assembly can easily tune the membrane architecture to achieve practical water filtration membranes.



[75,80]

Figure 3. Thickness-dependent permeability-selectivity trade-off for ultrathin, optimal, and thick LbL self-healing membrane layers.

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6. Ion Transport and Separation Mechanisms in Charged PEMs

The main benefit of membranes based on proton exchange membrane technology is their capacity to separate ions not just by size but also by charge; this is explained through the Donnan-Steric Pore Model. [19,77]

6.1 Desalination Performance and Charge Dominance

The rejection of negatively charged salt solutions against ionic membranes such as sulfonated poly(ether ether ketone) which means SH-PEMs follows the sequence of Na_2SO_4 > $MgSO_4$ > $MgCl_2$ > NaCl which signifies the counter-ion and the size of the anions hydrated radius of the salts solutions. The rejection of divalent salt like Na_2SO_4 is high due to electrostatic repulsion and Donnan exclusion would also contribute to that. As a counter-ion larger hydrated radius salt (SO_4^{2-}) 0.38 nm than small hydrated radius salt such as Cl^- 0.33 nm which could further increase the Donnan exclusion and give the membrane better rejection. [19,28,76]

6.2 Monovalent/Divalent Ion Selectivity

Membranes exhibiting very high negative surface charge density (~−1.2 C m−2) are typically called strong-hydrophilic cation exchange membranes (HPEM), even if their ionic conductivity is still relatively low compared to the charging with hydronium ions or hydroxide ions. These properties and the related performance are interpreted with a combined Donnan potential and fixed charge model.

However, in order to separate monovalent from divalent cations through cation exchange membranes (CEMs) or mix anion exchange membranes (AEMs), this intrinsic Donnan potential or the one generated by the surface charge has a negligible impact on the separation performance of the membrane. This is particularly true for strong-hydrophilic proton exchange

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membranes (SH-PEMs) which are present in well-established synthesis procedures and results in a low cloride/sulfate separation factor in the order of 18.7.

To achieve this ratio, several basic state-of-the-art methods could be applied like surface charge engineering at the membrane surface, utilization of charge selective interlayer, and the application of mechano-chemical operation like hybrid ion-exchange electro-dialysis or hybrid membrane distillation membrane electrolysis.

In 2015, our research group conducted a systematic study on the impact of counter ions covered through ion-exchange of SH-PEMs. Appreciable improvements in cation mixtures could be achieved from the strong-negative surface charge density of the membrane in particular with a stronger hydrated radius. In this, we achieved a Mg2+/Na+ selectivity factor of ~9.3, which was interpreted with the very different ionic permeation rates of the two cations followed by Cl-/SO4-2 as shown below. Here, the principle of differential solvation and the Donnan exclusion on the negatively charged membrane surface (Donnan-Stern-Peebles-Meyer) was applied on the DSPM strategy.

Note: In the figure, we determined the permeation rates through the membrane contacting with the two different ion mixture solutions.

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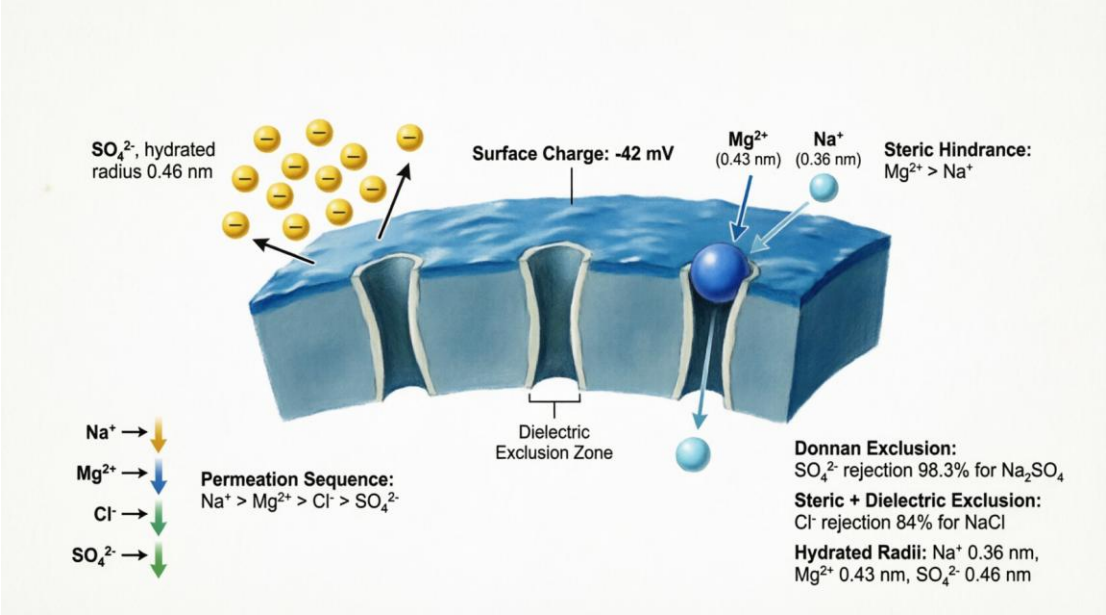

Figure 4. Ion transport and separation mechanisms in negatively charged PEMs, combining Donnan exclusion, steric hindrance, and dielectric exclusion.

7. State-of-the-Art Performance: Antifouling and Healing Metrics

Beyond initial separation metrics, practical viability relies on operational stability and resistance to organic fouling.

7.1 Cyclic Healing and Defect Recovery

Unlike the microcapsule systems that perish after 1-3 cycles, intrinsic catechol-Fe^{3+} networks possess permanent healing properties. When subjected to micro-cutting (~5 μm), state-of-the-art membranes completely close defects in 6 hours under ambient conditions [23]. Absolute recovery metrics report healing efficiencies of more than 94% for water flux, greater

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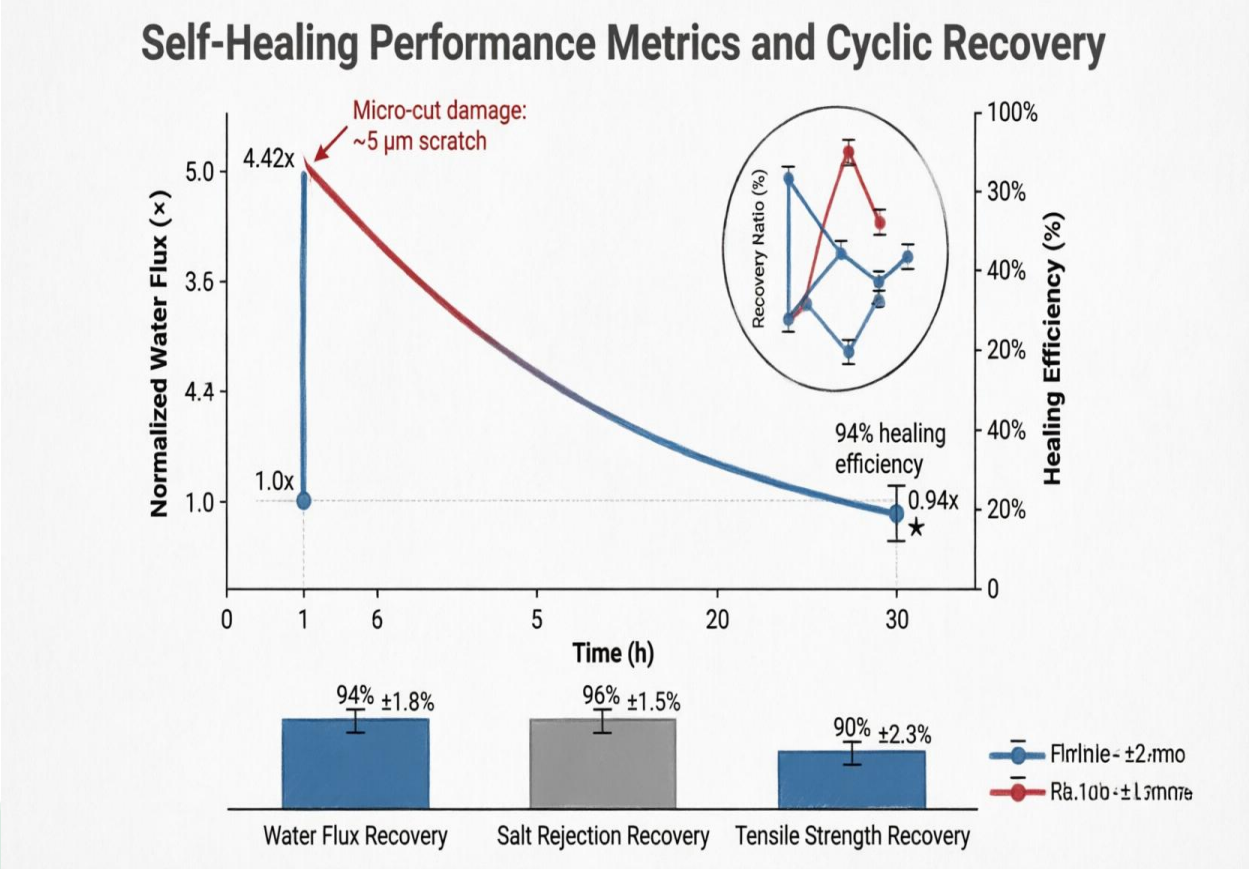
than 96% for salt rejection, and more than 90% for tensile strength over five consecutive damage-healing cycles [24]. Upon damage, normalized water flux surges (e.g., up to 4.42x pristine values) due to very low hydraulic resistance through the defect; then it drops to near-pristine flux which serves as direct quantitative proof of barrier restoration.



[23,24]

Figure 5. Self-healing performance metrics and cyclic recovery after micro-cut damage, including water flux, salt rejection, and tensile strength recovery.

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7.2 Antifouling Performance and Operational Stability

The membranes prepared based on the ultra-smooth surfaces (roughness < 5 nm) demonstrated a fundamental difference in performance relative to conventional membranes. The spin-assembled LbL coatings (SH-PEMs) led to a greatly reduced adhesion of foulant. All the SH-PEMs exhibited a much higher FRR (> 94%) relative to commercial membranes. The NF270 commercial membranes, for example, exhibited a much lower FRR (~78-82%) while the SH-PEMs represented a novel approach to designing membranes with ultra-smooth surfaces. Moreover, the extent of flux decline was significantly less for SH-PEMs over the entire testing period (~240 hours) where the total flux decline was <8%, while the same testing done with commercial membranes showed a flux decline >23%. While these promises have shown promise in the laboratory, long-term industrial validation is needed. This would involve subjecting the prepared SH-PEMs to validation through multi-month continuous pilot studies where the membranes would be systematically tested and evaluated for changes in permeability and fouling with seasonal variations and validated against extensive flux decline which is a common hurdle otherwise solved through extended chemical cleaning protocols.

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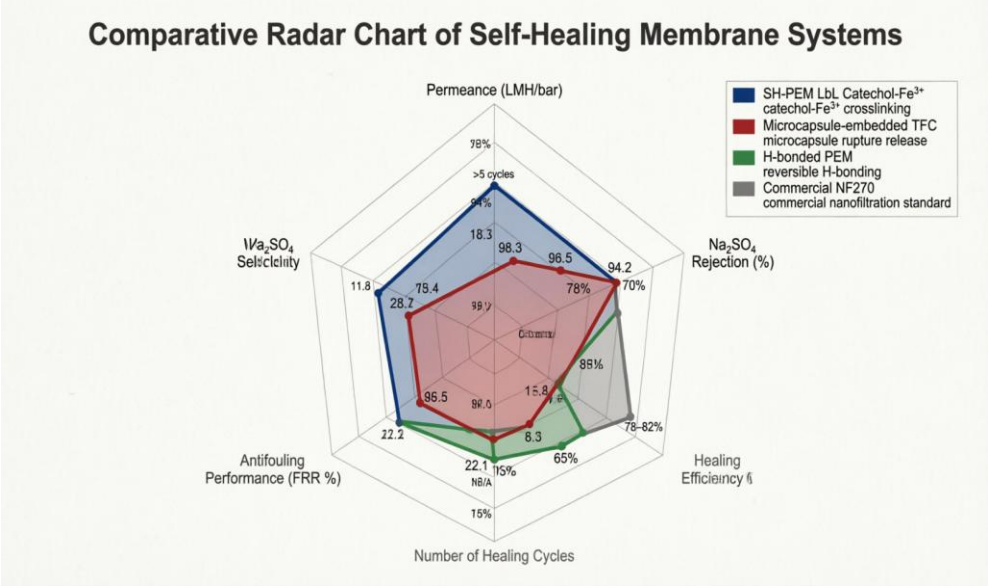

[20,33,78]

Figure 6. Comparative radar chart of self-healing membrane systems across permeance, salt rejection, healing efficiency, recovery cycles, antifouling performance, and ion selectivity.

8. Current Challenges and Future Perspectives

Technologies associated with thin-film membranes (e.g. reverse osmosis, nanofiltration, ultrafiltration, etc.) are mature and widely applied for the membrane separation of water in agriculture and fast-growing industries. In this aspect, the number of papers, patents, and products, though heterogeneous in quality and impact, is sufficient for a potential market penetration. There is also a clear technological need in the natural resource recovery sector (e.g. for rare metals from e-waste, e.g. Li^+ from electrochemical batteries). Metal-ion recovery is associated with a double-challenge, that is, the reliable application of catechol-Fe^{3+} bonds for electrophoretic ally deposited and functionalized

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LbL frameworks, and the need for ultra-low content of free tri-valent metal ions in the final membrane products, meeting WHO limits of 0.3 $mg \cdot L^{-1}$. Both challenges can be met when replacing the Fe^{3+} ions by other metal ions, e.g. Al^{3+} or Ti^{4+} etc. Studying if this is possible, and with which metal ions, or by applying post-treatment protocols based on the metal ion of interest, is one of the biggest challenges in the evolution of this technology. Once shown to function sufficiently for this metal ion recovery, the technology can be translated from laboratory to an industrial level, involving both scientific/technological (scale-up and/or a different kind of design) as well as economic aspects (Techno-Economic Analysis). For the first, the use of spray-assisted LbL assembly and in situ spectroscopic tracking for polymer interdiffusion and chemical re-coordination kinetics can be the solutions. For the latter, life-cycle assessments of desalination plants will strongly support the development of pilot-scale desalination plants. [30,79]

9. Conclusion

Cationic aquo-metal complexes (specifically Fe^{3+} ions) covalently bonded with polyphenolic compounds (like catechol) via chelation can lead to advanced membranes for ion separation. In this work, the nature of these coordination bonds is studied for their application for self-repairing membrane separation systems (some patents pending). Initially these coordinating ions were bound to the surface through layer-by-layer assembly (SALD) to create membranes with both high water permeance and monovalent or divalent ion selectivity (coion Donnan exclusion) through a selective layer with a tunable thickness (12–85 nm). The two-dimensional nature of the polymer metal ion network allows for the heavily coordinated Fe^{3+} ions to become exchangeable for cations in a bulk solution, and remove

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themselves from the network for immediate healing of an interrupted barrier. The healing efficiencies recorded were above 94%, and the layers retained their antifouling properties towards both neutral dye and protein foulants. Moving on from this system, the discovery that SH-PEMs (self-healing-polymeric-embedded membranes) can improve upon the mechanical performance and their ability to heal was made, and their unique structural and functional properties enable new approaches to specific corrosion resistance for materials. [18,62,64]

The precise spatial positioning of strongly coordinated cation aquo-metal complexes arising from metal ions added and incorporated for SH-PEM construction were tracked to understand if the content ions were purely a function of the layer themselves or if they altered the properties and functions of their surrounding compositional polymer. Following the determination that the metal ions did influence compositional polymer properties, these highly coordinated ion networks were used to mitigate metal-ion leaching. This discovery was used with an innovative and scalable continuous fabrication method (spray-LbL) for SH-PEMs. The improved scalability of this membrane approach allows for membranes to be made in conservatively, multi-month pilot-scale batches, and were validated economically at this scale. Their advantages make these SH-PEMs for multifunctional (ion specific and sensor) intelligent desalination infrastructure for the water-energy nexus an approximate future application.

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Table 1. Comparative analysis of self-healing mechanisms in state-of-the-art polymeric NF membranes. (Note: Data for the SH-PEM system represents a recent state-of-the-art benchmark synthesized from latest literature [14, 15, 18, 23], used here as a comparative standard against classical systems).

Membrane System	Healing Mechanism	Permeance (LMH/bar)	Na_2SO_4 Rej. (%)	Cl^-/SO_4^{2-} Selectivity	Healing Eff. (%)	Cycles
SH-PEM (LbL Catechol-Fe^{3+}) [14,15,18]	Intrinsic dynamic coordination	28.4	98.3	18.7	94.2	>5
Microcapsule-embedded TFC [8]	Extrinsic microencapsulation	15.2	96.5	8.3	78.0	1-3
H-bonded PEM [9]	Intrinsic H-bonding	22.1	89.4	6.5	65.0	Limited
Commercial NF270 [32]	None (Passive control)	11.8	97.0	9.8	N/A	N/A

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