

## Approaches of Membrane Modification for Water Treatment

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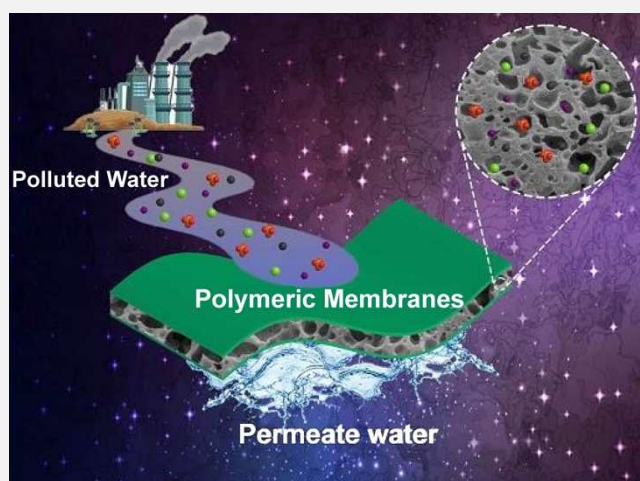


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### ABSTRACT

The environment and how to protect it is a crucial subject in the twenty-first century. The membrane field is one of the most reliable and cheapest methods, which is catching the eye these days. Although membranes are efficient and easy to handle, the vulnerability against fouling agents and poor stability are drawbacks. Fortunately, a diverse range of methods developed over the past few decades for membrane modification. Each modification method can offer some benefits that can enhance the membrane's overall performance. On the other hand, almost all of them also have some drawbacks. Therefore, knowing the aspect of each modification method can lead researchers to choose the best one based on the desired application. Generally speaking, membrane modification can culminate in better water permeation, higher rejection rate, more thermal and mechanical stability, higher surface hydrophilicity, and more extended durability. Therefore, a study about membrane modification methods and their advantages and disadvantages was needed. Herein, a short but thorough review is brought to indicate how membranes could modify with different methods, and their fortes and drawbacks are briefly discussed. In addition, new studies were brought in to elucidate how these methods are gaining real applications.



Therefore, a study about membrane modification methods and their advantages and disadvantages was needed. Herein, a short but thorough review is brought to indicate how membranes could modify with different methods, and their fortes and drawbacks are briefly discussed. In addition, new studies were brought in to elucidate how these methods are gaining real applications.

**Keywords:** Water treatment, biopolymer, green chemistry, membrane, nanomaterials

## 1. Introduction

There is a wide range of methods for treating water polluted with different contamination, such as membrane separations, photocatalysts [1,2], gravity method [3], active sludge separation [4], activated carbon separation [5], air flotation [6], and centrifugation [7] and membranes are one of the most reliable ones. In the last century, different types of membranes were developed and used. The most famous ones are ceramic-based, inorganic-based, zeolite-based, and polymeric-based [8]. However, polymeric membranes surpassed all the others in recent decades and somehow occupied all academic and industrial research and applications, although the reason is apparent [9]. Membranes with polymeric texture are affordable and accessible; they can offer mass production and are open to further modification [10]. Membranes are usually used for the remediation and purification of water from pollutants, and these pollutants are industrial dyes, proteins, oils, and even bacteria, which are considered organic [11]. The organic nature of pollutants means they can readily adsorb on the surface of membranes, which means pore clogging, which is not favored [12]. Unraveling this problem remains the most conventional subject in this area because adsorbing these molecules can lead to severe fouling of membranes.

Researchers worldwide have proposed numerous methods to solve membranes' high affinity toward organic molecules and structures [13]. Higher hydrophilicity means less tendency toward fouling agents, so the surface of a membrane with higher hydrophilicity has a better chance to protect itself [14]. Amination [15], chloromethylation



[16], sulfonation [17], adding hydrophilic segments [18], etc., are some popular approaches that can improve membrane hydrophilicity. Besides enhancing hydrophilicity [19], improving anti-bacterial features [20], and boosting gas solubility [21] are just some of the other aspects of the membrane which require modification. Considering the wide range of membrane applications, choosing the proper method to ease the modification process is vital and time-saving. Desalination [22], remediation of heavy metals [23], dye separation [24], fuel cells [16], artificial cells [25], gas separation [26], hydrogen separation [27], and (proton exchange membranes) PEMs [17] are areas where membrane modification is needed.

## 2. Membrane modification methods

In this mini-review, 7 methods of modification are introduced and briefly discussed. New studies in each method were brought as well as the merits and drawbacks of each method provided.

**Table 1.** Recent articles about membrane fabrication methods

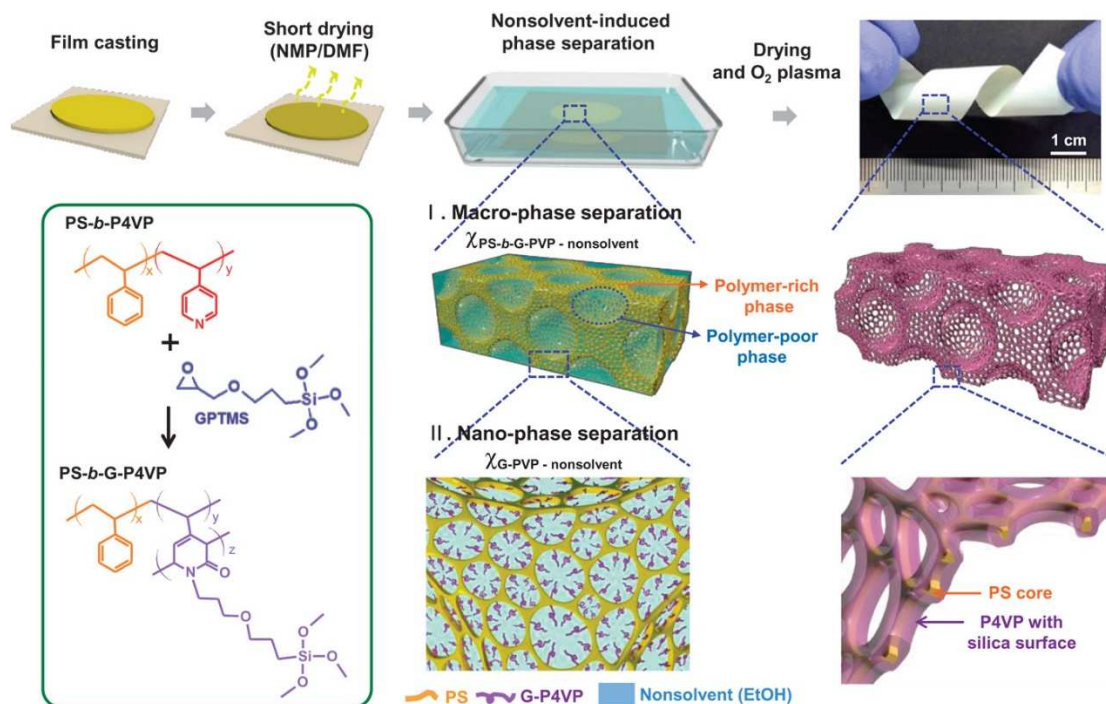
Technique label	Recent articles
Using co-polymers	[28–34]
Sulfonation	[35–38]
Nanomaterials	[39–42]
Surface modification	[43–46]
Physical modification	[47]
Amination	[48–50]
Chloromethylation	[51–54]

### 2.1. Using copolymer

When it comes to the membrane, it is crucial to combine both the properties of selectivity and permeation to boost the purification efficiency. Therefore, membrane modification seems to be vital and one of the best ways is using Co-polymer and fabrication of Copolymer membranes, and numerous prevalent techniques are used to produce them [55]. Copolymers (CPs) can offer better selectivity to produce porous membranes based on the high pore density caused by organized self-assembly [56]. CPs with a narrow range of disparity are vital for modifying membranes [57]. These narrow-range CPs can be fabricated successfully by following the Controlled radical polymerization process (CRP), which is a famous and popular approach to fabricating copolymers with distinct morphologies such as star-shaped, linear, etc. [58]. Although, besides the fabrication approach many other factors can determine the final geometry of the copolymers, such as solvent, temperature, chain length of components, and Florry-Huggins parameters [55,59]. Broadly speaking, CPs membranes are generated from solution, therefore choosing the right and the suitable solvent is necessary for determining the ultimate structure [15,60]. Eventually, it must be said although CPs can offer a lot, the modification with copolymers might not have a clear approach because, as said, this method has a wide variety, and each parameter can affect the result drastically; moreover, radical polymerization is not available for all polymeric membranes. PEG, PVP, and PES are the most used copolymers [61,62].

In one recent study, different polyether-block-amide type copolymers were studied regarding gas separation. These membranes were polyether block amide (Pebax) 1657, 2533, 3533, and 4533, which had a different allocation of rigid parts, and after close investigation with different instruments, it was found that membranes with the softest parts revealed the best properties because these membrane's polyethylene oxide considered soft phase, which can augment the intense interaction with carbon dioxide. More reaction with CO<sub>2</sub> means higher solubility selectivity of CO<sub>2</sub>/N<sub>2</sub> and, as a result, higher CO<sub>2</sub>/N<sub>2</sub> separation selectivity [63].

In another study, polystyrene-block-poly (4-vinyl pyridine) was modified with 3-glycidoxypropyl trimethoxysilane (GPTMS) via dual-phase separation and then dried at oxygen plasma. The fabricated porous membrane was used in the ion battery. The process is illustrated in **Figure 1**.



**Figure 1.** Modification of polystyrene-block-poly (4-vinyl pyridine) with 3-glycidoxypropyl trimethoxysilane (GPTMS) via dual nonsolvent-induced phase separation (NIPS) and dried with O<sub>2</sub> plasma to fabricate ultra-porous membrane. Reprint with permission from [63].

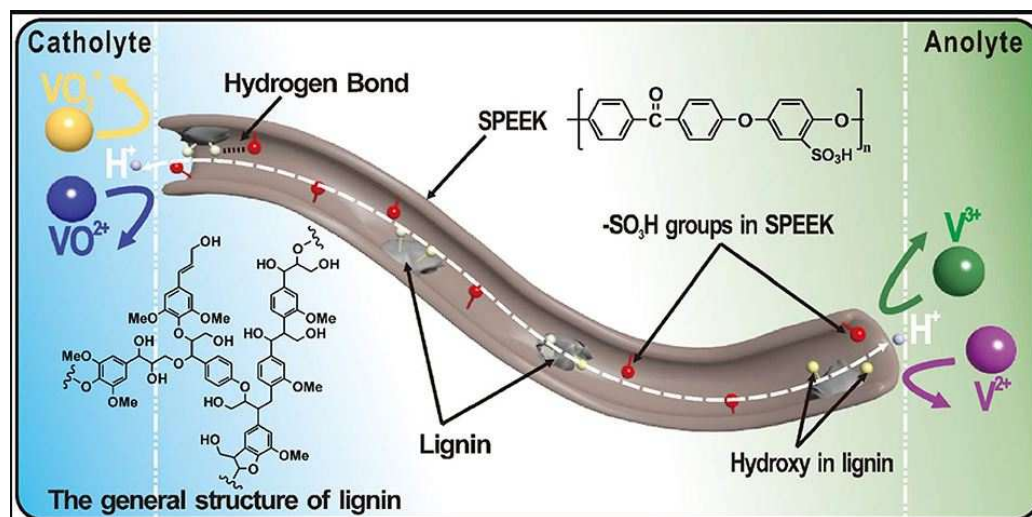
## 2.2. Sulfonation

Inorganic fouling is one of the most contentious problems in saline water purification; it remarkably weakens the saline water purification of the reverse seawater osmosis (SWRO) membranes [64]. Conventional nanofiltration (NF) membranes, which act as the pretreatment medium, are unable to fractionate the scaling ions, Ca<sup>2+</sup> effectively, and sulfate, culminating in fouling transfer from SWRO to the NF membranes [65]. On the other hand, large amounts of organic solvents are used in the food industry, petrochemicals, pharmaceutical industries, weaving industries, and textiles [6]. Some of these organic solvents are toxic and can endanger the environment [7]. Sulfonation is one the best way to solve both mentioned problems. H<sub>2</sub>SO<sub>4</sub>, SO<sub>3</sub>, and ClSO<sub>3</sub>H are prevalent molecules for sulfonation. In this method, one of these agents could be used as a co-solvent in the NIPS process. For example, using sulfuric acid as a co-solvent can enhance membrane stability during the process, and sulfonic acid segments can boost membrane hydrophilicity. In addition, sulfonic acid can also culminate in surface charges, which can enhance the rejection rate. Besides all merits that sulfuric acid can endow on the membrane, sulfonate also has vivid drawbacks [66]. For example, most membranes can easily impair against sulfuric acid, and there is no reliable approach to protect those membranes against this acid; Polyethersulfone (PES) and polyvinylidene fluoride (PVDF) are among them. Moreover, the remaining sulfuric acid can damage instruments and cause corrosion [10].

In one study, the polymer was prepared into a membrane by using the NIPS method with a mixed acid as a solvent. The poly (aryl ether ketone) (PEK-SPEDEK)-10 and PEK-SPEDEK-20 organic solvent separation membranes were prepared via sulfonation with sulfuric acid, which was used as a solvent for the casting solution. The results indicated that sulfonation leads to higher stability and higher solvent permeation in organic solvent separation [67].

Another study about sulfonation used sulfuric acid and methyl sulfonic acid as solvents via non-solvent reverse conversion and evaluated its organic solvent separation performance, with a specific focus on aging. Molecular weight cut-off (MWCO) regulation was achieved by changing the solvent of the pores before membrane drying.

The optimized membrane depicted excellent stability in the organic solvent, and its MWCO improved [68]. **Figure 2** shows a composite membrane modified with  $\text{SO}_3\text{H}$  to be used in a battery [33].



**Figure 2.** The general structure of lignin and schematic illustration showing the water channel in a composite membrane for vanadium redox flow battery. Reprint from [67].

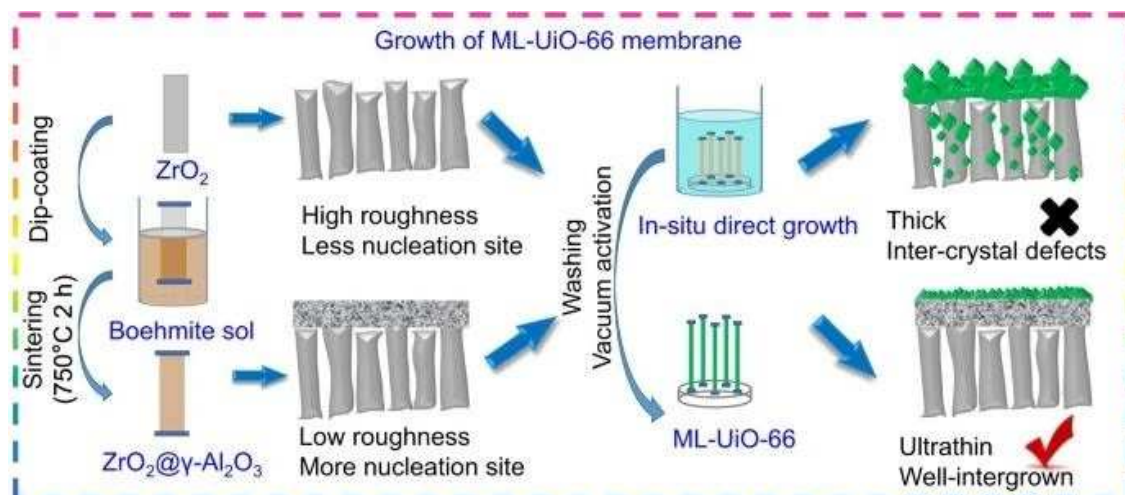
### 2.3. Nanomaterials

Using nanomaterials in the membrane field is a routine and daily thing. Nanomaterials are different and varied from each other, so each one of them can cause a unique and desirable feature in a membrane. One-dimensional, two-dimensional, and three-dimensional nanomaterials have been synthesized over the past few decades, and there seems to be no end and limitation in a new one [69]. Nanomaterials can bring about more stability, tunable porosity, better hydrophilicity, and pore distribution, which are favored for membrane modification [69,70]. Introducing nanomaterials to the membrane matrix or incorporating them between thin-film layers (polyamide layers) is the most popular way of using these particles [14]. On the other hand, most nanomaterials are inorganic. Because of that, most of them have poor compatibility with the polymer matrix [71]. Moreover, in nanofiltration membranes, pressure is so high that these particles can be washed out easily.

In one study, MIL-53 was incorporated into the PES membrane. MIL-53 is a metal-organic framework (MOF) with an ultra-porous structure and can be synthesized easily. Embedding these nanomaterials to the membrane matrix provided tunable and narrow channels in the membrane surface, which was used as a safe pass for water molecules transportation. Water channels mean high water permeation, which is favored. Although most membrane parameters, including dye rejection, water permeation, surface roughness, and hydrophilicity, enhanced much, the long-term stability probably did not improve because of the low interaction between MIL-53 and polyethersulfone (PES) matrix. In **Figure 3**, there is an illustration of the modification of the membrane with MOF particles to form a porous and robust membrane [72].

In one study, a molecular-level intra-crystalline defect chemistry strategy was proposed for the fabrication of one robust missing-linker UiO-66 (ML-UiO-66) membrane. Ultrathin ML-UiO-66 membranes showed perfect stability toward hot saline, chlorine, alkaline, and acidic solutions, and more importantly complete salt rejection without sacrificing high water flux ( $\sim 29.8 \text{ L m}^{-2} \text{ h}^{-1}$ ). In addition, the fabricated membrane revealed excellent long-term stability in a low and high concentrations of saline water. This work revealed a riveting approach toward the molecular-level design of highly permeable MOF membranes for challenging separation (e.g., harsh hypersaline water desalination) and a fundamental understanding of how introducing deliberate defects enhances permeation performance [73].

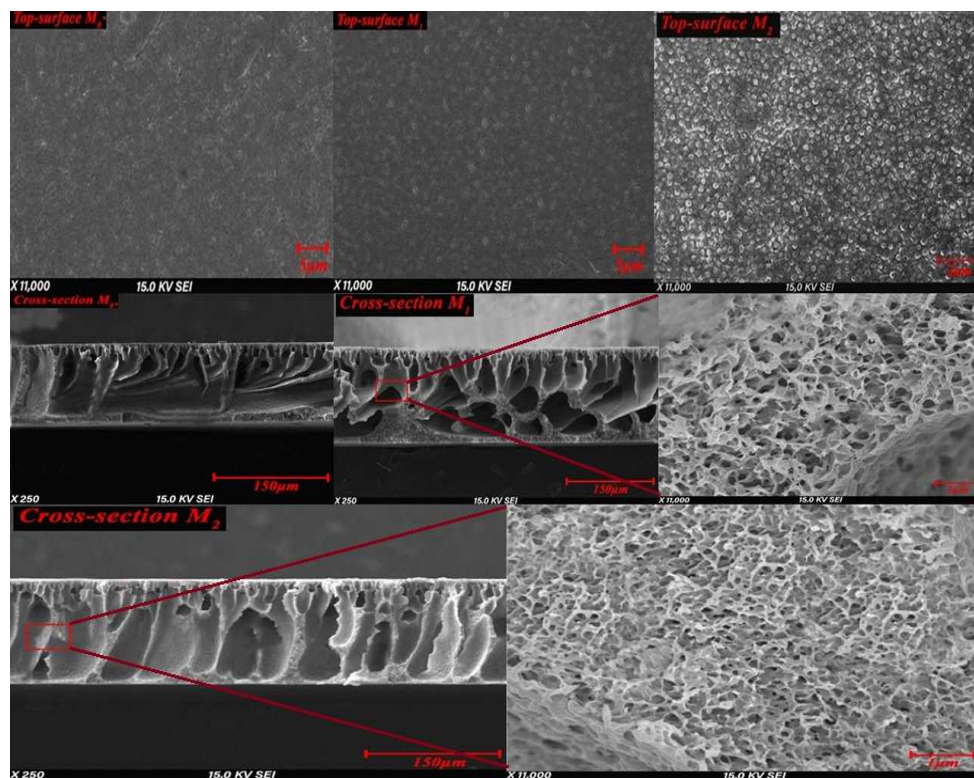




**Figure 3.** Fabrication of  $\gamma$ - $\text{Al}_2\text{O}_3$  interlayer and growth of ML-UiO-66 to fabricate a robust and porous membrane for fast water transport. Reprint with permission from [73].

#### 2.4. Surface modification

Surface modification is a general procedure in many fields, including membranes. The surface of membranes is where that purification and remediation happen because the interaction between membrane and solution occurs there [74,75]. Besides, the membrane surface considers the active site of the membrane and contains most of the functional sites. Therefore, a proper modification surface can lead to a severe improvement in whole membrane performance [74]. Thin-film composite (TFC) membranes, thin-film nanocomposite (TFN) membranes, and layer-by-layer (LBL) membranes are famous approaches regarding surface modification [9]. The effect of surface modification on the final morphology is drastic. Atomic force microscopy (AFM) and Field emission scanning electron (FESEM) are two instruments with widespread usage in morphological studies. As can be seen from **Figure 4**, after the deposition of each layer the surface and cross-section FESEM images of the membrane saw a significant change. The first layer was dopamine and the second layer was one special derivative of epoxy silane [76].



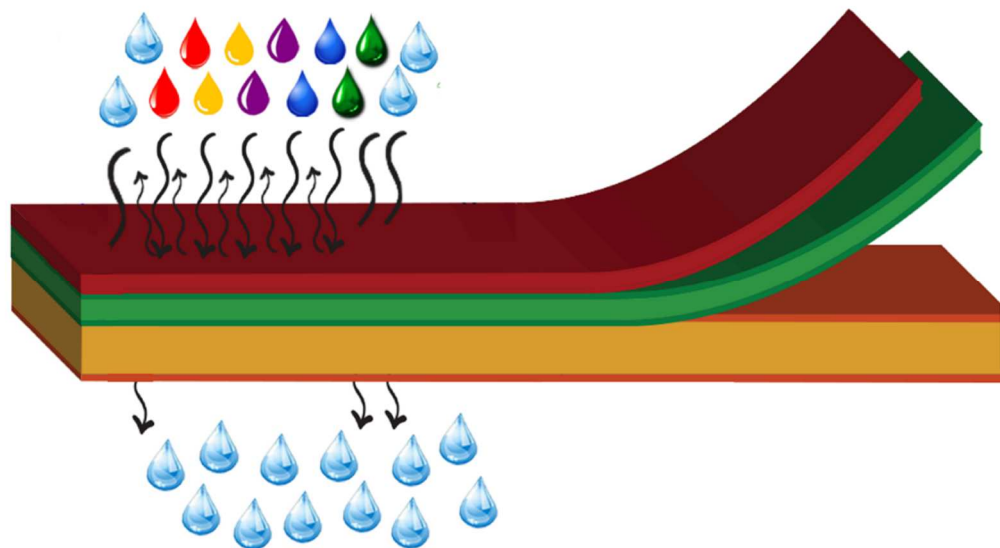
**Figure 4.** The top surface FESEM image of neat PVDF membrane ( $M_0$ ) and top surface FESEM images of the membrane after one and two-layer deposition ( $M_1$  and  $M_2$  respectively). The cross-section FESEM image of pristine PVDF membrane and cross-section FESEM images of modified PVDF membrane after one and two layers of deposition (as shown in the figure, the zoomed images of the cross-section revealed modified membrane possessed sponge-like texture). Reprint with permission from [76].

Some newly published articles around these mentioned approaches were brought in **Table 2**. Surface modification can cover some larger pores, enhance surface hydrophilicity, improve surface roughness and provide a dense layer with enough active sites for purification and separation application [77,78]. However, this method requires delicacy and is time-consuming, so its industrial usage still had to face difficulties and different limitations.

**Table 2.** Recent articles about membranes with surface modification approach

Technique label	Recent articles
Thin-film composite (TFC)	[15,19,22,24,51,56,61,64,65,79–88]
Thin-film nanocomposite (TFN)	[19,22,61,65,82,87]
layer-by-layer (LBL)	[85,89,90]

In one layer-by-layer approach, the PVDF membrane was modified with dopamine in a facile strategy. Dopamine is a popular catechol, used numerously in membranes, and can play as an intermediate layer in LBL modification. After coating the dopamine layer on the PVDF membrane, one multifunctional silane was used as a final layer. Amine groups of dopamine played an anchor role in the final layer. After modification, the optimized membrane surpassed the “trade-off” relation between water permeation and rejection rate and improved both. However, coating two layers on a PVDF membrane got so many times [76]. A layer-by-layer mechanism is brought in **Figure 5**.



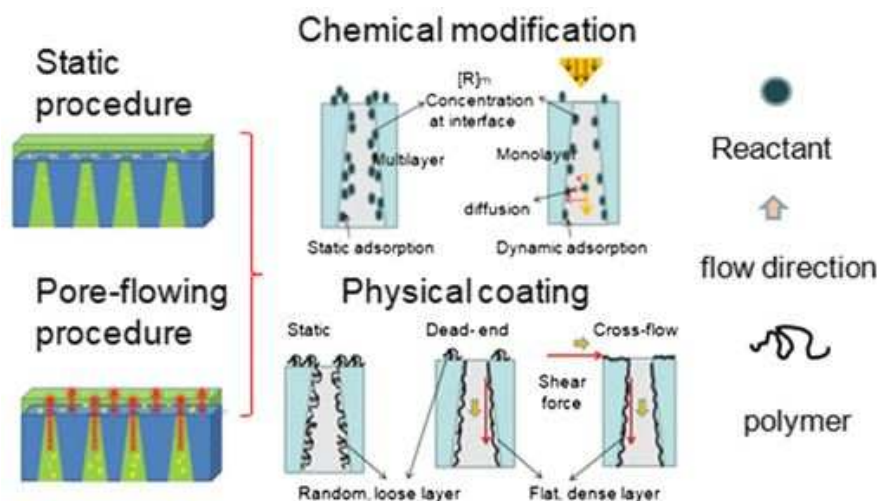
**Figure 5.** Layer-by-layer approach to modify membrane with dye rejection performance.

### 2.5. Physical modification

Facile modification is a forte of membrane technology, which is useful in many ways [91]. Even without chemical molecules and reagents, membrane modification can occur. Physical modification can comprise many different approaches, including heating, cooling, spinning, and physical coating [26]. The heating process can be used to remit the membrane wetting in membrane distillation (MD). Heating can affect membrane morphology, and morphology itself can affect pore size, hydrophilicity, surface roughness, etc. [92]. heating alongside cooling and spinning can also affect the chains array of membranes, which is an important factor in pore size and, more importantly, pore distribution [93,94]. Moreover, physical modification needs no chemical molecules, which is a distinct feature and eco-friendly feature. However, physical modification cannot affect critical features of the membrane, and most of the time, it needs a complementary method. Conversely, high temperatures are required to boost heating influences on the membrane, which can cause damage.

A simple heat process was used to fabricate a polytetrafluoroethylene (PTFE) flat membrane with high hydrophilicity and low free energy. Different morphological aspects of the membrane were investigated, including fibril breakage and the fusion rate of PTFE, and its impact on membrane features such as surface roughness and porosity was discussed. The membrane wetting time in a static absorption experiment and sweep gas membrane distillation was evaluated. In addition, the effect of the heat setting process on the membrane wetting was calculated and discussed. The outcomes showed that more heat could lead to more crystallinity, resulting in a lower surface free energy [95].

In another study, the surface of polyacrylonitrile (PAN) was modified through both chemical modification and physical coating, and the effect of two factors was scrutinized. Those factors were static and pore-flowing procedures. Ethanolamine was employed as a chemical modification, and the fabricated membrane via a pore-flowing procedure depicted a higher water permeation. Physical coating during the pore-flowing and static processes led to a dense, random, and flat layer, which changed the water permeation and anti-fouling features. The optimized membrane illustrated the highest water permeation. The kinetics played a vital role in membrane modification [96]. A brief illustration of mentioned methods is brought in **Figure 6**.



**Figure 6.** Illustration of the Difference in Chemical Modification during Static and Pore-Flowing Procedures and Different Coating Layer Morphologies Caused by Different Modification Procedures. Reprint with permission from [96].

## 2.6. Amination

Methanol, ethanol, dimethyl formaldehyde (DMF), diethylamine (DEA), and n-hexane are the most used solvents in the industry [4]. Protecting the environment and the only habitable planet against these mentioned solvents is an essential and real need in the modern world. Similar to sulfonation, the amination method can improve membrane features as well. Solvent resistance nanofiltration (SRNF) membrane is a crucial subject in an industrial world occupied by different solvents and numerous industries all over the planet. Amination can modify membranes in a way to purify water from these pollutants [97,98]. Laconically, molecules with amine groups ( $-\text{NH}_2$  and  $-\text{NH}-$ ) are potential agents for amination. Cost, size, and the number of functional groups can determine which amine is suitable for the modification process [98,99]. Besides SRNF application, amination can be employed in other applications because amination can enhance hydrophilicity, affect pore size, can change morphology, and can provide active sites for further modification [100]. The alkaline nature of amines can impair membranes, and a need to use only an optimum proportion of these molecules is a sure thing. On the other hand, controlling amination reaction is arduous because amines are active molecules side reactions in many cases are inevitable.

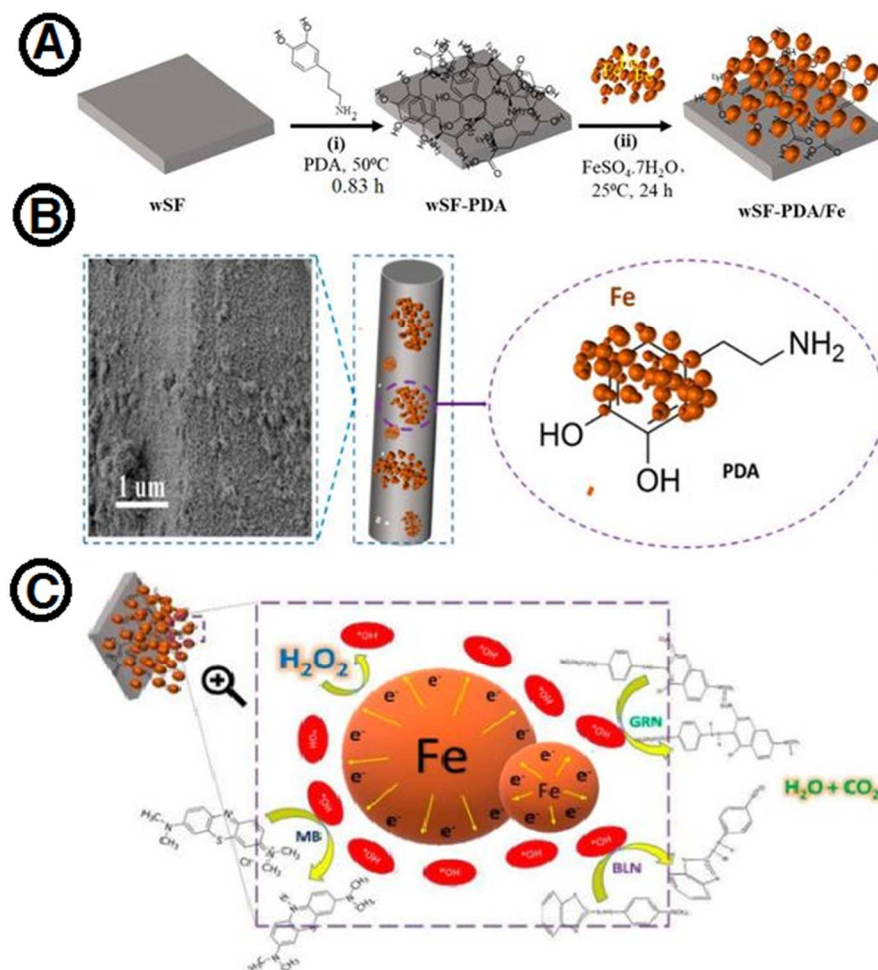
In a previously reported article, fumed silica nanoparticles were modified via 3-aminopropyl triethoxysilane (APTES), and then modified particles were incorporated into a mixed matrix membrane. The as-prepared membrane was cross-linked via 2-hydroxy (methanol) amino ethanoic acid (HDA) to be a conducive membrane in SRNF usage. Different amounts of amino-functionalized particles were embedded into the membrane, and the optimized amount was found. This method increased the durability of the fabricated membrane in a diverse range of solvents. Also, it elucidated that amination is a good candidate for solvent resistance conditions [101].

The effects of the amination agent on the support layers and the effects of the support layers with different degrees of amination on the performance of the effect of amination on thin-film nanofiltration membranes were studied thoroughly in one study. First, the effect of different amounts of triethylenetetramine (TETA) on the support was investigated, and the optimum percentage was found. Second, the effect of each support on TFC layers was studied, and the results were discussed point by point. After that, the strength of interaction between TFC layers and support membrane was studied through water permeation rate, and it was found a higher amount of amines means larger pore size and thinner active layer, which means a higher permeation rate. Accordingly, the mentioned thin-film nanofiltration membrane showed a unique surface structure with nail-fixed morphology (similar to the common polyamide layers morphology but not the same), and cross-section images revealed that the optimized membrane possessed a wave-like structure with a denser and thinner active layer [102].

In one another study, dopamine was grafted on the surface of silk fibers by rapid oxidative polymerization followed by loading/immobilization of Fe particles on polydopamine (PDA) grafted waste silk fibers. Hence, a



fiber-based catalyst was prepared for heterogeneous Fenton-like removal of toxic aromatic dyes. The surface morphology and chemical composition of the resultant catalyst were characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and Fourier transform infrared (FT-IR) spectroscopy. The effectiveness of catalytic property was assessed under the influence of  $\text{H}_2\text{O}_2$  concentration, dye concentration, temperature, and electrolytes [103]. One detailed illustration around this amination was brought in **Figure 7**.



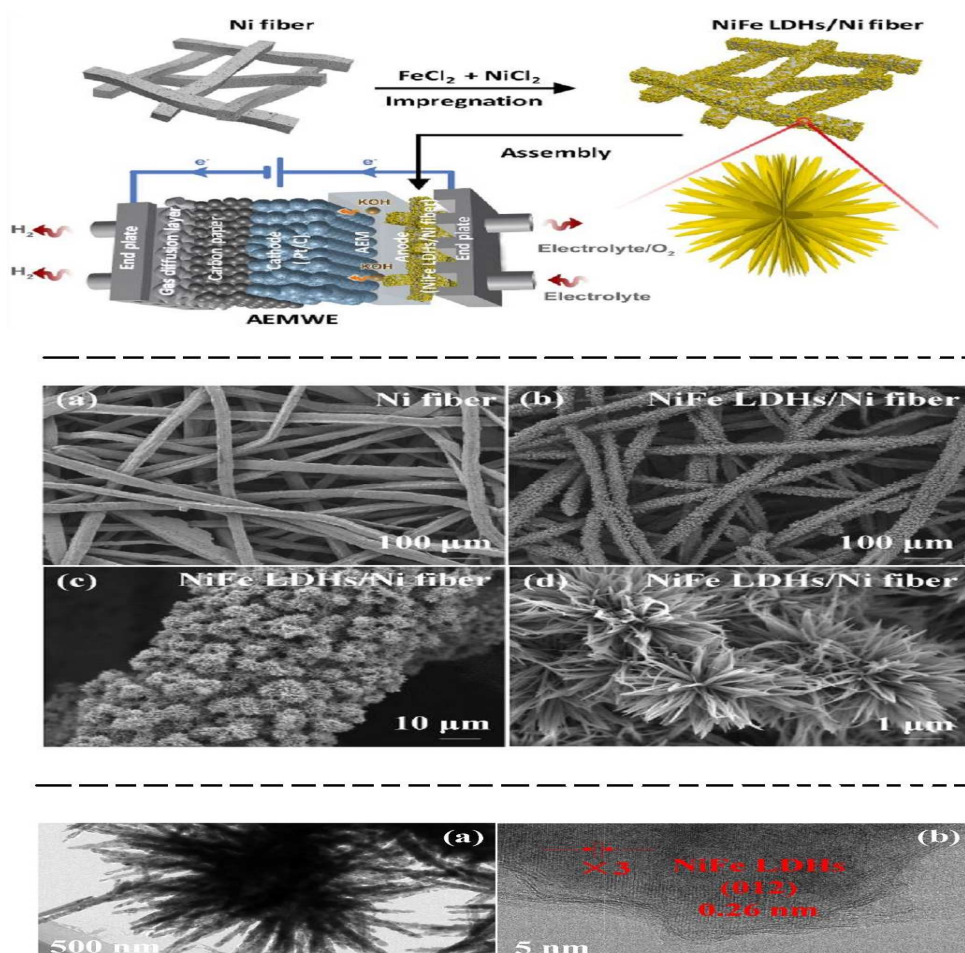
**Figure 7.** (A) Schematic illustration of the preparation process of (i) grafting of polydopamine (PDA) on waste silk fibers (wSF) and (ii) loading of iron on dopamine grafted waste silk fibers (wSF-PDA), (B) Schematic illustration of iron loading on waste silk, and (C) Schematic postulated the main mechanism of degradation of methylene blue, cationic violet X-5BLN, and reactive orange GRN dyes. Reprint with permission from [103].

## 2.7. Chloromethylation

Anion exchange membranes (AEMs) require high ion conductivity, and to achieve that, ion segregation is necessary. Ionic membranes are famous for being dense ones where ions are conveyed based on the favorite goal [16]. Chloromethylation could enhance AEMs in different ways, including raising the ion exchange capacity, enhancing resistance, and can lead to a high ion conductivity. HCl solution or a mixture of formaldehyde and HCl can lead to chloromethylation modification [16]. Although chloromethylation can offer some benefits, this modification method is quite challenging because it requires a precise optimization of the temperature, ratio, and time of reaction. Moreover, formaldehyde is a detrimental chemical agent with a negative effect on the environment, and the human body and its usage must be minimized.

A poly (ether imide)-based AEM was studied by Wang and colleagues. Numerous parameters of chloromethylation were investigated by this team and reported. The examined parameters were reaction time, reaction temperature, the allocation of chloromethylation agent, and polymer proportion. The results indicated that all studied parameters could affect chloromethylation drastically; however, the modification was governed by the concentration of the chloromethylation agent [104].

In one recent study, nickel fiber felt (Ni fiber) with a relatively flat surface as the substrate was selected, and obtained the self-supporting NiFe layered double hydroxides (NiFe LDHs) “nanoflower” cluster electrode directly and vertically grown on the Ni fiber substrate by a simple one-step impregnation method at room temperature and ambient pressure. The results show that the self-supporting NiFe LDHs/Ni fiber electrode not only has remarkable catalytic activity and stability in a three-electrode system but also serves to assemble as the anode for the anion exchange membrane water electrolyzer (AEMWE) single-cell [105].



**Figure 8.** The upper picture: Schematic preparation of the self-supporting NiFe layered double hydroxides (NiFe LDHs) as the anode of the anion exchange membrane water electrolyzer (AEMWE). The middle picture: the SEM images of Ni fiber electrode, (a) and NiFe LDHs/Ni fiber electrode (b–d). The bottom picture: TEM images of the NiFe LDHs (a,b) with 500 nm and 5 nm magnitude. Reprint with permission from [105].

### 3. Conclusions

Technology and science are marching ahead at a staggering speed, so appointing distinct boundaries between different fields and methods is almost impossible. Nowadays, the industrial world has led to severe water pollution, so purifying water is a crucial matter and needs more attention. The cheap and easy availability made the membrane an excellent tool for remediation and purification applications. Membranes vary in many aspects, and they can use

in numerous fields, but they need modification. Therefore, sorting how membranes can modify is a substantial issue, especially with a new frontier about their advantages and disadvantages. Better surface hydrophilicity, higher surface roughness, more and narrower channels, and pores, a thinner active layer, better rejection performance, higher water and solvent permeation, and robustness are merits of membrane modification.

Seven types of membrane modification were brought and discussed in this mini-review. In each part, a short explanation of the method and its benefits and drawbacks was delineated. In addition, new studies about each method were brought, and their final results were scrutinized as well. To boost the quality of the mini-review, relevant illustrations were depicted.

Overall, each method has its fortes and drawbacks, which put some limitations ahead of their applications. General modification methods like nanomaterials and surface modification have been pioneers in recent decades because they can endow different features and aspects. The new generation of nanomaterials, including MOFs, COFs, MXenes, transition metals, and nanohybrid particles, revolutionized the membrane field over past years and provided a circumstance with no limit because there is no boundary for new nanomaterials or modified ones. In addition, coupling membranes with other approaches like activated sludge or photocatalysts shed light on the new generation of membranes with promising performance. Laconically, it seems that in the coming days, the membrane field could face these rubrics:

- Focusing on the accurate mechanism of contamination rejection
- Embedding newly synthesized nanomaterials into different membrane
- Coupling membrane with other purification methods like activated carbon or sludge
- Using Ionic-liquid in the membrane, especially nanofiltration one
- Finding new interlayer coatings with better compatibility
- Promoting proton and anion exchange membrane

### Authors' contributions

All authors contributed to drafting and revising of the paper and agreed to be responsible for all the aspects of this work.

### Declaration of competing interest

The authors declare no competing interest.

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Not applicable.

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