

REVIEW

Cellulose Acetate Reverse Osmosis Membranes for Desalination: A Short Review

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ABSTRACT

Freshwater scarcity is a critical challenge that human society has to face in the 21st century. Desalination of seawater by reverse osmosis (RO) membranes was regarded as the most promising technology to overcome the challenge given that plenty of potential fresh water resources in oceans. However, the requirements for high desalination efficiency in terms of permeation flux and rejection rate become the bottle-neck which needs to be broken down by developing novel RO membranes with new structure and composition. Cellulose acetate RO membranes exhibited long durability, chlorine resistance, and outstanding desalination efficiency that are worthy of being recalled to address the current shortcomings brought by polyamide RO membranes. In terms of performance enhancement, it is also important to use new ideas and to develop new strategies to modify cellulose acetate RO membranes in response to those complex challenges. Therefore, we focused on the state of the art cellulose acetate RO membranes and discussed the strategies on membrane structural manipulation adjusted by either phase separation or additives, which offered anti-fouling, anti-bacterial, anti-chlorine, durability, and thermo-mechanical properties to the modified membranes associated with the desalination performance, i.e., permeation flux and rejection rate. The relationship between membrane structure and desalination efficiency was investigated and established to guide the development of cellulose acetate RO membranes for desalination.

1. Introduction

In the 21st century, due to climate change and further population growth, the world will be facing increasing pressures on energy and water shortages^[1,2]. Successful scientific and technological attempts to address these two shortages, especially freshwater scarcity, can also contribute toward helpful solutions on a global scale^[3]. Two strategies were implemented: one is to remediate the contaminated freshwater by means of environmental protection^[4-6], the other is to desalinate saline water,

e.g., brackish water and seawater, to achieve freshwater from ocean resources which are regarded as great and inexhaustible^[7]. Therefore, direct distillation and reverse osmosis (RO) membrane technologies are the major approaches to obtain freshwater from saline water currently^[2,8,9]. Considering huge energy assumption of the direct distillation, RO membrane technology is the most popular approach to gain freshwater, and 44 % of global freshwater by desalination was from RO plants^[8]. In the 60s and 70s of last century, two types of RO membranes: cellulose

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acetate-based membrane and polyamide-based thin-film composite (TFC) membrane, were developed rapidly and dominated all markets for desalination^[10]. Although the polyamide-based TFC membrane exhibited relatively high permeation flux and became the mainstream currently in desalination, cellulose acetate membrane with higher chlorine-resistance, high rejection rate and high durability is unique and irreplaceable in seawater desalination industry^[7].

Cellulose acetate RO membrane exhibited asymmetrical structure, as shown in Figure 1^[8].

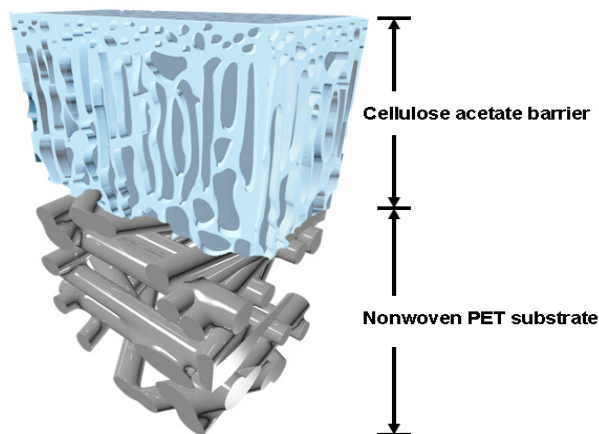


Figure 1. Representative structure of cellulose acetate RO membrane

Typically, the bottom layer of the RO membrane is polyester (e.g., polyethylene terephthalate, PET) non-woven substrate which mainly provided enough mechanical properties to the membrane; the middle and top layers are cellulose acetate fabricated by different approaches. The middle porous structure was created by phase-inversion method, where non-solvent induced a phase separation of cellulose acetate and extracted solvent (solvents) from cellulose acetate solution, and finger-shaped voids were formed after annealing treatment. The top skin layer of cellulose acetate membrane was generated by solvent evaporation and a non-porous dense layer was created for the selective separation of water molecules and sodium/chloride ions^[8]. The top barrier layer and middle porous support layer are well integrated, together with PET non-woven mat to establish a cellulose acetate RO membrane for desalination.

Currently, the major challenge for cellulose acetate RO membrane remained is water permeation flux that needs to be enhanced to meet the requirement for desalination^[11]. Every effort was made to address the problem by either adjusting the porous structure of the membrane by controlling the solvent-nonsolvent induced phase separation or creating nanocomposite top and middle layer by

forming additive-blending fabrication^[12-17]. In addition to improve the permeation flux and rejection rate, cellulose acetate RO membranes with designed structure also exhibited highly improved anti-fouling^[18-27], anti-bacterial^[28-38], anti-chlorine^[30,39], and thermos-mechanical properties^[40-49] that were beneficial to enhance the desalination performance of the membranes, which was the main topic of the review.

2. Solvent-nonsolvent Induced Phase-inversion

Cellulose acetate RO membrane fabricated by phase-inversion method was firstly demonstrated by Loeb and Sourirajan^[50] who employed cellulose acetate as the membrane material, and used formamide-acetone as the solvent and water as the nonsolvent, respectively. The phase separation behavior of the membrane preparation process was analyzed essentially by Strathmann^[51]. The phase-separation diagram of cellulose acetate-formamide-acetone-water system was typically shown in Figure 2.

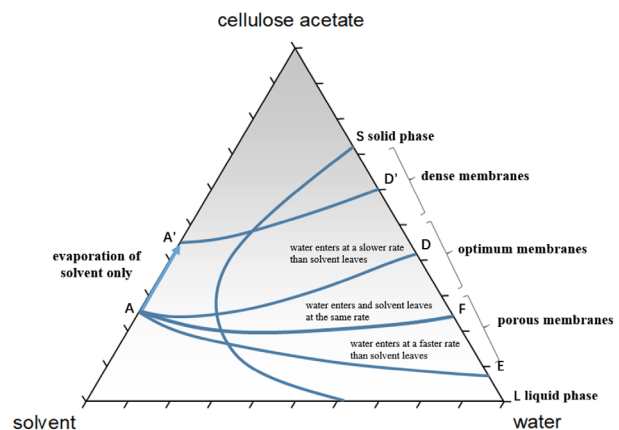


Figure 2. Phase diagram of the system acetate-formamide-acetone-water^[51]

It was clear to see that the structure of the top barrier layer and middle layer of cellulose acetate was established by the evaporation of solvent and exchange rate of solvent and nonsolvent, respectively. Therefore, the cellulose acetate RO membrane could be designed by following the phase diagram for different applications, emphasized on either high rejection rate or high permeation flux. Moreover, changing the solvent and nonsolvent compositions could further expand the categories of cellulose acetate RO membrane. Pilon et. al.^[52] adjusted the ratio of solvent and nonsolvent to lower the operating pressure and to obtain the corresponding RO membrane with high permeation flux as 7.79 L/m²h while remaining the rejection rate of 95.0 %. Ghosh et. al. also partially changed the solvent of acetone

to dioxane, and found that the permeate flux reached 10.78 L/m²h at 17.2 bar with 90.0 % rejection [53]. In Choi's work [12], with the increase of dioxane percentage, pure water flux of membranes went up. Considering salt rejection rate, the optimal volume ratio of acetone/dioxane is 1:2.9. Moreover, Manjikian et al. used tetrahydrofurfuryl phosphate instead of formamide as non-solvent to fabricate RO membrane [54], where they found that the permeation flux and rejection rate increased. The research also investigated the optimum acetone-formamide range for RO membranes. By changing acetone/water ratio, Kunst [55] developed a relatively low pressure RO membrane casting solution composition at 90 % level of salt rejection rate.

To overcome the biological attack and the compaction problem, cellulose diacetate (CDA), cellulose triacetate (CTA), and their blends were suggested for RO membrane fabrication [10]. Ebrahim et. al. used the mixture of CA and CTA extracted from Egyptian rice straw [56] as the barrier layer and the resultant RO membrane exhibited high permeation flux of 4.76 L/m²h at 14 bar and rejection rate of 93.3 %. Meanwhile, CTA was introduced onto RO membrane barrier layer as the dense composition, while the CTA-CA membrane improved the permeation flux from 4.56 to 5.44 L/m²h and the rejection rate of the membrane up to 82.74 % [57].

In addition to the factors mentioned above, annealing temperature and evaporation time are also important to

control the structure of cellulose acetate barrier layer, and therefore, improve the rejection rate of the membrane. Duarte et al. studied the effect of annealing temperature on CA-CTA membranes [58] and they found that the rejection rate increased with the annealing temperature rising up to 85 °C. Further temperature rise will drastically decrease the flux with relatively same rejection rate. Moreover, Manjikian et al. [54] changed the evaporation time from 0.25 to 3.0 min, and the rejection rate of the membrane increased at first and then decreased after the optimum evaporation time passed. Pageau [59] also investigated the effect of solvent evaporation time from 0.5 to 3.0 min: the permeate flux slightly increased within 1.0 min of evaporation, longer minutes of evaporation will cause a significant flux drop.

In summary, the dense barrier layer of cellulose acetate RO membrane is controlled by evaporation of solvent, such as acetone, and a phase inversion process. The barrier layer could be even denser by incorporation of dioxane, or involving other component, such as CTA. However, porous middle layer is mainly adjusted by the ratio of solvent-non-solvent. Herein, the increased amount of non-solvent, e.g., water, will lead to larger pore size of the middle support. Meanwhile, change of the solvent to dioxane can further improve the water permeability. The effects of parameters including barrier layer composition, solvent, nonsolvent, annealing conditions on the permeation flux (normalized by pressure) and rejection were illustrated in Table 1.

Table 1. Fabrication and performance of cellulose acetate RO membranes

Membrane type	Composition of barrier layer	Solvent	Nonsolvent	Evaporation time (min)	Annealing temperature (°C)	Permeation flux (LMH/bar)	Rejection (%)	Ref
Batch 18	CA	acetone	magnesium perchlorate solution	4.0	23.0-25.0	0.39	91.7	[60]
Batch 301				2.0	Pure water Pressure treatment	0.38	97.3	
Batch 47	CA	acetone	formamide	0.5-1.0	80.0	0.22	97.5	[54]
				0.5	81.0	0.17	98.3	
					71.6	0.67	87.1	
S-2	CA	acetone+ dioxane	formamide	2.1×10 ⁻³	81.0	0.56	90.0	[53]
	CA	acetone	THFP	10.0	75.0	0.21	96.5	[54]
			DMF	3.5	60.0	0.077	94.0	
			DMSO	3.0	62.0	0.071	85.0	
			urea solution	6.0	63.3	0.037	92.5	
			glyoxal solution	17.0	70.3	0.27	92.5	
			TEP	2.0	79.0	0.28	95.9	
			acetic acid	2.0	77.5	0.071	85.0	
			NM2P	16.0	75.0	0.081	95.0	
optimized formulation	CA+CTA	acetone+ dioxane+ acetic acid	methanol	0.5	80.0-85.0	0.68	82.7	[61]
	CTA	acetone+ dioxane	methanol	1.5	without annealing	0.38	97.3	[62]
			maleic anhydride			0.12	96.5	

Note: THFP denotes tetrahydro furfuryl phosphate

DMF denotes dimethyl formamide

DMSO denotes dimethyl sulphoxide

TEP denotes triethyl phosphate

NM2P denotes N-methyl-2-pyrrolidone

Conventional cellulose acetate RO membranes developed in the 1960s and dominated major market for desalination based on the continuous optimization of the membrane structure and composition. However, the demand on breaking through the flux limitation of current cellulose acetate RO membrane is still remained as a challenge, due to the increased human society's needs. Moreover, the trade-off between the permeation flux and rejection rate needs to be overcome in such a way that a membrane with high flux and high rejection rate will be required simultaneously. Fortunately, new nanoscaled materials such as nanoparticles, nanofibers, and nanofillers inspired a new pathway to improve the performance of the RO membranes.

3. Additives-based Nanocomposite

Nanoscaled materials, such as nanoparticles, nanofibers, nanotubes, and even 2D-nanosheets, can be integrated with cellulose acetate to form nanocomposite which may serve as barrier layer and middle support layer for RO membranes^[4]. The cellulose acetate nanocomposite RO membranes offer unique properties to the membrane, such as anti-fouling, anti-bacterial, anti-chlorine, high thermo-mechanical properties which all associated with improvement of the permeation flux and/or rejection rate in the desalination process^[63]. Moreover, these nanoscale additives may also change the structure of cellulose acetate by formation of water channels^[64] for water molecule's transportation, which directly induces high permeability of the membrane^[65], as shown in Figure 3.

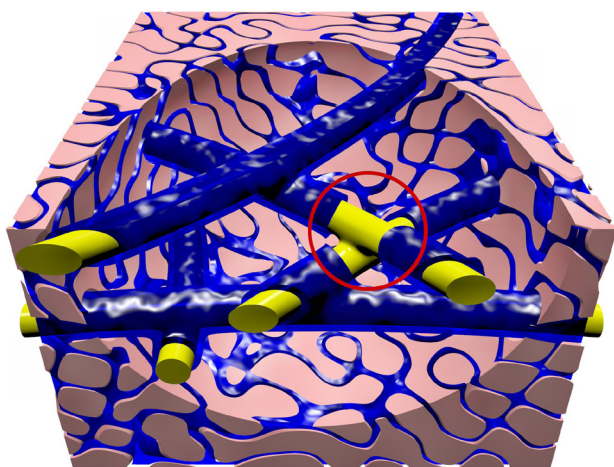


Figure 3. Schematic representation of the nature of water channels in the nanocomposite barrier layer^[64]

Cellulose nanofibers were overlapped to form 3D networks which serve as directed water channels (blue) formed by the connected tubular structure between the nanofibers and the polymer matrix (pink). The cut-out

in the red circle sketches the cross-linked nature of the nanofiber interconnects. Molecular cavities in the polymer matrix, as shown in Figure 3, which also contribute to the overall water flow through the barrier layer. Therefore, different types of nano-additives were embedded into cellulose acetate polymer matrix to enhance the membrane performance for desalination.

3.1 Anti-bacterial Properties

Typically, inorganic nanoparticles including Ag, ZnO, TiO₂, hydroxyapatite, and bio-organic species such as chitosan, as well as ammonium- and bromo-modification of cellulose acetate issued anti-bacterial properties to the RO membranes.

According to Gul's work^[66], when blending silver nitrate into CA/PEG casting solution, the fabricated membranes presented improved flux (0.31 L/m²h), salt rejection (94.5 %) and anti-E. coli properties. Similar results were proved by TiO₂ modified membranes with 95.4 % salt rejection^[67]. Sprick et al.^[68] introduced casein-coated Ag nanoparticles into cellulose acetate and fabricated RO membranes by physical blending and chemical attachment approach, the latter casted membrane exhibited 2.6 times higher rejection rate than pristine membrane without adding Ag. Moreover, when CA membranes were modified with silver nitrate by in-situ reduction^[13], the flux increased from 0.80 to 0.95 L/m²h with relatively high salt rejection rate of 95.8 % compared with pristine membranes. The modified membranes also exhibit antibacterial activity against E. coli and B. subtilis. A new composite membrane containing cellulose acetate / graphene / Ag nanoparticles / Cu nanorods (CA-G-Ag-Cu) has been employed^[69] to kill both Gram-positive and Gram-negative bacteria growth; meanwhile, both flux and salt rejection rate improvement were observed in permeability tests. Faria et al.^[70] employed graphene oxide (GO) nanosheets and Ag nanoparticles to further improve anti-bacterial properties of the membrane. As a result, the integrated GO-silver nanoparticles-CA membranes own a high rejection rate of 90 % against E. coli. In addition to Ag, ZnO nanoparticles were also used to enhance anti-bacterial properties of cellulose acetate RO membrane. Khan et. al.^[14] dispersed ZnO nanofillers in CA matrix and they found that fabricated membranes had a high Fe²⁺ uptake capacity. Further permeability results suggest that the membranes may be suitable for water treatments. According to Ohland's work^[71], incorporation of hydroxyapatite (Hap) into CA membranes by plasma treatment caused permeation flux increase from 5.85 to 7.65 L/m²h while high rejection of NaCl still remains. In Fei's work^[72], surface modified CTA-RO membranes with 3-chloro-2-hydroxy-

propyl-trimethyl ammonium chloride (CHPTAC) showed salt rejection rate of higher than 92 % with slight increase in flux and high bactericidal rates.

When antibacterial alkyl bromide modified CDA powder was used as the casting polymer^[73], both flux and rejection rate can reach up to the CTA-RO membrane. This new method of modification offers a new way for antibacterial improvements of CA membranes. Further improvement by introducing both quaternary ammonium and bromoacetyl groups^[74] to CDA has shown bactericidal rates against *E. coli* and *S. aureus* more than 99.9 %. In addition, salt rejection and permeate flux are high with 96.76 % and 17.41 L/m²h, respectively.

3.2 Anti-fouling Properties

Hydrophilic species such as graphene oxide, hydrophilic polymers, and surface-grafting with hydrophilic species may decrease surface tension and therefore offer anti-fouling properties to the membrane. Ahmad et. al.^[75] employed silica into PEG/CA membranes, it turned out that not only the fouling resistance enhanced, but also was tested an 11 % increase in salt rejection up to 92.0 %. Meanwhile, permeate flux also increased from 0.35 to 2.46 L/m²h. When blending with CA, polyvinylchloride (PVC) modified RO membranes^[15] also provided high salt rejection (93.3 %) and permeate flux of 13 L/m²h. The foulants rejection rate ranked 96.0-99.2 %. A composite CA membrane containing graphene, Ag nanoparticles and Cu nanorods^[69] shown improved membrane desalination and biofouling properties simultaneously compared with neat CA membranes. Chemical attachment of silver nanoparticles/CA membranes in Sprick's study^[68] have successfully prevented leaching of silver nanoparticles with better salt rejection and antimicrobial properties than conventional physical blending ones^[76].

The improvement of membrane hydrophilicity is a potential advantage over anti-protein and hydrophilic foulants. According to Morsy's study^[77], surface grafting of 2-acrylamido-2-methylpropanesulfonic acid (AMP-SA) suggested an increase in surface hydrophilicity with high salt rejection of 99.03 % and 6.00 L/m²h of flux. In Chen's research^[65], GO embedded CTA flat sheet membrane exhibited higher hydrophilic property with increased flux from 1.67 to 4.74 L/m²h. Simultaneously, salt rejection rate decreased at the same time which may due to the cause of internal water channels formation allowing passage of water and salt. Similar conclusions were also drawn in Shi's work^[16] when it comes to GO-CA membrane.

In terms of surface modification, surface-initiated polymerization of 2-hydroxyethyl methacrylate (pHEMA)

reported by Worthley^[78] had a 24 % improvement in resistance to seawater biofouling with only 6 % decrease in salt rejection and water flux. Instead of modification of CA-RO membranes to enhance anti fouling property, Sachit^[79] suggested that pretreatment of feed water by ultrafiltration for improving the fouling problem should also be noted.

3.3 Anti-chlorine Properties

The addition of CNT can improve the chlorine resistance of cellulose acetate RO membranes. Wasim et. al.^[39] incorporated sepiolite and polyvinylpyrrolidone (PVP) into cellulose acetate membrane which presented high NaCl and MgSO₄ rejection rate of 94 % and 92 % respectively after 2 hours of chlorination. Shafiq et. al.^[67] employed TiO₂ nanoparticles to enhance anti-chlorine properties of the membrane and they found that the presence of TiO₂ exhibit negligible decline in salt rejection, with 15wt % of TiO₂ provide high salt rejection rate of 95.4 %.

3.4 Improved Thermo- and Mechanical Properties

Nano-additives such as CNT, GO, and silica nanoparticles can also improve the thermal or mechanical properties of cellulose acetate RO membranes.

Generally, GO modified CA membranes^[65,80] have shown improved thermal stability with increasing amount of GO which induced stronger interactions between nanosheets and cellulose acetate. Such conclusions are also been proved by polyvinylchloride (PVC) blend CA membranes^[15,81,82], both thermal and mechanical properties improved with high rejection rate and relatively high permeate flux. According to Sabir's study^[83], when blending surface engineered (SE) multi-walled carbon nanotubes (MWCNTs), modified CA membranes exhibited higher weight loss temperature indicating an improvement in thermal stability with more MWCNT content with corresponding salt rejection rate of 99.8 %. Mechanical stability increased with embedding of silica nanoparticles^[75] in CA membranes from 1 to 4 % (w/v), the salt rejection and permeation flux also increased as mentioned before this section. Similar results showed the improvement in thermal properties^[84] alongside with the maximum salt rejection of 95.0 %. In Sabir's work, both tensile strength and Young's modulus increased with more fumed silica particles^[85] in RO membranes.

Besides, long-term filtration process can testify the stability of modifiers as well as durability of membranes' anti-fouling property^[86]. It has been proved by Chede^[76] that after 26 days of filtration, the salt rejection drop of ca-

sein-Ag CA (2.8 %) is smaller than plain CA membranes (18.2 %). The permeation flux decline was 18 % compared to other membranes. Such results indicate that the introduction of Ag nanoparticles in membrane structure was efficient and durable. PVC/CA membranes prepared by Gendi^[81] were proved to be able to work for long hours without breaking. During the 36 days' durability test, the rejection rate remained at the level of 98 - 99 %, with permeation flux of 37.2 L/m²h.

3.5 Improved Permeability and Rejection Rate

Some additives, such as modified coal^[87,88] can improve the permeation flux and rejection rate of the composite by simply blending with cellulose acetate. The composite membrane exhibited high flux of 38.70 L/m²h and high rejection rate of 94.0 %, compared with the pristine CA membrane. Besides, when 0.005 wt% GO was used as an additive in CA membranes^[16], the water flux reached 13.65 L/m²h, 2.3 times higher than pristine CA ones, with 82.03 % salt rejection rate. Meanwhile, by surface grafting of 2-acrylamido-2-methylpropanesulfonic acid (AMP-SA)^[77], CA-RO membranes reached higher rejection rate of 99.03 % and water flux of 6.00 L/m²h. Other surface grafting of 2-acrylamidopropane-2-methyl sulfonic acid^[56] increased water flux from 4.76 to 8.30 L/m²h and rejection rate reached 93.5 %. In Perera's study^[89], by adjusting CA concentration together with swelling and annealing treatment, CA thin film composite's salt rejection reached 94.0 % without significant sacrifice of water flux.

All these cellulose acetate composite RO membranes demonstrate high permeation flux and high rejection rate due to the integration of additives, as summarized in Table 2.

Table 2. Modified CA-RO membranes with enhanced desalination efficiency

Membrane	Additives	Flux (LMH/bar)	Rejection (%)	Anti-bacteria	Anti-fouling	Anti-chlorine	Thermo-mechanical strength	Ref
1	PEG200 + SiO ₂	0.165	98.4				×	[85]
2	PEG300 + SiO ₂	0.4	95.0				×	[84]
3	PEG400 + TiO ₂	0.02	95.4	×		×	×	[67]
4	PEG400 + MWCNT	0.15	99.8				×	[83]
5	PEG600 + SiO ₂	3.22	92.0	×			×	[75]
6	PEG600 + Ag	0.06	94.5	×				[66]
7	PEG600 + AgNO ₃	0.15	95.8	×				[13]

8	PEG600 + MWCNT	0.15	99.8					×	[83]
9	Modified coal	0.22	94.0						[88]
10	MWCNT	2.32	90.6						[12]
11	Ca-sein-Ag	1.44	93.0	×				×	[41]
12	Graphene oxide	2.6	90.0					×	[80]
13	Graphene + Ag-Cu	3.12	98.0	×					[69]
14	Modified coal	1.75	94.0						[88]
15	Grafting AMPS	0.59	93.5						[56]
16	Br-CA	0.81	95.6	×				×	[73]
17	PVC	0.93	99.0					×	[81]
18	PVC	0.43	93.3		×			×	[15]
19	PVP + sepiolite	4.99	98.3	×			×		[39]
20	Hapf	0.51	89.2						[71]
21	CHPTAC	2.28	92.0	×				×	[72]
22	DMOA	1.12	96.8	×				×	[74]
23	AMPSA	0.4	99.0						[77]

4. Conclusion and Perspective

Cellulose acetate RO membranes were fabricated by phase-inversion approach and employed to brackish water and seawater desalination to address the critical issue of scarcity freshwater on a global scale. The generation mechanism of cellulose acetate top barrier layer and sub-porous support layer was discussed comprehensively based on phase separation diagram of cellulose acetate-formamide-acetone-water system. The structure of dense barrier and porous substrate could be controlled by fine-tuning the solvent-nonsolvent induced phase separation of cellulose acetate, where high permeability and high rejection rate of the RO membranes could be designed and fabricated for desalination performance. The birth of new nanoscaled materials, such as nanoparticles, nanofibers, nanotubes, and nanosheets would break up the bottle neck limit which is the trade-off relationship between membrane permeability and selectivity. In this case, the nanocomposite RO membranes with both high permeate flux and rejection rate could be prepared for efficient energy saving desalination. Moreover, the nanoscaled additives also offer anti-fouling, anti-bacterial, anti-chlorine properties with high thermo-mechanical strength of CA-RO membranes which are all beneficial in improving desalination performances.

However, the fundamental physics and chemistry principles behind membrane formation mechanisms need to be

further explored, especially for the purpose of overpass the trade-off between the permeation and rejection rate which hinders the development of RO membranes. Considering the unlimitness needs and requirements in human's society, the revolution of industry and growth of population require creation of new concept RO membranes with high permeation flux, high rejection rate, high durability and low cost, which is the direction we should follow in desalination industry.

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