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Smart membranes for oil/water emulsions separation: A review

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ABSTRACT

Oily wastewater poses a significant impact on both environments and human societies. Especially, the treatment of oil/water emulsions for separating oil from water is challenging due to the high stability of oil/water emulsions. Smart membranes, known as stimuli-responsive membranes, are one of the emerging technologies that have been paid wide attention for separating oil/water emulsions in recent years. Smart membranes possess the unique features of switchable wettability between hydrophilicity and hydrophobicity after being triggered by external stimuli and have desired anti-fouling properties. This review summarizes the development of smart membranes for oil/water emulsions separation during the past five years (2018 – present). It was found that solvent stimuli-responsive membranes are the most popular type of smart membranes for oil/water emulsions separation. For multi-stimuli-responsive membranes that can respond to more than one stimulus, future research should focus on developing appropriate fabrication strategies to increase the separation and anti-fouling performances of the membranes. Additionally, surface coating, surface grafting, and copolymer blending are the most popular methods for smart membranes fabrication. However, these methods might not be universally applicable to the different types of stimuli-responsive membranes.

1. Introduction

Water pollution has become an increasingly critical challenge to the environment due to the growth of the global economy, fast industrialization, and increasing population. Oily wastewater, recognized as a major type of water pollution, is generally produced from industries, oil spills, vehicle transportation, and household sewage [1,2]. Oils under excessive quantities are often harmful to the surrounding environment. These compounds threaten aquatic and plant life, killing fish, leaving birds and mammals susceptible to hypothermia or overheating, and adversely affecting plant growth. Oily wastewater comes in different forms (e.g., water-in-oil emulsions and oil-in-water emulsions), which require different oil and grease removals methods, such as adsorption [3,4], dispersion [5,6], biological treatment [7,8], flotation [9,10], and burning [11]. However, these technologies are subject to potential limitations: low efficiency, low stability, high reagent costs, high energy consumption, and secondary pollution [12,13]. Desirable oil/water emulsion separation technologies have low costs, simple operation, high permeation, high separation efficiency, excellent stability, and no secondary pollution during fabrication and separation processes [14,15]. Membrane separation has been studied widely because of such advantages as simplicity, high permeation, high efficiency, low energy consumption, good stability, no secondary pollution, environmental friendship, and wide applicability [16–18].

Effective oil/water separation with the membrane technology is closely related to surface wettability (i.e., hydrophobicity, hydrophilicity, oleophobicity, and oleophilicity) and morphology. These factors directly affect how liquids respond when in contact with membrane surfaces [19]. For oil/water separation technologies, two typical types of

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Nomenclature

CA	contact angle
CF_3AZO	7-[(trifluoromethoxyphenylazo)phenoxy] pentanoic acid
CST	critical solution temperature
DMAEA	2-(dimethylamino) ethyl acrylate
HEMA	2-hydroxyethyl methacrylate
HFBMA	fluorobutyl methacrylate
ILs	ionic liquids
LBL	layer-by-layer
LCST	lower critical solution temperature
NPs	nanoparticles
OTES	octyltriethoxysilane
P(3-MTH)	poly-3-methylthiophene
PA	polyamide
PAN	polyacrylonitrile
PCL	polycaprolactone
PDA	polydopamine
PDEAEMA	poly(N,N-diethylaminoethyl methacrylate)
PDHH	PDMAEMA-co-PHEMA-co-PHFBMA
PDMAEMA	poly(N,N-dimethylaminoethyl methacrylate)
PDMS	polydimethylsiloxane
PFDTMS	1H,1H,2H,2H-perfluorodecyltrimethoxysilane
PGMA	poly (glycidyl methacrylate)
PHEMA	poly(2-hydroxyethyl methacrylate)
PHFBMA	poly(Hexafluorobutyl methacrylate)
PILM-1	poly (1-vinyl-3-butylimidazolium acrylate)-based membrane
PILM-5	poly (1-vinyl-3-octylimidazolium hexafluorophosphate)-based membrane
PILMs	PIL-based membranes
PILs	poly(ionic liquid)
PNIPAM	poly(N-isopropylacrylamide)
PVCL	poly(N-vinylcaprolactam)
PVDF	polyvinylidene fluoride
SDS	sodium dodecyl sulfate
SI-ATRP	surface-initiated atom-transfer radical polymerization
SiO_2	Silicon dioxide
SnO_2	tin (iv) oxide
SSM	stainless steel mesh
TFOA	3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl acrylate
$TFSI^-$	bis(trifluoromethane)-sulfonimide
TiO_2	titanium dioxide
TMC	trimesoyl chloride
T-ZnO	tetrapod-ZnO
UCST	upper critical solution temperature
UV	ultraviolet
V_2O_5	vanadium(V) oxide
Vis	visible light
WCA	water contact angle
ZIF	zeolitic imidazolate framework
ZnO	zinc oxide

materials have been defined and applied, i.e., filtration materials such as mesh, membrane, filter, and film, and absorbent materials such as porous media, powder/particle, gel, and nano-composite [20]. Membrane filtration is one of the most effective methods to treat oily wastewater. Especially the wetting properties, also known as wettability, of the membrane surface can be elaborately tailored by switching between hydrophilic-oleophobicity and hydrophobic-oleophilicity, thus efficiently enhancing the selectivity during oil/water separation [21,22].

Membrane surface properties, such as morphology, structure, surface chemistry, resistance in complex environments, anti-corrosion, and anti-fouling performances, are often considered for membrane designing [23–25]. To better coordinate membrane surface properties for effective oil/water separation, three kinds of unique wettability materials, i.e., oil-removing materials, water-removing materials, and smart materials, have been employed to fabricate membranes [26,27]. The oil-removing materials allow oil to easily pass through the membrane and retain water on the superhydrophobic/superoleophilic surface, which are usually used for heavy oil/water separation (i.e., heavy crude oil, viscous crude oil, and diesel fuel) [28,29]. For example, Fan et al. prepared a graphene-wrapped polyphenylene sulfide fiber membrane with enhanced chemical resistance and hydrophobicity for efficient oil/water separation and rapid crude oil adsorption under all-weather conditions [28]. In another study, polyvinylidene fluoride (PVDF) membrane coated with eggshell powders was developed by vacuum pumping eggshell powders and sodium alginate suspensions onto a PVDF membrane surface for separating high-viscosity crude oil-in-water emulsions [30]. However, oil can easily damage or inhibit the superwetting surfaces of oil removing materials [31]. On the other hand, the water-removing materials allow water to flow through and retain oil on the surface with superoleophobic/superhydrophilic properties, usually used for light oil/water separation (i.e., gasoline, kerosene, petroleum ether) [32–34]. Liu et al. fabricated cigarette filter coated meshes using a facile electrospinning technique for separating immiscible oil/water mixture (light or heavy oil/water combinations) on-demand (water-in-oil or oil-in-water emulsions) [35]. However, oil-removing and water-removing materials are featured by a single type of wettability on the membrane surface. The obtained membranes usually have various limitations, such as poor oil-water selectivity and low porosity [23].

In recent years, smart materials for controllable separation have been developed with switchable wettability for use in complex situations. The membranes tailored by using smart materials (i.e., smart membranes) provide a promising solution for efficiently separating complex oil/water mixtures [26,36]. Smart membranes, also known as responsive membranes, have the potential to improve membrane separation efficiencies and anti-fouling performance in complex oil/water emulsions environments with various external stimuli, such as pH [37,38], light-ultraviolet (UV) radiation [39,40], temperature [41,42], gas [43,44], electro-voltage [45], multi stimuli [46,47]. Moreover, smart membranes with anti-fouling and self-cleaning properties can mitigate the degradation of membrane separation performance [48–50]. Smart membranes can sense, process, and respond to environmental signals, thus reacting to environmental changes in a controlled manner [48]. For example, a magnetic pH-responsive electrospun nanofibrous membrane was fabricated via electrospinning and dip-coating for on-demand oil/water separation [38]. Liu et al. prepared a thermo-responsive membrane by grafting poly(N-isopropylacrylamide) (PNIPAM) with the surface-initiated atom-transfer radical polymerization (SI-ATRP) method [41]. Li et al. developed CO_2-responsive cellulose nanofiber aerogels by grafting poly(N,N-dimethylaminoethyl methacrylate) (PDMAEMA) polymer brushes via surface-initiated atom transfer radical polymerization for switchable oil/water separation [51]. However, despite the advantages mentioned above, smart membranes still suffer from the limitations of complex synthesis, high material costs, high energy consumption, etc. Various reviews on smart membranes have been published, e.g., the recent developments in stimuli-responsive smart gating membranes [52], smart membranes based on two-dimensional materials for various applications [53], and smart membranes for water purification [54]. A systematic comparison of different types of smart membranes will be desired to improve and develop smart membrane technologies.

The principle of smart membranes for oil/water separation is related to membrane wettability, characterized by the contact angles (CA) of

liquid droplets on the membrane surface [55]. On an ideal smooth surface in air, the CA (θ) is determined by Young's equation [56]. However, the actual surface is rough. Wenzel equation and Cassie-Baxter equation were developed and used for homogeneous wetting states and heterogeneous wetting states, where there is an intrinsic wetting threshold (θ*) [57,58]. For a smooth surface with a certain chemical composition in which CA (θ) is smaller than θ* (θ<θ*), increasing roughness can enhance its wettability for superhydrophilicity (i.e., θ~0°); conversely, if θ>θ*, superhydrophobicity (i.e., θ~150°) can be achieved by roughening surface. Similar to the in-air situation, underwater oil wettability of (super)olephobicity and (super)oleophilicity can also be achieved. Consequently, membranes with selective "liquid removing" ability can be designed by manipulating surface roughness and surface energy (chemical composition). Superhydrophobic and superoleophilic "oil removing" materials can be fabricated, allowing oil to pass through the membrane while repelling water on the membrane surface. Similarly, superhydrophilic and superoleophobic "water removing" materials can be fabricated to selectively let water pass through the membrane while repelling oil [59–61].

In this regard, we systematically reviewed the recent development of smart membranes or stimuli-responsive membranes for oil/water emulsions separation during the past five years, focusing on fabrication strategies and respective pros and cons. We searched the relevant literature published in the last five years using the keywords "smart membranes", "switchable membranes", "controllable membranes", "reversible membranes", "responsive membranes", "membrane", "fiber", "oil water emulsions", "oil water mixture", and "oil water separation", through various databases, including Elsevier, Wiley, Springer, and American Chemical Society. Finally, viewpoints on existing limitations and potential solutions for future research and development were also drawn.

2. Types of smart membranes for oil/water emulsions separation

Smart membranes are made of stimuli-responsive materials and have unique properties (e.g., anti-fouling and self-cleaning) and switchable wettability on the surface between hydrophobicity and hydrophilicity and vice versa triggered by external stimuli. The external stimuli can be divided into physical, chemical, and multi-ones. Typical physical stimuli include temperature, light, electric, and magnetic. While chemical stimuli include pH, ion, and solvent (Table 1). Multi-stimuli is with two or more of the environmental stimuli [49]. A stimuli-responsive surface can be created by incorporating corresponding polymers or particles on the membrane surface, thus introducing functional groups for the wettability switch. The obtained membranes could achieve unique properties for on-demand oil/water separation [62,63].

The numbers of publications associated with smart membranes for oil/water separation are shown in Fig. 1., together with the breakdown for membrane types. From the collected 305 publications, solvent stimuli-responsive membranes are the most studied type of smart membranes for oil/water emulsions separation, followed by the pH stimuli-responsive membranes and multi-stimuli-responsive membranes.

Table 1
The example of stimuli-responsive polymers for each type of external stimulus.

The external stimuli	Example of stimuli-responsive polymers
Physical stimulus	
Light	inorganic oxides polymers, organic polymers, or semiconductors, such as TiO_2, ZnO, SnO_2, and V_2O_5
Temperature	PNIPAM, PVCL, and PDMAEMA
Electric field	polythiophene, polypyrrole, and polyaniline
Magnetic field	superparamagnetic nanoparticles
Chemical stimulus	
pH	acidic polymer groups: carboxyl, phosphate, and boronic groups basic polymer groups: amines and pyridines
Ion	polyelectrolyte, PILs, and poly(acrylic acid)
Gas	guanidine, amines, and amidines, such as PDEAEMA and PDMAEMA
Solvent	polar or non-polar functional group polymers

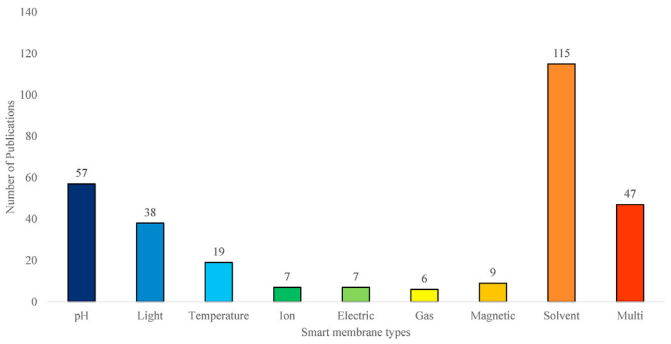

Fig. 1. Publications of smart membranes for oil/water separation in recent five years (2018- present).

2.1. pH stimuli-responsive membranes

In the past five years, pH stimuli-responsive membranes (Fig. 1.) are one of the most reported types of smart membranes due to their advantages such as excellent switchable wettability, excellent reversibility, rapid response, easy operation, simple equipment, including low energy requirement for operation, good reusability, and wide applications [64,65]. pH stimuli-responsive membranes contain acidic or basic polymer groups on their surface. These polymer groups receive or release protons depending on the pH value of the surrounding environment, which can change the wettability of the membrane surface and switch from a hydrophilic state to a hydrophobic state and vice versa (Fig. 2.) [66–69].

The acidic polymer groups most widely added on pH stimuli-responsive membranes include carboxyl, phosphate, and boronic groups. The membranes become negatively charged when protons are released in a basic environment. Basic polymer groups on the membrane, including amines and pyridines, become positively charged in an acidic environment [63,64]. Changing membrane surface charges would increase surface energy and allow water molecules to be trapped and pass through the membrane surface while repelling the permeation of oil droplets on the membrane surface. When membrane surface charges balance, surface energy will reduce, and the membranes become more hydrophobic, which allows oil to pass through and repel water on the membrane (Fig. 2.). For example, polydopamine (PDA) and cystamine dihydrochloride were grafted on a fabric surface to achieve a thiol-functionalized fabric. When pH changed from acid to alkaline, the as-prepared fabric surface exhibited a rapid switch of wettability from superhydrophobicity to superhydrophilicity [31]. Wang et al. coated a stainless-steel mesh with copolymer PDMAEMA-co-PHEMA-co-PHFBMA (PDHH) and Silicon dioxide (SiO_2) nanoparticles. The mesh could be switched from a hydrophilic state to a hydrophobic state once pH was above 7, while the wettability of the mesh changed from hydrophobicity to hydrophilicity for pH <7. Because PDMAEMA segments on the mesh exhibited pH responsiveness, 2-hydroxyethyl methacrylate (HEMA) segments raised the hydrophilicity by stretching the structure of PDHH. In contrast, fluorobutyl methacrylate (HFBMA) segments reduced the energy of the surface. All of these worked to make water pass through the mesh [70]. Copolymers of 2-(dimethylamino) ethyl acrylate (DMAEA) and 3,3,4,4,5,5,6,6,7,7,8,8,8-tridecafluorooctyl acrylate (TFOA) were also grafted on beadlike porous PS fibrous membranes. At pH < 2, DMAEA changed the wettability of the membrane surface from hydrophobicity to hydrophilicity by swelling itself, allowing water to pass through [71].

It has been shown that pH stimuli-responsive membranes exhibited highly controllable oil/water emulsions separation performance with a high separation efficiency of 99.95%, while the oil purity after separation was >95% [67]. Moreover, the membranes exhibited anti-fouling

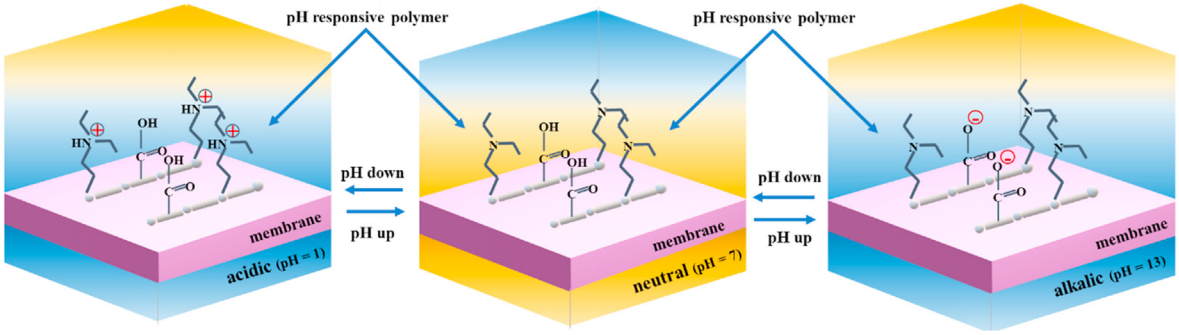

Fig. 2. Schematic diagram of the wettability change on membrane surface after being triggered by pH.

properties in various oil/water emulsions such as toluene, hexane, gasoline, kerosene, and chloroform [45,71,74]. After multiple application cycles, pH stimuli-responsive membranes could still achieve more than 98% of oil/water emulsions separation efficiency and excellent anti-fouling performance [75–78]. It also exhibited excellent durability, chemical stability, reusability, and self-cleaning [79,80]. However, pH stimuli-responsive membranes also have various disadvantages, such as secondary pollution (e.g., the addition of acidic or basic solutions for triggering membranes) and high operation costs [13,45]. Moreover, some pH-responsive polymer materials are insensitive, and response processes often take time, which may also limit their use in industries [31]. The application of pH stimuli-responsive membranes needs a pre-wetting process before separation [13], and salts, which are produced by chemical reactions, can be accumulated in the system due to the addition of acid or base, suppressing effective wettability switching after a few cycles [81,82].

2.2. Light stimuli-responsive membranes

Light stimuli-responsive membranes, also known as photosensitive membranes, exhibit a transition of surface wettability after UV or visible light (Vis) irradiation. Under dark or Vis illumination, the irradiated surface can quickly revert to its initial state. This transformation procedure can be repeated numerous times (Fig. 3) [36]. Light stimuli-responsive membranes have many advantages, such as remarkable physical, chemical, and thermal stability, and are suitable for repetitive use. Additionally, the membranes can be readily controlled based on the adjustment of wavelength, time, intensity, and direction of the light source [59,83]. Moreover, light stimuli-responsive membranes feature high photosensitivity, low device complicacy, easy fabrication, easy operation, low cost, excellent anti-fouling, self-cleaning, and low toxicity [84–88]. However, light stimuli-responsive membranes require a long time for the response and recovery process [89]. A complicated design is also required to ensure the complete exposure of the membranes to light [90].

In terms of the fabrication of light stimuli-responsive membranes, inorganic oxides, organic polymers, or semiconductors, such as titanium dioxide (TiO_2), zinc oxide (ZnO), tin(iv) oxide (SnO_2), and vanadium(V) oxide (V_2O_5), have been added onto the membrane surface. These materials can switch hydrophilicity and hydrophobicity of the membrane surface, similar to the mechanism of pH stimuli-responsive membranes. After UV or Vis irradiation, the polymers on membrane surface will undergo changes in charges and chain structure. This phenomenon allows water molecules to be trapped and to pass through while repelling oil droplets on the membrane surface and vice versa (Fig. 3) [54,59,83].

Especially, azobenzene exhibits great reversible photoisomerization of dipole moment and morphology. It can transform between *trans*- and *cis*-structures under UV/Vis light illumination (e.g., changing the wettability of dipole moment of azo groups with UV/Vis irradiation onto the membrane surface) [40]. Chen et al. grafted azobenzene groups on a SiO_2 roughened polypropylene membrane and found that the membrane could switch wettability between hydrophobicity and hydrophilicity after being triggered by UV/Vis light irradiation. The light-responsive polymers on the membrane exhibited hydrophobic properties in the trans state of 7-[(trifluoromethoxyphenylazo)phenoxy] pentanoic acid (CF_3AZO). Then, the light-responsive polymers on the membrane could change into the cis state under UV (365 nm) irradiation, exhibiting hydrophilicity, and could reverse to the trans state under Vis (440 nm) [40]. Yang et al. decorated a polyamide (PA) mesh with tetrapod-ZnO (T-ZnO), polydimethylsiloxane (PDMS) hydrophobic coating, and 4-Aminoazobenzene moieties. The as-prepared mesh exhibited reversible light-responsive wettability between hydrophobicity and hydrophilicity under UV/vis illumination: changed from hydrophobicity to hydrophilicity under UV light and from hydrophilicity to hydrophobicity under Vis light [83].

Light stimuli-responsive membranes have been reported for their good separation performance and efficiency. For example, the flux of Zeolitic imidazolate framework (ZIF-8)/graphene oxide membrane under light irradiation was 1.5 times higher than without light irradiation while exhibiting an ultra-high oil/water separation efficiency of

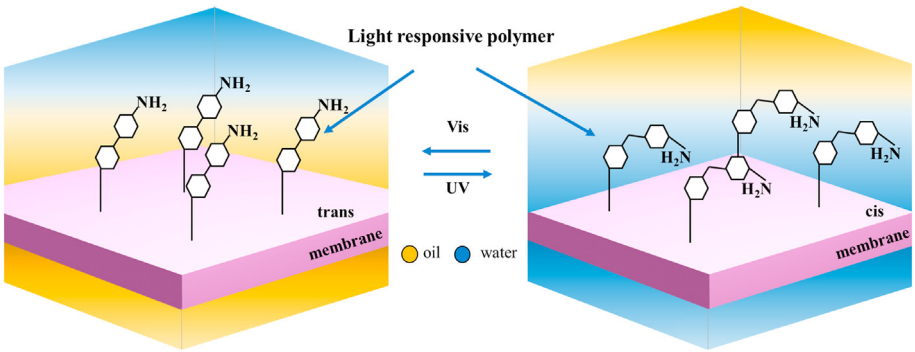

Fig. 3. Schematic diagram of wettability change on membrane surface after being triggered by light (UV/Vis).

99.99% [86]. In another research, by adding cobalt molybdate with bismuth molybdenum oxide, a heterojunction membrane achieved a high oil/water separation efficiency of 99.67% and simultaneously removed water-soluble organic pollutants and insoluble oils from oily wastewater, exhibiting excellent stability and recyclability [89]. Light stimuli-responsive membranes also showed outstanding chemical tolerance and mechanical stability [40]. After UV/light irradiation, it was shown that the membranes showed high underwater oil contact angles, water fluxes, and high stability after multi cycles use. After ten cycles of oil/water mixture separation, the membrane still had a flux recovery ratio of more than 95% [77,79]. For various emulsions (i.e., petroleum ether, toluene, *n*-hexane, 1,2-dichlorethane, and *n*-hexadecane), the membranes exhibited a good oil/water emulsions separation efficiency of up to 99.1% and high fluxes up to 654 L m^{-2} h^{-1} [84].

Light stimuli-responsive membranes with self-cleaning properties have also been developed. Yue et al. fabricated a membrane using ZIF-8/graphene oxide composite via a layer-by-layer (LBL) method, which showed superior superoleophobicity under visible light and underwater anti-fouling properties [86]. The nitrocellulose membrane was fabricated by grafting TiO_2 nanoparticles on the membrane surface to produce tunicate cellulose nanocrystals, which exhibited rapid self-cleaning properties against contamination (5w% oleic acid in ethanol solution) by changing its superhydrophilicity and underwater superoleophobicity after UV irradiation [87].

2.3. *Temperature stimuli-responsive membranes*

Temperature stimuli-responsive membranes or thermo-responsive membranes typically involve a change in polymer structure on their surface around a critical solution temperature. This membrane type can achieve a high oil/water separation efficiency of over 98% [91,92], stable recyclability, excellent chemical tolerance, and temperature stability [93,94]. Thermo-responsive polymers respond to a critical solution temperature (CST) which can be divided into a lower critical solution temperature (LCST) and an upper critical solution temperature (UCST). In the case of LCST, the thermo-responsive polymer chains on the membrane surface will expand, stretch, or be swollen in solution at temperatures below its LCST. While at the temperature above LCST, the thermo-responsive polymer chains will separate from other chains and shrink or collapse.

For the case of UCST, the thermo-responsive polymer chains will follow an opposite temperature dependence to LCST (Fig. 4.) [54,95]. At temperatures below the UCST, the thermo-responsive polymer chains will get separated from other chains and shrink or collapse onto the surface of membrane. The polymer chains will expand, stretch, or be swollen in the solution at temperatures above its UCST. The transition of thermo-responsive polymer chain structure influences the membrane's pore size by swelling or collapsing the thermo-responsive polymer chains on the membrane, which changes the water flux through the membrane. When the temperature is below the LCST of the membrane, temperature-responsive polymers are in a swollen state, thus reducing the membrane pore size and leading to hydrophilicity. When the temperature is above the LCST of the membrane, temperature-responsive polymer chains are in a collapsed state, enlarging membrane pores and leading to hydrophobicity.

Similarly, temperature-responsive polymers with UCST behavior will collapse when the temperature is below UCST and will be swollen when the temperature is above UCST. Changing the membrane pore size will lead to water flux passing through membrane pores. Although the surface is hydrophobicity, the enlarged membrane pores are the main reason for a higher water flux obtained under the temperature above the LCST compared to the temperature below the LCST. Accordingly, the oil/water separation efficiency will decrease as the temperature increases due to the increase in water flux [54,96]. However, thermo-responsive polymers with the UCST behavior in water are quite rare, and most researches focus on polymers with the LCST behavior [97].

Temperature stimuli-responsive polymers (e.g., PNIPAM, poly(N-vinylcaprolactam) (PVCL), PDMAEMA) have various levels of LCST thermal responsiveness. PNIPAM is the most commonly used temperature stimuli-responsive polymer for smart membranes fabrication because of its simplicity in fabrication, low cost, and appropriate LCST of about 32–33 °C [98]. For example, PNIPAM brushes were grafted on a polycaprolactone (PCL) porous membrane: at a temperature below the LCST (20 °C), PNIPAM brushes were swollen in water, and the membrane exhibited hydrophilicity; at a temperature higher than the LCST (50 °C), PNIPAM brushes collapsed in the water and exhibited hydrophobicity [41]. In another study, the polyacrylonitrile (PAN) nanofibrous membrane was added with PNIPAM and TiO_2 nanoparticles to obtain temperature stimuli-responsiveness. The membrane exhibited amphiphilic properties, i.e., changing to hydrophilicity at a temperature below its LCST (25 °C) and changing to a hydrophobicity state when the temperature above its LSCT (50 °C) [92]. Similarly, ZnO nanoparticles and octyltriethoxysilane (OTES) were grafted on cotton fabric with poly (glycidyl methacrylate) (PGMA) brushes. It was shown that the water contact angle (WCA) of the surface was 75° at 20 °C, exhibiting hydrophilicity; the WCA of the surface was 165° at 78 °C, exhibiting superhydrophobicity [91].

Temperature stimuli-responsive membranes utilize surface energy change and thermo-responsive polymer chain structure transformation depending on temperature. This phenomenon is the basis for forming

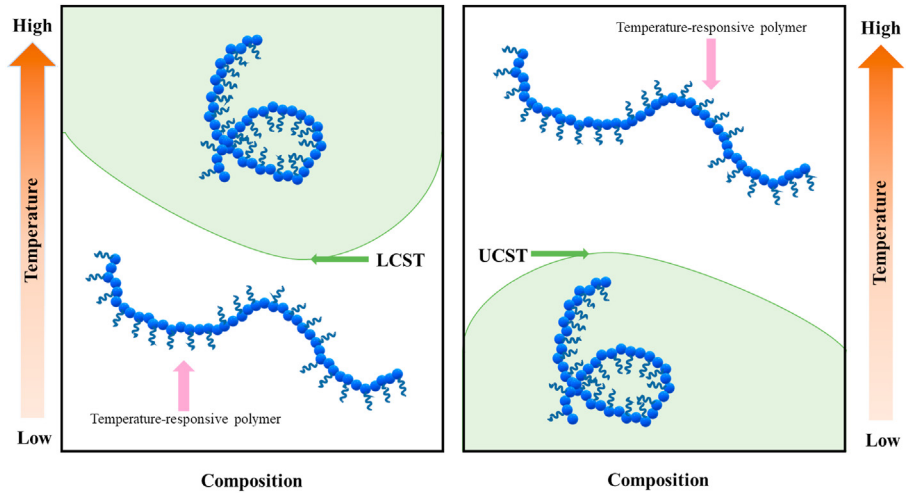

Fig. 4. Schematic diagram of composition changes of temperature-responsive polymer chains when temperature changes.

thermo-responsive nano valves on the membrane surface under temperature triggering [96,99]. Moreover, temperature stimuli-responsive membranes are often featured by low cost, no secondary pollution, and easy applicability without chemical addition to the separation system and thus less contamination to the membrane surface with chemicals [100,101]. Additionally, the membrane can also exhibit excellent recyclability. For example, a tetraethyl orthosilicate and tetrabutyl titanate-based polyvinylpyrrolidone nanofiber membrane were added with SiO_2, TiO_2, Si, and Zn nanoparticles on the surface. The membrane exhibited excellent recyclability after nine cycles of oil/water mixtures (toluene, kerosene, petroleum ether, hexane, and chloroform) separation with efficiencies of more than 99.5%, and exhibited stability in various harsh conditions (e.g., strongly acidic and alkaline conditions) [102]. However, temperature stimuli-responsive membranes have the weakness of slow response time, and the application of the membrane can incur significant energy consumption due to the need for stable temperature control during the separation process [103,104].

2.4. Ion stimuli-responsive membranes

Ion stimuli-responsive membranes exhibit switchable wettability according to ionic strength or counterions, reversible by cation/anion exchange or adding an ion on membrane surfaces. Polymers containing charged groups, e.g., polyelectrolytes, poly(ionic liquid) (PILs), and poly(acrylic acid), are commonly used for fabricating such membranes [105–107]. PILs are major ion stimuli-responsive polymers due to their thermal stability, great dissolution, good catalytic activity, non-flammability, high ionic conductivity, and tunable polarity [105,106].

It was shown that the hydrophobicity of ionic liquids (ILs) surface could be adjusted by dropping the different solutions of anions following sequence as follow: bis(trifluoromethane)-sulfonimide ($TFSI^-$) > PF_6^- > $CF_3SO_3^-$ > ClO_4^- > NO_3^- > BF_4^- > Br^-. When the length of cationic alkyl chains increased, the hydrophobicity of ILs monolayers increased. Moreover, the hydrophobicity of ILs surface could also be tuned by adding anion species to the membrane (Fig. 5) [108]. Like stimuli-responsive membranes, the transition of polymer charges and chain structure can influence the formation of a charged barrier that allows water molecules to be trapped and pass through but repels oil droplets on the membrane surface. A PILs-based mesh membrane was fabricated and coated with N-vinylimidazolium ILs and divinylbenzene. The membrane exhibited hydrophilicity as the PIL-based membrane (PILMs) was changed to poly (1-vinyl-3-butylimidazolium acrylate)-based membrane (PILM-1), which allowed water to pass through while repelling oil. Moreover, the membrane exhibited hydrophobic properties by changing PILMs to poly (1-vinyl-3--octylimidazolium hexafluorophosphate)-based membrane (PILM-5), which allowed oil to pass through while retaining water [108]. ZIF-8 nanoparticles and PIL were grafted on cotton cloth fabric to obtain switchable wettability of the membrane. The membrane exhibited superhydrophilicity/underwater superoleophobicity after adding ZIF-PIL(Br) to the membrane and was switched to superhydrophobicity/superoleophilicity after adding ZIF-PIL(PF_6) to the membrane [109].

An ion stimuli-responsive membrane showed superhydrophobicity for oil removal and superhydrophilicity for water removal under oxidation and reduction states, respectively; it achieved an oil/water separation efficiency of over 88% and had anti-fouling properties for viscous oils (kerosene, engine oil, soybean oil, and corn oil), and excellent acid resistance and salt tolerance [110]. Membrane-based PILs exhibited an excellent oil/water separation efficiency of over 99% in batch processes and recyclability of over 99% of continuous separation after multiple oil/water separations [108]. However, PILs lead to slow diffusion through micropores and mesopores of the membrane, which is a significant limitation to its practical application [111].

2.5. Electric field stimuli-responsive membranes

Electric field stimuli-responsive membranes are made of electroactive polymer materials. The switchable properties of the membrane are related to the interaction between the droplets and the conductive polymer substrate (e.g., carboxylate, sulfonate, and the ammonium ionic group). Changing the electric field and adding a conducting droplet and a counter electrode could be used to accurately adjust the CA of the droplet, leading to the control of surface wettability [61,112,113].

Typical electric field stimuli-responsive polymers such as polythiophene, polypyrrole, and polyaniline have been applied to fabricate smart surfaces [59]. For example, a carbon membrane was coated with poly-3-methylthiophene (P(3-MTH)). The wettability of the membrane could be switched by doping and without doping of P(3-MTH) with ClO_4^- under an electric field, corresponding to hydrophilicity (doped state) and hydrophobicity (undoped state), as shown in Fig. 6 [45]. The silver-coated porous copper mesh was infused with oil (e.g., cyclohexane) to obtain superhydrophobicity to allow oil to pass through and repel water. After changing the electrical potential, the as-prepared mesh changed to superhydrophilicity to allow water to pass through and repel oil. Moreover, the as-prepared mesh could be switched from superhydrophilicity to superhydrophobicity by rinsing with organic liquids (e.g., ethanol) and infusing oil again [112].

Electric field stimuli-responsive membranes achieved an oil/water separation efficiency of 99.5% for surfactant-stabled emulsions with various oil concentrations in less than 30 s after being triggered under the electric field and efficiency higher than 97% for both oil-removing and water-removing in oil/water separation process [112–114]. Moreover, after multiple oil/water separation cycles, the membrane still exhibited a separation efficiency higher than 98% [115]. However, the main drawbacks of electric field stimuli-responsive membranes are related to the use of the high-potential electric field, which makes it difficult to alter the surface wettability. Additionally, the membranes have a low reaction rate

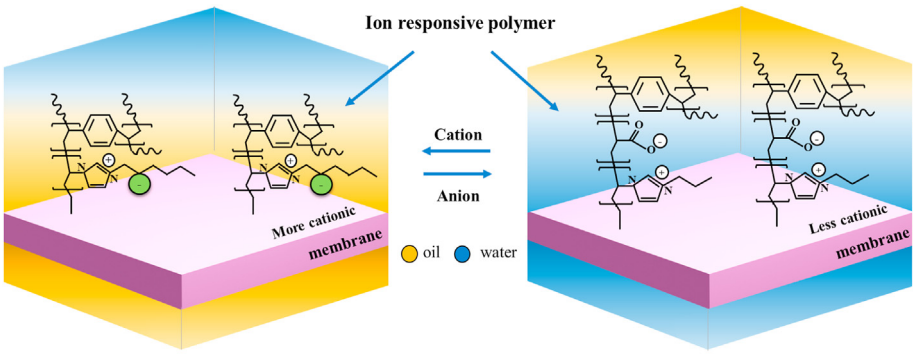

Fig. 5. Schematic diagram of wettability change on membrane surface after being triggered by ion (poly (ionic liquid); PILs).

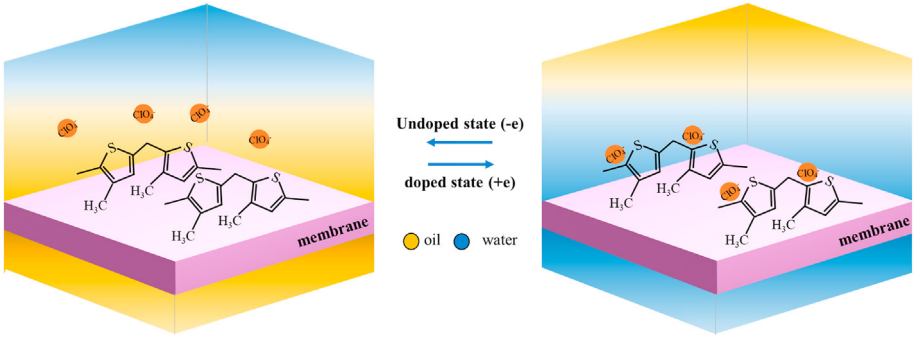

Fig. 6. Schematic diagram of wettability change of membrane surface after being triggered by P(3-MTH) with ClO_4^- under an electric field.

and small deformations, high activation fields requirement, high initial cost, and need specific supporting facilities [90,116].

2.6. Gas stimuli-responsive membranes

Gas stimuli-responsive membranes exhibit switchable wettability after being triggered by gas stimulus (e.g., O_2, N_2, CO_2, and CH_4), as shown in Fig. 7 [117]. The membranes were designed to recover the gas stimulus after separation, which reduces secondary pollution. CO_2 is one of the most desirable triggers due to its non-toxicity, low cost, and renewability [59,81]. Typical gas-responsive polymers include guanidine, amines, and amidines, such as poly (N, N-diethylaminoethyl methacrylate) (PDEAEMA) and PDMAEMA [118]. The gas-responsive membranes have various advantages, i.e., being easily reversible, environment-friendly, cost-effective, non-invasive upon operation, no additional chemicals, and no secondary pollution [43,88]. However, gas stimuli-responsive membranes have low mechanical strength. The gas stimulus can be lost in the water or oil phase leading to the use of a large amount of gas in the system, incurring a high operational cost [118,119].

Gas stimuli-responsive membranes showed good separation performance. For example, Ag/AgCl decorated CO_2-responsive cotton membranes exhibited switchable wettability after being triggered by CO_2. The separation efficiencies of such a membrane were higher than 97.50% for various oil/water mixtures [120]. Similarly, a CO_2-responsive cotton fabric membrane was decorated with DMAEMA. The membrane showed switchable hydrophilicity and hydrophobicity after being triggered by CO_2/N_2. Meanwhile, the membrane achieved an over 90% separation efficiency for various oil/water mixtures, including vegetable oil, *n*-hexane, cyclohexane, and toluene [118]. In another study, after five oil/water separation cycles, gas stimuli-responsive membranes still exhibited high separation efficiencies of more than 98% [121].

2.7. Magnetic stimuli-responsive membranes

Magnetic stimuli-responsive membranes exhibit switchable surface wettability under a magnetic field. Magnetic stimuli-responsive membranes were grafted with superparamagnetic nanoparticles on the membrane surface. It could increase hydrophilicity and remove contaminants on the membrane surface through perturbing magnetic particles in the magnetic field while tailoring the pore characteristics and size [122,123]. However, magnetic stimuli-responsiveness requires intense magnetic fields, which may increase initial cost and reaction time [122]. The membrane surface could be switched between a smooth and rough surface by adjusting the microstructure under the magnetic field. The microstructure changing (e.g., magnetorheological elastomer micropillars and Fe_3O_4 magnetic nanoparticles) could be deformed between lie down and perpendicular, influencing switchable wettability and self-cleaning properties by changing the CA and surface energy of membrane surface [61].

The membrane exhibited anti-fouling properties after 6 h of feeding with organic salt, inorganic salt, and synthetic oily wastewater. Wang et al. fabricated a magnetically responsive anti-fouling nanofiber membrane for separating oil/water emulsions [125] (Fig. 8). It was found that the as-prepared membrane exhibited high performance of oil/water emulsions separation in various emulsions types such as hexadecane, octane, and rapeseed oil (efficiency of 98.04%, 96.59%, and 92.67%, respectively). Moreover, the membrane exhibited a high oil/water emulsions separation efficiency of 95% after eight separation cycles and exhibited anti-fouling and easy-cleaning properties. Elsewhere, novel magnetic stimuli-responsive membranes were fabricated by adding superparamagnetic nanoparticles onto the end of polymer chains to achieve magnetic stimuli-responsiveness [126].

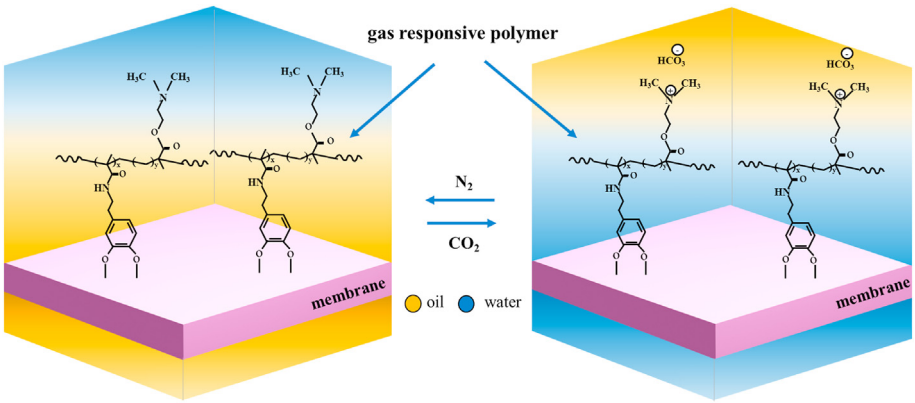

Fig. 7. Schematic diagram of wettability change of membrane surface after being triggered by CO_2/N_2.

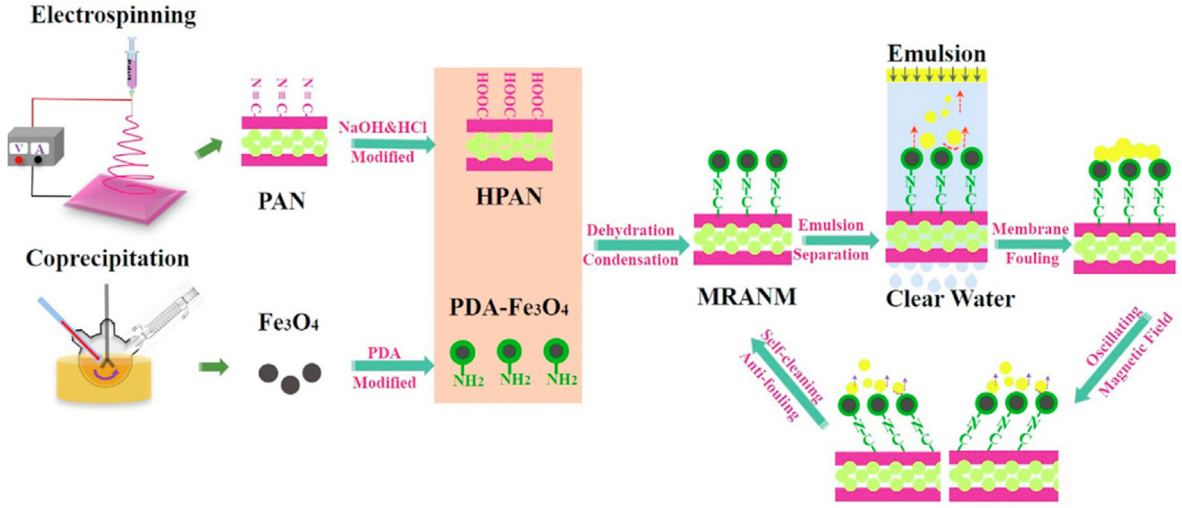

Fig. 8. Schematic diagram of the preparation of magnetic stimuli-responsive membranes [124].

2.8. Solvent stimuli-responsive membranes

Solvent stimuli-responsive membranes, one of the most popular smart membrane types, have various advantages, such as simple fabrication, mild reaction conditions, low energy consumption, short time consumption, and no secondary pollution, leading to high potential for large-scale application [125,126]. Solvent stimuli-responsive membranes can switch surface wettability quickly after prewetting with the solvent solution due to the chemical reaction between the solution and the polar or non-polar functional group of solvent stimuli-responsive polymers on the membrane surface. Upon prewetting the membrane with the polar solvent solution, especially water, the polar solvent solution would form a barrier layer on the membrane surface and repel the permeation of oil droplets. While prewetting the membrane with a non-polar solvent solution, e.g., oil, the non-polar solvent solution will form a barrier layer and repel the permeation of water droplets on the membrane surface (Fig. 9) [127]. This phenomenon leads to the ability to separate oil/water systems and oil/water/solid three-phase systems [128]. The wettability of solvent stimuli-responsive membranes can be varied by prewetting using specific polar or non-polar solutions, including polar or non-polar solvent, oil, or water, also known as solvent-induced, oil-induced, and water-induced membranes, respectively. Before the prewetting process, the membrane exhibits amphiphilic properties (e.g., hydrophobicity and hydrophilicity) [129,130]. After the prewetting process, the membrane could change wettability from hydrophobicity to hydrophilicity and vice versa, depending on the types of solution used in the prewetting process (i.e., solvent, oil, or water) [59].

Solvent stimuli-responsive membranes can achieve outstanding separation performance. It can be applied to separate various types of oil/water mixtures under ambient pressure, including immiscible light oil/water mixtures, heavy oil/water mixtures, miscible oil-in-water, and water-in-oil emulsions stabilized by surfactants, with a separation efficiency of more than 99% [131–133]. Moreover, the membranes can separate heavy oil-water-light oil ternary mixtures [134,135]. After multiple separation cycles, it can still achieve a high oil/water mixture separation efficiency of over 96% [132,133], and it also exhibited excellent chemical resistance to acid, mild alkali, and salty from the environment [136]. However, for the separation of heavy oil/water mixtures, heavy oil droplets are easily accumulated on the porous structures, leading to decreased water permeation [137].

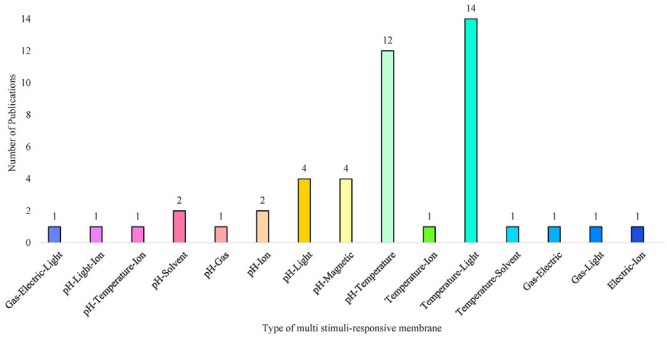

Fig. 10. Publications of multi-stimuli-responsive membranes for oil/water separation between 2018 and 2022.

A typical example of solvent stimuli-responsive membranes is the zein/PAN fiber membrane decorated by zein/SiO_2 spheres on the membrane surface. After prewetting the surface with oil or water, the membrane exhibited on-demand oil or water separation with a high separation efficiency of more than 97% and a high separation flux of more than 400 L m^{-2} h^{-1} for both oil/water emulsions and surfactant-

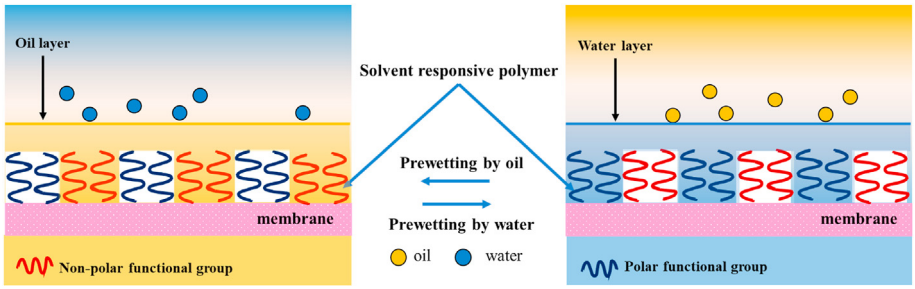

Fig. 9. Schematic diagram of wettability change of membrane surface after prewetting by oil and water.

stabilized oil/water emulsions [129]. Moreover, the solvent stimuli-responsive membranes exhibit superamphiphobic properties by adding zinc oxide particles and organic silane onto the surface of the membrane. After ethanol prewetting, the membrane showed switchable wettability from superamphiphobicity to superhydrophobicity. While upon water prewetting, the membrane changed its wettability from superamphiphobicity to superhydrophilicity. Moreover, the membrane possesses a high oil/water emulsions separation efficiency for both ethanol prewetting (ethanol-induced) and water prewetting (water--induced). It was shown that the separation efficiencies after ten oil/water separation cycles were higher than 98% for various oil/water mixtures (e.g., *n*-hexane, hexadecane, trichloromethane, toluene, methylene dichloride, and tetrachloromethane) [138,139].

2.9. Multi-stimuli-responsive membranes

Multi-responsive membranes undergo surface wettability change with the effect of several external stimuli [59,125]. It generally contains various types of responsive polymeric materials on the membrane surface, and typical examples include gas-electric field-light-responsive membranes,

Table 2
The advantages and disadvantages of smart membranes and their operating conditions and performances for oil/water emulsions separation.

Types	Advantages	Disadvantages	Examples of operating conditions and performances
pH stimuli-	excellent switchable wettability properties, reversibility, rapid response, easy operation, low energy consumption, and high efficiency of oil/water separation [13,63,66–68,78,148]	secondary pollution, high operation costs, needs for pre-wetting before the separation process, and accumulation of salts in the system from adding acid or base [13,45,81,82]	PDHH/SiO_2, conditions: pH > 7; hydrophobicity, flux: > 10,000 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, ≥ 98% [70] Fe_3O_4/DDA/TiO_2, conditions: pH = 2; superoleophobicity, flux: 9400–13000 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, >99% [74]
Light stimuli-	physical, chemical, and thermal stability, good controllability regarding wavelength, time, and intensity, remote operation, inexpensive, and no secondary pollution [36,40,59,68,83–85,89]	long time for the response and recovery process, need a complicated design, and massive energy consumption [40,45,88–90]	ZIF-8/GO, conditions: visible light; superoleophobicity, flux: 110 ± 6 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, 100% [86] $CoMoO_4$@Bi_2MoO_6, conditions: visible light; superhydrophilicity, flux: 1449.12 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, 99.67% [89]
Temperature stimuli-	no secondary pollution, inexpensive, easily applied without direct addition of chemicals, remote-control mechanism, and stability of wettability after continuous separation [59,96,99–101,149]	slow response, huge energy consumption [45,96,103,104]	PNIPAM/PCL, conditions: <20 °C; hydrophilicity, >50 °C; hydrophobicity, flux: > 60 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, 92% [41] PNIPAM/TiO_2@PAN, conditions: <25 °C; hydrophilicity, >50 °C; hydrophobicity, flux: > 7700 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, 98% [92]
Ion stimuli-	thermal stability, remarkable dissolution, non-flammability, high ionic conductivity, wide electrochemical window, tunable polarity, basicity/acidity, and electrochemical stability [108]	slow diffusion during adsorption and desorption is a significant limitation that severely restricts its practical application [111]	ZIF-8@PIL, conditions: dropped PF_6^-; superhydrophobicity, dropped Br^-; superhydrophilicity, flux: > 68 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, 99% [109] PPy-AOT, conditions: oxidation states; superhydrophobicity, reduction states; superhydrophilicity, flux: 40 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, >80% [110]
Electric field stimuli-	fast response, no need for chemical adding, easily mass-produced, and energy-efficient [45,116]	hazardous and difficult to alter the wettability of membrane, requirements on high activation fields, increased initial cost, and need for specific supporting facilities [45,90,116]	P(3-MTH), conditions under an electric field: dropped ClO_4^-; hydrophilicity, undoped; hydrophobicity, flux: 3000 $Lm^{-2}h^{-1}$ 0.4 bar, efficiency: oil/water mixture, 99.5% [45] Electrodeposited copper mesh, conditions: voltage application; superhydrophilicity, efficiency: oil/water mixture, 98% [115]
Types	Advantages	Disadvantages	Examples of operating conditions and performances
Gas stimuli-	inexpensive, can be added and removed easily in a large volume over one or multiple cycles without accumulating, environmentally friendly, remote operation, and no secondary pollution [43,81,88,118]	low mechanical strength, loss of gas stimulus in the water and oil phase, and high synthesis costs [88,118,119]	p(DMA-DMAEMA)@DMA/cotton, conditions: CO_2; superhydrophilicity, N_2; superhydrophobicity, efficiency: oil/water mixture, >90% [118] Ag/AgCl@PCM, conditions: CO_2; superhydrophilicity, flux: 9918.60 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, 98.50% [120]
Magnetic stimuli-	easily controlled by an external magnetic field, and anti-fouling under a magnetic field [122]	strong magnetic field and high temperature needed, and increased magnetic viscosity can decrease exposure time between the membrane and magnetic field [122]	MRANM, conditions: magnetic field; hydrophilicity, flux: 2764 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, > 92.67% [124] Superparamagnetic NPs, conditions: magnetic field; hydrophilicity, flux: >2000 $Lm^{-2}h^{-1}$, efficiency: synthetic oily wastewater [150]
Solvent stimuli-	simple fabrication, mild reaction conditions, low energy consumption, short time consumption, no secondary pollution, and fast recovery [125,126,128]	need pre-wetting before use for separation, separation of heavy oil/water mixtures can accumulate, block, and prevents water permeation [137]	Sodium-bentonite-fabricated graphene (SBG), conditions: water prewetted; hydrophilicity, oil prewetted; hydrophobicity, flux: 625 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, >98% [131] Chitosan@PCL, conditions: water prewetted; superhydrophilicity, oil prewetted; superhydrophobicity, flux: 2609 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, >94.6% [135]
Multi stimuli-	simultaneous control of different characteristics, reversible multi-stimuli-responsive wettability triggered by various stimuli [59,125,142,143]	complicated to fabricate, needs for design and preparation of advanced systems, long-time fabrication, and increased initial cost [143,151]	NPs-SSM/PFDTMS, conditions: 200 °C; superhydrophobicity, UV irradiation; superhydrophilicity, flux: > 42.7 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, 99% [145] P(AN-co-AM)/P4VP/PVDF, conditions: pH 7 and 25 °C; hydrophobicity, pH 3 and 55 °C; hydrophilicity, flux: > 60,528 $Lm^{-2}h^{-1}$, efficiency: oil/water mixture, 99.5% [152]

pH-light-responsive membranes, and temperature-light-responsive membranes [140–142]. However, the fabrication process of multi-responsive membranes is complicated and requires sophisticated design and preparation, which is time-consuming and costly [143,144].

It has been reported that multi-stimuli-responsive membranes can achieve an oil/water emulsions separation efficiency of >92% for different types of oil and have good chemical stability and self-healing or self-recovery ability [101]. It was shown that the membrane triggered by UV irradiation and temperature exhibited an oil/water separation efficiency of >99% [145]. pH-UV stimuli-responsive membranes exhibited reversible wettability after being triggered by changing pH values and UV irradiation [142]. A membrane triggered by light and temperature achieved a high oil/water emulsions separation efficiency in both surfactant-free and surfactant-stabilized emulsions (e.g., *n*-hexane-in-H_2O, toluene-in-H_2O, petroleum ether-in-H_2O (petroleum/H_2O), Sodium Dodecyl Sulfate (SDS)-stabilized *n*-hexane-in-H_2O, SDS-stabilized toluene-in-H_2O, and SDS-stabilized petroleum ether--in-H_2O (petroleum/SDS/H_2O) with an oil/water volume ratio of 1:99) [146]. Light-temperature responsive membranes exhibited a high separation efficiency of 99% for oil-in-water nano-emulsions with excellent anti-oil/ionic-fouling properties, good recyclability, and desirable stability [147].

Different multi-stimuli-responsive membrane designs for oil/water separation have been reported. Temperature-light-responsive and pH-temperature-responsive are among the most reported in the past five years, as shown in Fig. 10. These responsive membranes achieve good complementarity in properties towards more efficient oil/water separation in complex environments. The advantages and disadvantages of smart membranes and their operating conditions and performances for oil/water emulsions separation are summarized in Table 2.

3. Fabrication strategies of smart membranes

The main strengths of stimuli-responsive membranes are their controllable properties, high permeability, and anti-fouling properties [153,154]. Stimuli-responsive membranes can be fabricated using various methods, such as copolymer blending, surface grafting, surface coating, and plasma treatment [155,156]. The fabrication strategies of smart membranes are based on different mechanisms which affect the applications of the obtained membrane. Various factors need to be considered when choosing appropriate fabrication strategies, including types of polymers, membrane surface properties, and membrane chemical properties [157]. Surface coating is found to be the most used fabrication method, followed by surface grafting and copolymer blending. The brief definitions of these fabrication strategies are shown in Table 3.

Table 3
The descriptions and key features of the major fabrication strategies for smart membranes.

Fabrication strategies	Descriptions	Features
Surface coating	surface coating is a simple method in which the polymers with opposite characteristics (e.g., ionic charges or charge transfers) are coated on the membrane surface.	formation of polymers with changed surface properties while keeping the bulk properties of the primary polymers unchanged.
Surface grafting	surface grafting employs the main polymers as the backbone, to which branches of second polymers are grafted, to modify the primary polymer's surface characteristics.	formation of polymers with changed surface properties while keeping the bulk properties of the primary polymers unchanged.
Copolymer blending	copolymer blending is relatively simple, as it merely involves the mechanical mixing of two or more polymers to achieve a combination of specific properties of a single polymer.	formation of a polymer mixture free of permanent bonds while possessing properties different from original polymers.

3.1. Single stimuli-responsive membranes

The main fabrication strategies for single stimuli-responsive membranes are surface coating, surface grafting, and copolymer blending. The schematic diagram of the main fabrication strategies for a single stimuli-responsive membrane is shown in Fig. 11.

3.1.1. Surface coating

Surface coating is a method to change the surface of membranes by simply coating stimuli-responsive polymers on their membranes (Fig. 11) [158]. A coated membrane can achieve high permeability due to improved porosity and pore size [155]. Surface coating can be done by various methods such as spray coating [159,160], dip coating [161,162], layer-by-layer coating [163,164], and electrodeposition [165,166]. Surface coating is relatively low-cost, simple, and suitable for wide applications [167].

The surface coating method has been used to fabricate pH stimuli-responsive membranes, light stimuli-responsive membranes, and solvent stimuli-responsive membranes. For example, Cai et al. fabricated pH stimuli-responsive membranes from a porous anodic aluminum oxide membrane with the dip-coating method. The membrane was coated with poly (4-vinyl pyridine) (which acted as pH-responsive component) and polystyrene block copolymers [67]. Li et al. fabricated solvent stimuli-responsive membranes by a feasible mussel-inspired dip-coating method. The as-prepared membrane was coated with PDA and an amphiphilic synthetic copolymer. After that, the PDA-coated membrane was immersed in a mixture of dopamine, tris(hydroxymethyl) aminomethane, and polymers solution to obtain the polymer/PDA-coated membrane [90]. Yue et al. fabricated light stimuli-responsive membranes using the LBL method for which zeolitic imidazolate frameworks/Graphene and trimesoyl chloride (TMC) was grafted on a PDA-support substrate [86].

3.1.2. Surface grafting

Surface grafting can be done in two ways: grafting to a membrane and grafting from a membrane (Fig. 10). A covalent bonding is generally needed during the grafting process. Surface grafting has been widely used for smart membrane fabrication. It can integrate many stimuli-responsive polymers on the membrane surface to achieve a high permeability [168].

The grafting to membrane is the easiest method for grafting stimuli-responsive polymers through chemical bonding or physical attaching onto the membrane surface. This process is initialized by attaching the polymer, which consists of an active functional group polymer, onto stimuli-responsive polymers to make brush-like polymer chains. The brush-like shape of the attached stimuli-responsive polymer chains can increase the permeability of the membrane because the gap between polymer chains significantly increases the hydrodynamic force of water droplets on the membrane surface [169]. With the grafting to the membrane way, the membrane will have a low density of stimuli-responsive polymer chains on the membrane surface because of the difficulty and limitation of attaching the active functional group of polymers on the membrane surface, indicating a low number of polymer chains that are attached to the membrane per unit of the surface area [198,199]. Moreover, it will have low stability when the polymer chains are cross-linked with other chains.

The grafting from the membrane is started by attaching initiator molecules onto the membrane surface. Then, stimuli-responsive polymers are grown on the initiator molecules by adding the controlled monomer and stimuli-responsive polymers through various methods (e.g., chemical initiator, UV/gamma irradiation, or plasma) [170]. The grafting from the membrane way can potentially increase the grafting

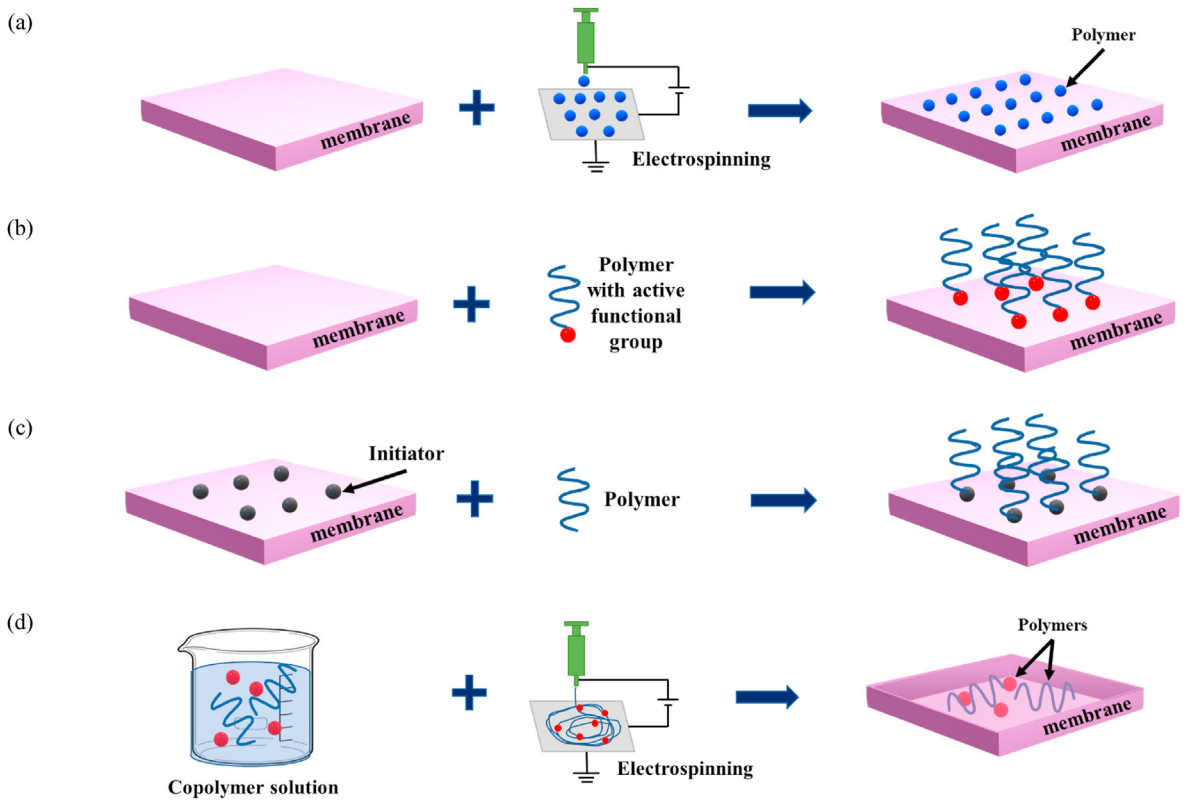

Fig. 11. Schematic diagram of the main fabrication strategies for a single stimuli-responsive membrane: (a) surface coating, (b) surface grafting; grafting to membrane, (c) surface grafting; grafting from membrane, (d) copolymer blending.

density of the membrane surface. Moreover, it is cheaper than the grafting to membrane because the polymer purification process is not required before the grafting process. However, it is less accurate and controllable in determining the shape of polymer chains and the position of stimuli-responsive polymer chains on the membrane surface [155, 171].

The surface grafting method has been mainly used to fabricate pH stimuli-responsive and temperature stimuli-responsive membranes. For example, Wang et al. grafted copolymers of DMAEA and TFOA on beadlike porous PS fibrous membranes by using a UV photografting method to obtain pH-responsive membranes [71]. Liu et al. grafted PNIPAM brush on PCL porous membrane by surface grafting with an SI-ATRP method. Afterward, the membrane was grafted using PNIPAM brush chains to obtain temperature stimuli-responsive membranes [41].

3.1.3. Copolymer blending

The copolymer blending method is defined as blending of polymers containing special functional groups with a bulk polymer. The main advantages of copolymer blending are simplicity and repeatability. However, the main disadvantages of copolymer blending are low permeability, limited stimuli-responsive properties, and unstable surface properties of bulk polymers because the polymers may lose their structural and functional properties after the blending process [155].

The copolymer blending method has been applied to fabricate pH and temperature stimuli-responsive membranes. For example, Wu et al. fabricated a momordica-charantia-like nanofibrous membrane. The membrane was modified by copolymer blending with a mixture solution of zeolitic imidazolate framework-8 and polyacrylonitrile particles to obtain a pH stimuli-responsive membrane. After the modification, the membrane achieved hydrophilicity [30]. Sun et al. fabricated a nanofibrous membrane with a mixture solution of PNIPAM, PAN, and TiO_2 to obtain temperature stimuli-responsive membranes [92].

3.2. Multi-stimuli-responsive membranes

For multi-stimuli-responsive membranes, the fabrication method often combines multiple methods to obtain unique characteristics for each responsive polymer. For example, Gao et al. prepared biomimetic TiO_2-titanium meshes by copolymer blending with a mixture solution of TiO_2 particles, PVDF, and perfluorooctanoic acid. Then, the meshes were modified by the one-step dip-coating method using poly (vinylidene difluoride) to obtain biomimetic TiO_2−titanium meshes with light-temperature-responsive properties [101]. Qiu et al. fabricated a superhydrophobic membrane by blending a mixture solution of SiO_2, 1H,1H, 2H,2H-perfluorodecyltrimethoxysilane (PFDTMS) with ethanol, and TiO_2 to achieve PFDTMS/nanoparticles (NPs)/Stainless steel mesh (SSM) membrane. Then, the membrane was coated by spraying particles of TiO_2 and SiO_2 modified by PFDTMS on the membrane surface to obtain light-temperature-responsive membranes [145].

4. Conclusions and future perspective

During the oil/water emulsions separation process, smart membranes are designed and developed to reduce the accumulation of oil droplets and oil fouling on the membranes and increase the efficiency of oil/water emulsions separation, with improved anti-fouling properties and switchable surface wettability. This review presents a brief overview of smart membranes for oil/water emulsions separation reported in the past five years, covering pH, light, temperature, solvent, and multi-stimuli-responsive membranes. It is found that solvent stimuli-responsive membranes are the most popular type of smart membranes due to such advantages as simple fabrication, mild reaction conditions, low energy consumption, short time consumption, no secondary pollution, and quick recovery. Moreover, it can separate both oil/water systems and oil/water/solid three-phase systems. Followed by pH stimuli-responsive

membranes, which possess the strength of excellent switchable wettability, excellent reversibility, rapid response, and simple operation. The pH stimuli-responsive membranes have uncomplicated equipment, low energy consumption, good reusability, and high oil/water separation efficiency. Similarly, the multi-stimuli-responsive membranes are one of the most reported due to their capability to respond to more than one stimulus. The membranes can simultaneously control different characteristics of each responsive polymer contained on the membrane and reversible multi-stimuli responsive wettability.

Various methods have been proposed to fabricate smart membranes, including surface coating, surface grafting, copolymer blending, and plasma treatment. However, the former three are among the most popular types of smart membrane fabrication methods due to their advantages for fabricating single stimuli-responsive and multi-stimuli-responsive membranes. Designing smart membranes with lower costs, more rapid responsiveness, greater anti-fouling ability, and ease of use for a wide range of applications would be more interesting to develop for future work. Artificial intelligence and machine learning techniques are expected to play a great role in smart membranes design, optimization, and properties prediction to reduce and save initial costs and the time consumed.

Declaration of competing interest

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