Membranes for Oil/Water Separation: A Review

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Recent advancements in separation and membrane technologies have shown a great potential in removing oil from wastewaters effectively. In addition, the capabilities have improved to fabricate membranes with tunable properties in terms of their wettability, permeability, antifouling, and mechanical properties that govern the treatment of oily wastewaters. Herein, authors have critically reviewed the literature on membrane technology for oil/water separation with a specific focus on: 1) membrane properties and characterization, 2) development of various materials (e.g., organic, inorganic, and hybrid membranes, and innovative materials), 3) membranes design (e.g., mixed matrix nanocomposite and multilayers), and 4) membrane fabrication techniques and surface modification techniques. The current challenges and future research directions in materials and fabrication techniques for membrane technology applications in oil/water separation are also highlighted. Thus, this review provides helpful guidance toward finding more effective, practical, and scalable solutions to tackle environmental pollution by oils.

1. Introduction

Water pollution is a significant challenge for the 21st century. The contamination levels increase due to the inputs from different pollution sources, such as domestic, industrial wastewater, and oil leakage in petroleum sites.[1] Among various pollutants, massive daily amounts of oily wastewater are produced due to oil spill accidents during industrial petroleum operations and

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processing.[1,2] This oily wastewater has severe adverse effects on the environment, as previously witnessed during several oil spill accidents in the marine and terrestrial environments due to oil leakage from petroleum transport pipelines, storage tanks or transport facilities, and blowouts in oil wells.[3] The treatment of oily wastewater can be performed via numerous technologies such as adsorption,[4] dissolved air flotation,[5] coagulation,[6] gravity separation,[7] and biological treatment.[8] However, these methods have several limitations, such as low removal efficacy, high operational cost, high energy consumption, corrosion, and production of contaminated solid waste from spent adsorbents.[9] The rapid growth in separation technologies has resulted in various membranes that could overcome the shortcomings of other treatment methods.[10] Thus, membranes have become more desirable for oil/water separation for industrial applications because of their small footprint, environmental friendliness, cost-effectiveness, high productivity, low chemical consumption, automation capability, steady operation, and excellent separation efficiency.[11] Indeed, pore blockage or fouling remains among the major obstacles facing the membrane separation process since it decreases the flux, restricts the permeation rate, increases the energy consumption, and decreases the membrane lifetime. Therefore, substantial research efforts have been focused on enhancing the properties of membranes either via developing novel materials or introducing surface modifications for conventional membranes.[12,13] Although few reviews were published on oil/water separation membranes, they are either outdated or focused on a specific membrane type. This review features the most recent progress in membrane separation and its practical application in oil/water separation. It includes four sections that cover essential factors related to the advancement in membrane technologies are highlighted, including: 1) membrane properties and characterization for oily waste water separation; 2) the employed materials to fabricate membranes (i.e., metallic, organic, inorganic, and hybrid materials), 3) primary methods for designing membranes (i.e., mixed matrix nanocomposites and multilayers nanocomposites), and 4) membrane fabrication techniques and surface modification methods.

2. Membrane Properties and Characterization

The main objective of the oil/water separation membrane is to achieve high separation efficiency and excellent performance.

These desired objectives are related to membrane surface properties such as hydrophobicity/hydrophilicity, charge, permeate flux, and roughness, affecting membrane separation performance.[3] The critical factor is membrane wettability, which will be covered in the following section that is essential for water flux and decreasing membrane fouling.

2.1. Membrane Wettability

Surface wettability is an essential property that distinguishes membranes, especially oil/water separation. Membrane materials have four types of wetting properties: hydrophobicity, hydrophilicity, oleophobicity, and oleophilicity. Each membrane material has opposite properties: hydrophobicity/oleophilicity and hydrophilicity/oleophobicity.[3,14] In applying oil/water separation, membranes materials are described either as oil-removing with hydrophilicity (water-loving)/or oleophobicity (oil-hating), in which oil passes through the material. In contrast, the water is repelled or water-removing by hydrophobicity (water-hating) /oleophilicity (oil-loving). Thus, water is allowed to cross and prevent oil on the surface of membranes.[15] Wei et al.[16] reviewed the recent advances of oil/water separation membranes special wettability. Further review by Gu et al.[17] focused on superwetting separation membranes based on carbon and 2D laminar membranes for the application of selective ion and water transport.[18] The surface properties such as wetting are measured with contact angle, which influences hierarchical membrane structure.[19] Contact angle (Θ) is described by a three-phase contact line of a deposited drop, as shown in **Figure 1**; membrane wettability detailed was reviewed by Wei et al.[16] Interfacial tension among the solid–vapor (γ_{SV}), solid–liquid (γ_{SL}), and liquid–vapor (γ_{LV}) surface tensions on the surface of the membrane can be calculated using the formula of Young–Dupre equation:[14,20]

$$\cos\theta = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} \tag{1}$$

The relation between surface roughness and wettability shows that increasing of surface roughness improve surface wettability which is caused by surface chemistry as described in Figure 1b, which is determined by the Wenzel contact angle (CA) (θ_W) as following:

$$\cos\theta_W = r\cos\theta \tag{2}$$

where θ_W is the apparent contact angle, and r is the roughness factor which is defined as the ratio of the actual area of the solid surface to the obvious surface area, and θ is the Young's CA.

To describe a heterogeneous rough surface CA is described by Cassie–Baxter (Figure 1c)

And the following formula:

$$\cos\theta_{CB} = r_f f_{SL}\cos\theta + f_{SL-1} \tag{3}$$

where, f_{SL} refer to the solid–liquid fraction under the contact area, rf is refer to roughness ratio of the wet part of the as defined solid surface.

Surface roughness has a great role in the membrane's surface wettability. Surface wettability increases with increasing surface roughness, leading to the membrane surface's hydrophilic or hydrophobic property.[21] As a result, membrane surface features based on the contact angle can be divided into two approaches for water and oil; when the contact angle is below 90°, the surface is hydrophilic. The appearance of deposited water drops on a membrane with a high hydrophilic surface is rapidly spread, but oil drops are spherical and can roll. Conversely, if the contact angle is above 90°, the surface is hydrophobic. The appearance of deposited water drop on the hydrophobic surface of membranes forms a spherical shape, but oil drop can be spread on the surface and pass through the membrane.[22] Furthermore, wettability development leads to enhanced efficiency for oil/water separation. For example, the hydrophilic property of surface-enhanced superhydrophilic property is by enhancing the surface roughness via different modification methods, either blending membrane polymers with hydrophilic additives (hydrophilic polymers, inorganic nanoparticles, and amphiphilic copolymers) or membrane surface modification (surface coating and surface graft polymerization).[23,24] Furthermore, the superhydrophilic/underwater superoleophobic contact angle is (zero degrees). In contrast, superhydrophobic/superoleophilic contact angle up to 150°.[25] Polyvinylidene fluoride (PVDF) polymeric membrane synthesized via electrospinning exhibited a contact angle for water and oil separation of 153° and nearly zero. This observation means that the membrane is superhydrophobic/superoleophilic surface feathers.[26] The advanced oxidation process is an effective emerging technology that can be integrated with membrane separation and deal with water-soluble organic pollutants.[27] Photocatalytic offers several advantages, such as sustainability, low energy consumption, and green technology that degrade organic pollutants to CO_2 and H_2O as a harmless product.[28] Several photocatalytic materials were used to prepare advanced photocatalytic membranes, such as TiO_2,[29,30] α-Fe_2O_3, ZnO, CeO_2, and $Ti_3C_2T_x$ (MXene) based heterogeneous based membrane,[31] and inspired beam structure house.[32] Several techniques were implemented for water purification, including oxidized carbon spheres-based heterogeneous membrane.[33] For instant, heterojunction membranes from g-C_3N_4
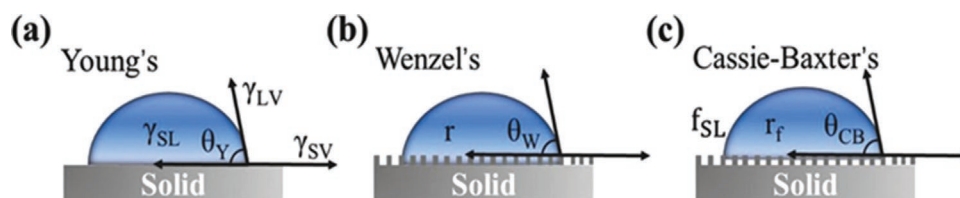

Figure 1. Schematic representation of membrane exposed to a liquid droplet. a) Young's, b) Wenzel's, and c) Cassie–Baxter's. Adapted with permission.[16] Copyright 2018, Wiley-VCH.
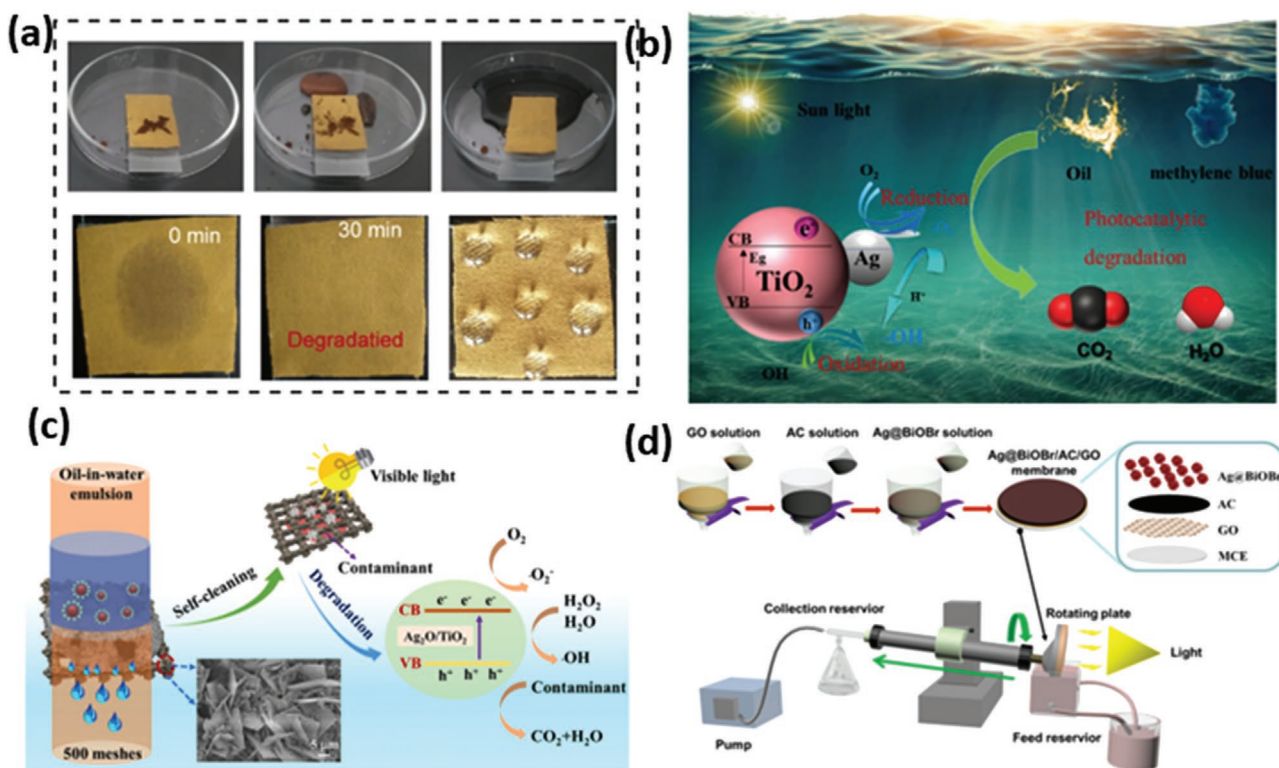
Figure 2. a) The upper images show coated cotton fabric self-cleaning performance and Ag-TiO$_2$@PDMS photocatalytic coated cotton fabric efficiency in the bottom. b) Photocatalysis mechanism of the same materials in (a) for methylene blue and oil. Reproduced with permission.[39] Copyright 2022, Springer Nature B.V. c) photocatalytic and superwetting of Ag$_2$O/TiO$_2$@CuC$_2$O$_4$ membranes for the application of dye removal and oil/water separation, Reproduced with permission.[40] Copyright 2021, Elsevier Ltd. d) Figure illustrates rotating reactor and preparation of integrated photocatalysis-adsorption-membrane separation from Ag@BiOBr/AC/GO in rotating reactor for RhB removal. Reproduced with permission.[27] Copyright 2020, Elsevier Ltd.

nano/microspheres are used for water purification under direct sunlight.[34] Santos et al. reviewed photocatalytic membrane for oily water separation fabricated from nanoparticles and nanocomposites.[35] CNTs-based membrane catalysts were developed for purifying oily wastewater,[36] for example, GR/GO ultrathin membranes,[37] and CNTs/nanoparticles multifunctional hybrid membranes.[38] **Figure 2**a showed cotton fabric coated with synthesis of Ag/TiO$_2$@PDMS with self-cleaning property, high flux, photocatalytic ability under visible light, and recyclability and the photodegradation mechanism is discussed in Figure 2b.[39] He et al. developed superwetting and photocatalytic membranes using Ag$_2$O/TiO$_2$@CuC$_2$O$_4$ nanocomposite.[40] The membrane exhibited a superwetting and photocatalytic behavior due to hydrophilic CuC$_2$O$_4$ nanosheets and belt-like Ag$_2$O/TiO$_2$, the mechanism is illustrated in Figure 2c. Additionally, Zhang et al. integrated a photocatalysis-adsorption-membrane separation system in the rotating reactor for the removal of organic pollutants as shown in Figure 2d.[27] This system is the efficient removal of rhodamine B (RhB) and realizes long-term utilization.

2.2. Membrane Permeability

Membrane permeability is essential in evaluating membrane performance. There are two kinds of membrane surface design, either superhydrophilic or superhydrophobic. In the case of superhydrophobic membranes, the surface allows only the oil to pass through the membrane. However, such membranes are easy to foul, resulting in flux decline, decreased efficiency, and limited practical applications. On the contrary, when a membrane's surface is superhydrophilic, it removes oil while penetrating water.[41] The treated medium was strongly retained when membrane surfaces were hydrophobic and improved membrane permeability. The percentage of flux improvement of the membrane during gravity-driven separation processes is calculated using the following equation (Equation 4).[42]

$$J = \frac{V}{A\Delta t} \tag{4}$$

where: J = permeation flux (L m^{-2} h^{-1}), V = permeate volume of water (L), A = the effective membrane area (m^2), Δt = the permeation time (h).

In the case of the permeability at pressure-driven membranes processes, the permeation flux (L m^{-2} h^{-1} bar^{-1}) was measured at the applied pressures via equation.[43]

$$J = \frac{V}{A\Delta t\Delta P} \tag{5}$$

where: J = permeation flux (L m^{-2} h^{-1}), V = permeate volume of water (L), A = the effective membrane area (m^2), Δt = the permeation time (h), and Δp = pressure.

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Flux decline is a serious problem usually caused by membrane surface fouling, related to traditional hydrophobic-oleophilic membranes through oil/water separation. Hence, there are significant efforts in research to address this issue.[43–46] For instance, high permeation oil/water separation was achieved on hybrid membranes based on carbon nanotube (CNT) addition to polymers. The permeation fluxes of the hybrid membrane reached 4592 L m^{-2} h^{-1} bar^{-1} and remained linear with transmembrane pressure in the range of 0.1–0.7 bar.[47]

2.3. Membrane Fouling Resistance and Mechanical Property

Membrane fouling is a critical factor that being considered when developing oil-water separation membranes. Membrane fouling results from the aggregation of solid particles or oil droplets during separation processes leading to clogged membrane pore on the surface. This phenomenon reduces the performance of membrane separation and decreases membrane lifetime. Furthermore, damage caused by membrane fouling hampered productivity and decreased membrane filtration efficiency. Most pressure-driven membrane (MF, UF, and NF system) for oil/water separation are affected by fouling which contains heterogeneous particles deposited on the membrane surface. Different kinds of materials cause various foulant particles, which may be organic fouling. Causes of organic fouling in natural water include amino sugars, polysaccharides, proteins, and polyhydroxy-aromatics. Additionally, inorganic fouling results from the rejection of inorganic ions or salts on the separation membrane surface. Fouling sources can also multifouling from organic, inorganic, and microbial contamination.[44,48,49]

As Darcy's law in Equation (6) defines the fouling mechanism as resistivity and block flow.

$$R_{\mathrm{t}} = \frac{\Delta P}{j\mu_0} \tag{6}$$

where R_{t} total resistance to flow, J is the permeate flux, μ_0 the solution viscosity, ΔP is transmembrane pressure, the resistance model is expressed according to previously reported work.[50]

Membrane fouling is a point of interest due to its adverse effects on filtration processes. Therefore, membrane fouling is being controlled through three methods. The first method is reducing membrane fouling in the filtration system via controlling the mass transfer of feed solution into the membrane modules. The second method is improving the cleaning procedures of membranes. The third method is fouling resistance through membranes based on oil/water separation.[51,52] Overall, membrane fouling resistance can be improved by improving the membrane surface hydrophilicity.[53,54] For example, carboxylated multiwalled MWCNTs (cMCNTs)/GO nanohybrid was synthesized and then embedded in polysulfone hollow fiber membranes (PHFMs) for oil/water separation application. This is resulted in water flux of (487.9 ± 25.4 mL m^{-2} h^{-1} mmHg^{-1}) with high purity, high antifouling property with 90.5% flux recovery, and oil rejection of (98.7 ± 1.2%).[55]

Membranes' mechanical properties are essential in applying oil/water separation. Membranes separation should withstand temperatures and high operational pressures during separation processes. The membrane should be flexible rather than brittleness to achieve a membrane with suitable properties toward temperature and pressure.[56] For example, a mechanically robust core/shell structured PI/CA nanofibers were developed with flexible PAA and improved the surface roughness of a CA shell.[57] Furthermore, the chitosan-modified PP (CS-PP) membrane exhibited high superhydrophilicity and superoleophobicity via dip-coating and hot pressing method and achieved a tensile strength of 6.78 MPa.[58]

2.4. Membrane Technology Processes

In different wastewater treatment sites such as, desalination, chemical, food processing, and pharmaceutical industries, membrane separation technology are present. In oily wastewater treatment, membrane separation technology is efficient through both gravity and pressure-driven membrane processes. Different types and sizes of rejection particles can be removed through pressure-driven membrane filtration. The filtration system under pressure involves MF, UF, NF, and reverse osmosis (RO) in oil/water separation.[54,59] Membrane processes have a similar concept. However, every membrane process has a different surface pore size. Therefore, the membranes can remove unevenly sized particles, as illustrated in **Figure 3**. The other filtration systems, MF, UF, RO, and NF membranes, can be fabricated from polymeric and inorganic materials.[56,60] The performance of the membrane depends on different features, including; membrane thickness, permeability, filtration time, feed concentration, and transmembrane pressure.[56] MF is used to remove suspended micro and nanosized solids particulates with pore sizes ranging from 0.05 to 10 μm. In MF membranes, proteins penetrate the membrane freely and apply low pressure on the membrane process for separation with low energy cost and degradation resistance under heating.

UF membrane is the most suitable method for oily wastewater treatment due to its appropriate pore size being between 0.01 to 0.2 μm which is close to oil drop size in the emulsion, which ranges between 0.1 to10 μm. As a result, UF membrane can efficiently reject most oil droplets and the permeate water free from oil and can remove oily microemulsions with excellent efficiency and low required energy.[61,62] On the other hand, NF membranes have high properties such as hardness removal, lower power, reject organic molecules on the surface, excellent oil removal, and salts removal. Additionally, the RO membrane is the most applicable membrane process for seawater desalination and water treatment, which can remove monovalent ions and viruses. Both NF and RO have required a high applied pressure.[59]

Moreover, many vital parameters affect the separation permeability and efficiency of the oil/water separation membrane. The most critical factors include membrane porosity, pore size, and rejection coefficient from a practical viewpoint. To calculate membrane porosity ε (%) according to Equation (7); it should be measured as volume of the pores divided by the total size of the porous membrane.[63]

$$\varepsilon = \frac{(W_{\mathrm{w}} - W_{\mathrm{d}})}{\rho_{\mathrm{w}} A L} \times 100 \tag{7}$$

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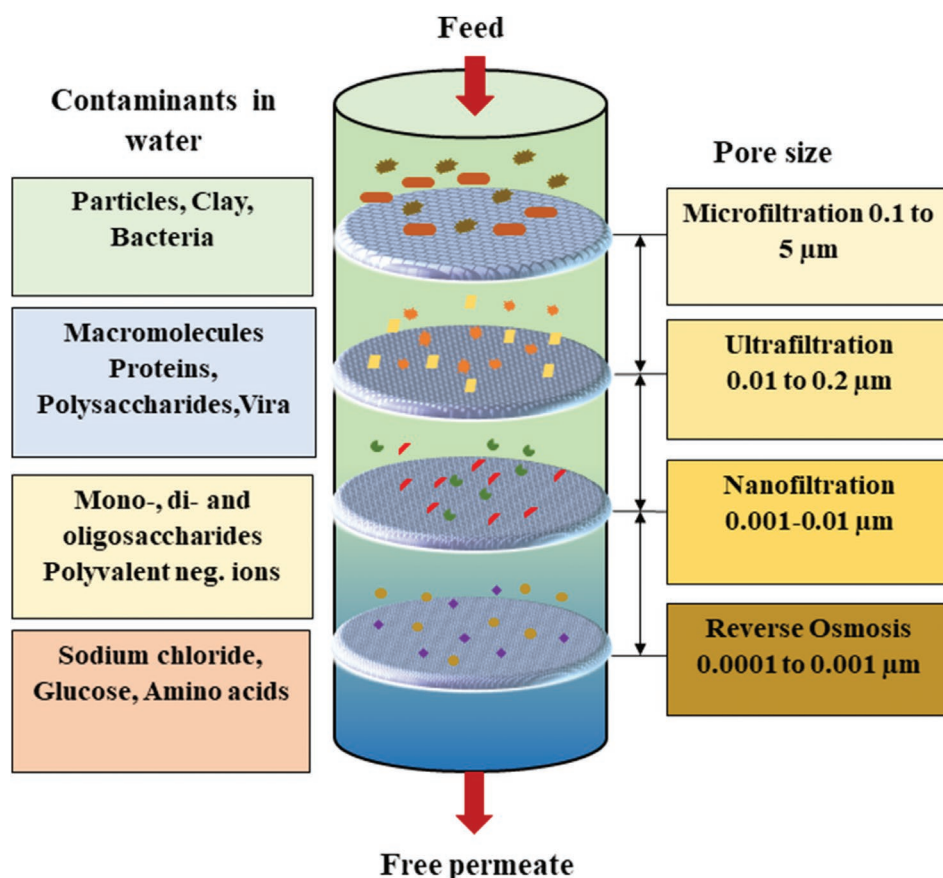INTERFACES**
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Figure 3. Schematic illustration of pressure-driven membrane filtration processes. Reproduced with permission.[65] Copyright 2017, Elsevier B.V.

where W_w is sample weight before drying, W_d is the weight of the sample after drying (g), ρ_w is the density of water.

The filtration velocity method calculated the mean pore size (rm) and can also be calculated through the GueroutElford-Ferry equation.[63]

$$r_m = \sqrt{\left[\frac{(2.9 - 1.75\varepsilon)\,x8\eta l Q_T}{\varepsilon A \Delta P}\right]} \qquad (8)$$

where (ε) membrane porosity (%); (η) the water viscosity (8.9×10^{-4} Pa s); (l) the membrane thickness; (Q_T) the permeate volume per unit time; (ΔP) the applied pressure (1 bar) and (A) is the effective membrane area. In addition, the rejection coefficient is a measure of the ability of the membrane to separate the liquid from the feed liquid solution that can be calculated via Equation (9):[64]

$$R = \left(1 - \frac{C_{permeate}}{C_{feed}}\right) \times 100\% \qquad (9)$$

where R (%) is rejection percentage, $C_{permeate}$ is permeate concentration, and C_{feed} is feed concentration. Furthermore, molecular weight cut-off (MWCO) membranes are the lowest molecular weight of a solute, and the membrane rejects 90% of the solute. MWCO membrane is considered an essential parameter for membrane efficiency and permeation

mechanism. The pore size can predict MWCO of porous membranes and different solute molecules rejection.[62]

3. Membrane Materials

Membrane material with appropriate properties is a key to obtaining high efficiency of separation, permeability, and selectivity.[66] Several materials can be used to fabricate membranes, as shown in **Figure 4**. Materials fabrication can be generally classified according to their basic materials categories. The first category can be further divided into the following groups: metallic, organic (i.e., polymeric), inorganic, and hybrid membranes. Additive materials are also categorized into hydrophilic, amphiphilic (i.e., copolymer), and nanoparticles.

3.1. Metallic-Based Membrane

Metallic membranes attract much interest in fundamental research with a specific mesh, super water repellency, excellent corrosion resistance, good durability, and buoyant property towards oil/water separation. These membranes have practical features such as high thermal stability, low cost, excellent mechanical properties, and surface coating ability, achieving the desired surface wettability.[67] Wang et al.[68] reviewed porous metallic membranes for oil-water separation and techniques to
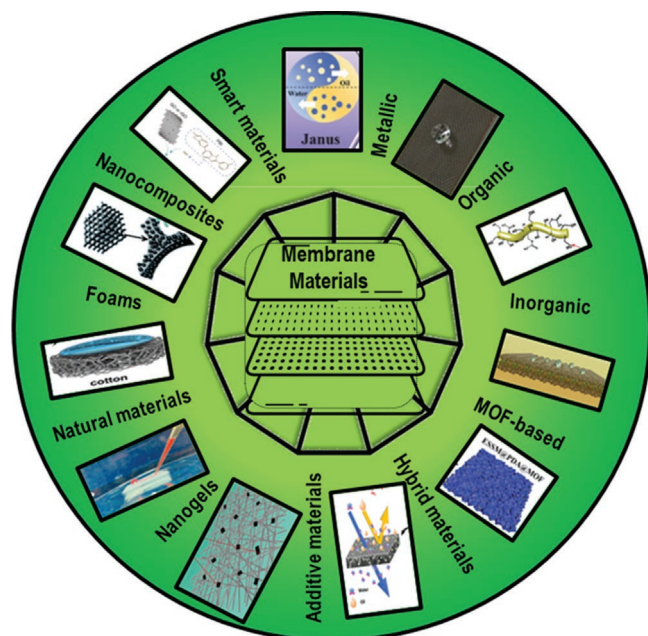
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Figure 4. A chart illustrates the most common membrane materials.

improve their surface properties using different coating techniques. **Figure 5**i shows a 3D superhydrophobic foam iron (SHFI) prepared for continuous oil/water separation. The SHFI was coated with zinc-iron electrodeposited in an alkaline cyanide-free glycerol electrolytic bath, followed by modification of tetradecanoic acid. The resulting SHFI foamed showed high superhydrophobicity (WCA of 158°) and sliding angle of 2°, excellent chemical stability, good anticorrosion, self-cleaning ability, and mechanical stability. Besides, SHFI could achieve high oil/water separation efficiency of 99.3%.[69] Figure 5ii shows another example of a metallic membrane using a superhydrophobic copper mesh using the immersion process as a platform for oil-water separation. The developed membrane was enfolded directly to form a boat-like device that can be used as oil skimmer device. The copper pores allowed the permeation of oils while repelling water completely with 99.5% separation efficiency before efficiently removing the membrane for storing without requiring mechanical handling or recycling. This metallic device can retain its enhanced oil collection for more than ten cycles of oil-water separation.[67]

3.2. Organic-Based Membranes

Polymers are widely used as organic materials in membrane technology due to their advantages, including: chemical stability, high water flux, good mechanical strength, low cost, and ease to manufacture process.[70] However, polymeric materials have limitations of surface hydrophobic, which causes membrane fouling that leads to many negative effects on membrane separation (i.e., decreasing the flux, restricting the permeation rate through the membrane, increasing the required energy, need for continuous cleaning, and decreasing membrane lifetime).[71] Therefore, it is necessary to develop and improve polymeric membrane hydrophilicity. A wide range of poly-

meric materials is used for oil/water separation membrane fabrication. These polymers include; include polyvinylidene fluoride (PVDF),[72,73] polyvinyl alcohol (PVA),[74] polyethersulfone (PES),[75] polysulfone (PSF),[76] polyvinyl chloride (PVC),[23] polypropylene (PP),[77] polyimide (PI),[57] polyethylene (PE),[78] cellulose,[79,80] polyamide (PA),[81] polyacrylonitrile (PAN),[82] and chitosan.[83] **Table 1** summarizes the most common polymeric materials-based membranes used in oil/water separation and their most prominent properties. Low solubility causes weak interactions between the polymer and the solvent. Thus, the solvents have been used to dissolve the polymer that will interact with, and for example, polysulfone is dissolved in N-methyl pyrrolidone (NMP) or *N,N*-dimethyl formamide (DMF).[84] Hence, the chemical structure of each polymer reflects the ability of each polymer to react with solvents. PVDF-based membranes are commonly used for polymeric membranes' oil/water separation applications due to their excellent physical, chemical, and mechanical properties. However, pristine PVDF membranes are mainly hydrophobic, thus promoting organic membrane molecules when immersed in the solution and promoting membrane fouling.[85] As a result, many techniques have been used to improve their characteristics and water permeability to meet oil/water separation (e.g., super hydrophilic/underwater superoleophobic membranes and high flux/high separation efficiency in the oil-in-water mixture). For example, tannic acid (TA) and *N,N*-diethylethylenediamine (DEDAPS) were used to modify a hydrophobic PVDF membrane surface to hydrophilic, and thus resistance to oil-fouling decreased. This contributes to water flux to 4701.6 ± 385.2 L m^{-2} h^{-1} and increases oil rejection (>96%) in multiple consecutive cycles.[85] Furthermore, blending PVDF with other hydrophilic polymers is an alternative way to enhance the separation of the water-in-oil mixture. For example, blending of PES with PVDF using electrospinning showed an improvement in PVDF membrane's wettability and thermal properties after different cycles with good separation performance. The developed PES/PVDF membrane exhibited superoleophilicity/superhydrophobicity. It exhibited WCA, oil contact angle, and contact angle for other liquids of 130°, 18°, and ≥120°, respectively.[86] Muthukumar et al.[87] tuned the PVDF membrane fabricated via phase inversion process morphology using iron alkoxide as a pore-forming additive (PFA) followed by 5% HCl treatment. Due to acid leaching, the porosity was increased, and the membrane efficiency towards oil rejection was 96.5%. In addition, membrane exhibited antibiofouling property against *Escherichia coli* (*E. coli*) and *Bacillus subtilis* (*B. subtilis*).

Polysulfone (PSF) is a hydrophobic polymer commonly used in the membrane in industrial applications. To improve water permeability and fouling resistance, adding hydrophilic materials is the best choice to change membrane to hydrophilic molecules and promote the formation of large pores.[92] For instance, the thermally induced phase separation (TIPS) technique with temperatures from 5 °C to 40 °C was employed to change membrane hydrophobicity at low-temperature, resulting in the excellent rejection of methylene blue.[93] To this end, PSF membrane was prepared using the phase inversion method with PVP, P(VP-AN), PVAc grafted bentonite as the amount of VP prepared as an additive, and AN monomer was used grafted onto bentonite surface over other monomers
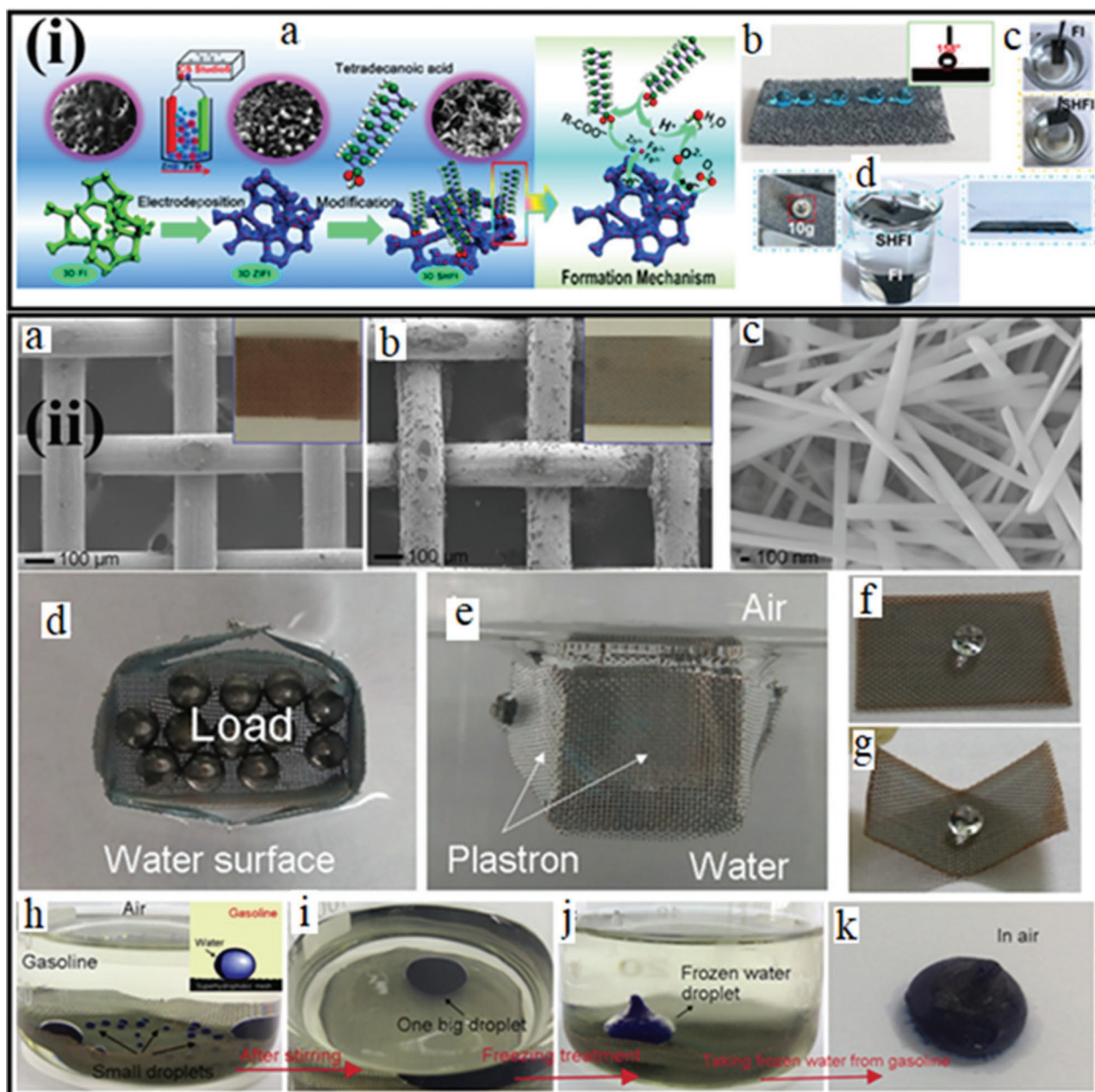
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Figure 5. Examples of metallic membranes: (i) schematic diagram of superhydrophobic 3D ZIFI preparation and associated digital photos of water droplets and immersed membrane in water under external force (a–d). Adapted with permission.[69] Copyright 2020, Elsevier B.V. (ii) membrane surface morphology via FESEM images shows superhydrophobic copper boat floating top view on a water surface and a side view of the weight-loaded superhydrophobic boat immersed in water (a–k). Adapted with permission.[67] Copyright 2017, Elsevier B.V.

via polymerization technique. The membrane performance was enhanced in terms of hydrophilicity by reducing in contact angle of membranes by (47% and 40%) and fouling resistance (fouling attenuated coefficients (≤0.50)).[94] PAN is extensively used for membrane preparation with adequate chemical and thermal stability, solvent resistance, and poor hydrophilicity. The presence of nitrile (−CN) groups in PAN react with other chemicals, leading to the functionalizing of PAN during the fabrication process.[82] **Figure 6**i shows the PAN membrane's hydroxylamine-induced phase inversion process. Fabrication processes include the addition of NaOH into the coagulation

bath, which induces in situ hydrolysis of −CN groups in the PAN chain to −COOH groups. The developed PAN membranes showed superior antifouling properties and recyclability attributed to their ultralow-oil-adhesion property.[92] The obtained PAN membrane induced a high flux ranging from (2200 to 3806 L m^{-2} h^{-1} bar^{-1}) and showed separation efficiency in various oil-in-water emulsions. This is attributed to a contact angle <1° (left) and an underwater oil droplet on the membrane at 156° (right). Cellulose acetate (CA) is another polymeric material that has been widely used in oil/water separation. CA has many advantages: abundant sources, excellent fouling

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Table 1. Common polymeric membrane materials, properties, solvents, and chemical formulas.

Material	Properties	Solvents	Formula	Refs.
Polysulfone (PSF)	• High thermal stability • High mechanical properties • Hydraulic stability • Superior chemical resistant • Widely used in many separations processes • Hydrophobicity	N-methyl pyrrolidone (NMP) N,N-dimethylformamide (DMF)	$(C_{27}H_{26}O_6S)_n$	[88]
Polyethersulfone (PES)	• Transparent amorphous • High-T_g polymer • High mechanical strength • Thermal and chemical resistance	N, N-dimethylacetamide (DMAC)	$(C_6H_4\text{-}SO_2\text{-}C_6H_4\text{-}O)_n$	[89]
Cellulose acetate (CA)	• Hydrophilicity • Moderate flux • High salt rejection properties • Thermal stability nontoxicity poor mechanical strength	Dichloromethane (DCM), acetone	$(C_6H_7O2(OH)_3)_n$	[90]
Polyacrylonitrile (PAN)	• Good chemical and thermal stability • Antisolvent property • Its intrinsic nature of poor hydrophilicity	N-methyl-pyrrolidone (NMP)	$(C_3H_3N)_n$	[82]
Nylon6	• High mechanical strength • High resistance to abrasion chemicals • Extensive surface area • High porosity • High hydrophobicity	Formic acetic acids	$(C_6H_{11}NO)_n$	[63]
Polyvinylidene fluoride (PVDF)	• Good chemical resistance • High mechanical strength, • Thermal stability • Excellent aging resistance, • Very important for the actual application of separation membranes • Good process • Hydrophobicity	N, N-dimethyl acetamide (DMAc), Dimethylformamide (DMF) N-methyl-2-pyrrolidone (NMP).	$(C_2H_2F_2)_n$	[72]
Polyamide (PA)	• Hydrophilic material • Low coefficient • Good toughness • Resistance to abrasion and fatigue • High tensile strength	–	$C_{18}H_{35}N_3O_3$	[91]

resistance properties, environment friendliness, and biodegradability.[95] However, the hydrophilic nature of these materials slows the diffusion of water. This property plays a role in retarding coagulation during the phase inversion process and thus results in a denser skin layer and subsequently lower flux and exhibits low mechanical strength (chemical and thermal resistance).[96,97] Several studies suggested blending CA with other polymers or additives to overcome these limitations. For instance, investigated membranes made from CA were mixed with a polymer additive with both hydrophilic and oleophobic groups (i.e., perfluoroalkyl poly ethoxy acetic acid (FPEOAA)). Thus, water-permeable blend CA membranes showed pure water flux increasing from 0 to 34 L m^{-2} h^{-1} bar^{-1} with improved surface hydrophilicity (WCA of 57.1°) and (enhanced tensile stress of 1.57 MPa and tensile strain of 3.14%).[98] In another study, nanofiber membrane from CA polymer and hydrophobic PSF polymer as a core and shell alternatively with 0.1 wt% of ZnO nanoparticles (NPs).[95] The membrane had a good oil/water separation and exhibited antibacterial activity against *E. coli* bacteria. Figure 6ii shows the fabrication of deacetylated

CA (d-CA) membrane via electrospinning and deacetylation process of CA nanofiber membranes. The d-CA nanofiber membrane worked as oleophobic in water, superamphiphilic in air, and superhydrophilic in oil and worked perfectly with gravity. The d-CA nanofiber membranes resulted in the highest separation flux of 38000 L m^{-2} h^{-1} and separation efficiency of 99.9% to separate a mixture from chloroform/water under gravity.[96]

Ma et al.[57] developed flexible polyamide acid (PAA) fibrous oil-water separation membranes. The PAA was synthesized with ether linkages in backbone and heavily fluorinated polybenzoxazine (F-PB). The electrospinning technique was used to form CA and PAA as a coaxially nanofiber; the PAA core was treated at high temperature to obtain core/shell structured from CA/polyimide nanofiber membranes. Furthermore, the fiber's surface was modified with F-PB, with and without silica nanoparticles (SNPs, **Figure 7**i). Compared with CA fibrous membranes, the developed membranes show much better mechanical properties with tensile stress and tensile strain of 130 MPa and 52%, respectively. There have been many attempts to
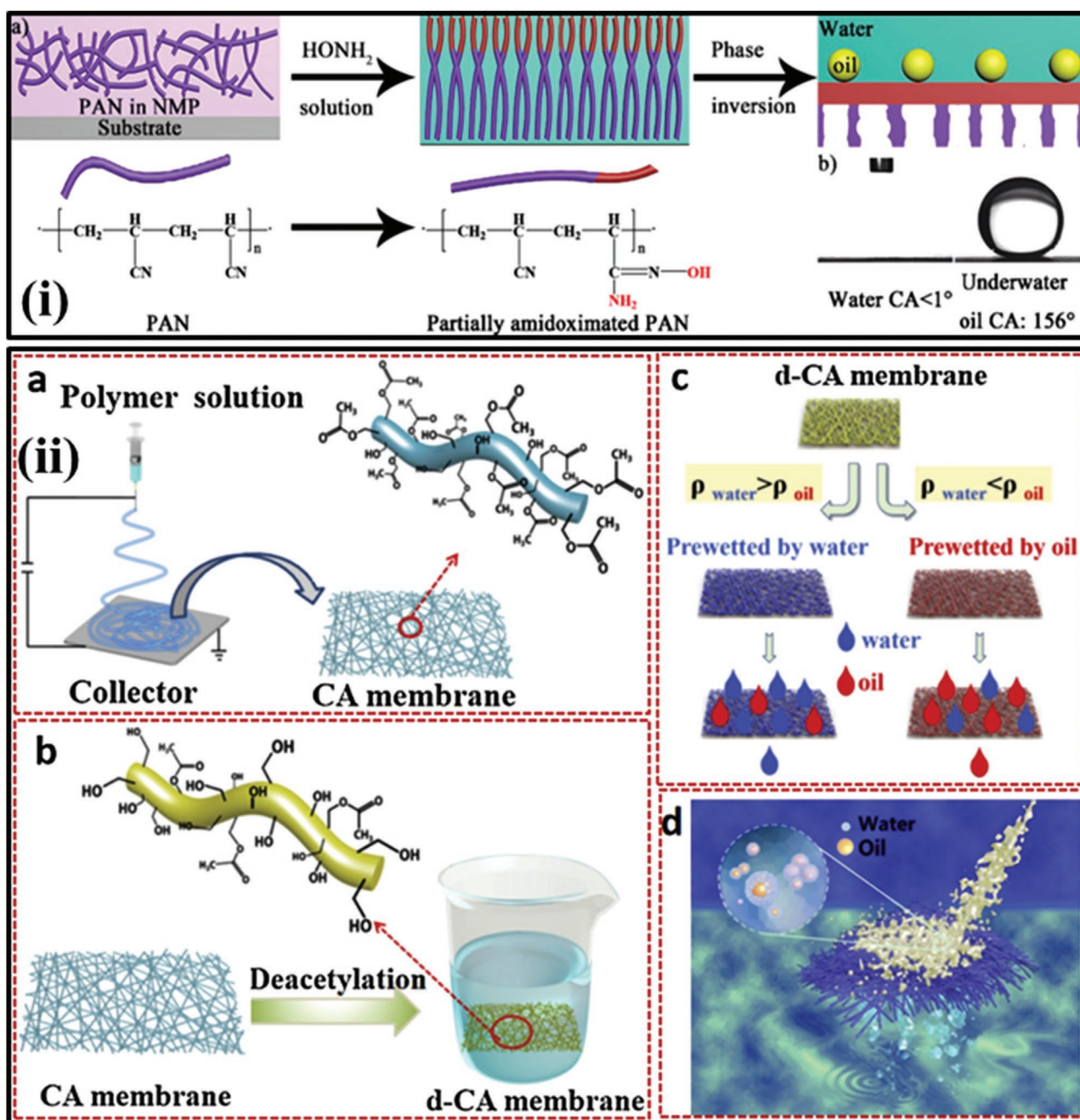
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Figure 6. i-a) schematic illustration of PAN membrane with a hydroxylamine-induced phase-inversion technique and corresponding water droplet on the membrane. Adapted with permission.[92] Copyright 2017, Elsevier B.V. ii-a) Electrospinning setup of CA nanofiber membrane, b) deacetylating of CA nanofiber d-CA nanofiber membrane. c) Schematic diagram of oil/water mixture based on selective separation, d) schematic diagram illustrating emulsified oil/water separation. Adapted with permission.[96] Copyright 2019, Elsevier B.V.

decrease polyethersulfone (PES) commercial membrane fouling which are: 1) membrane surface modification, 2) pretreatment of feed, 3) operating parameters, and 4) cleaning procedure.[99] The surface modification technique focuses on improving the membrane surface properties such as wettability and polarity without any changes in the bulk materials of the membrane.[100] Surface modification process/techniques can achieve using: 1) coating, 2) chemical, 3) composite, 4) blending, 5) grafting, and 6) a combination of these methods; these techniques

are discussed in details in ref. [100]. Extension of the surface modification process, including coating, can be achieved via several methods such as UV-assisted graft polymerization,[101] graft polymerization initiated either by plasma or corona discharge,[102,103] plasma and corona modification.[104,105]

Prince et al.[75] developed a PES ultrafiltration (UF) membrane; they introduced a simple method to increase the PES hollow fiber membrane hydrophilicity via carboxyl, hydroxyl, and amine-modified graphene attached to poly acrylonitrile-co-maleimide
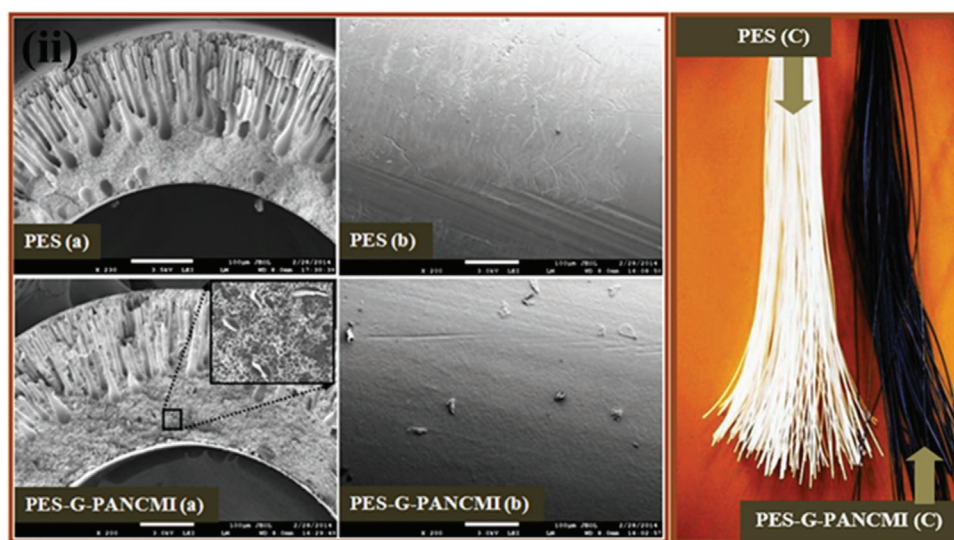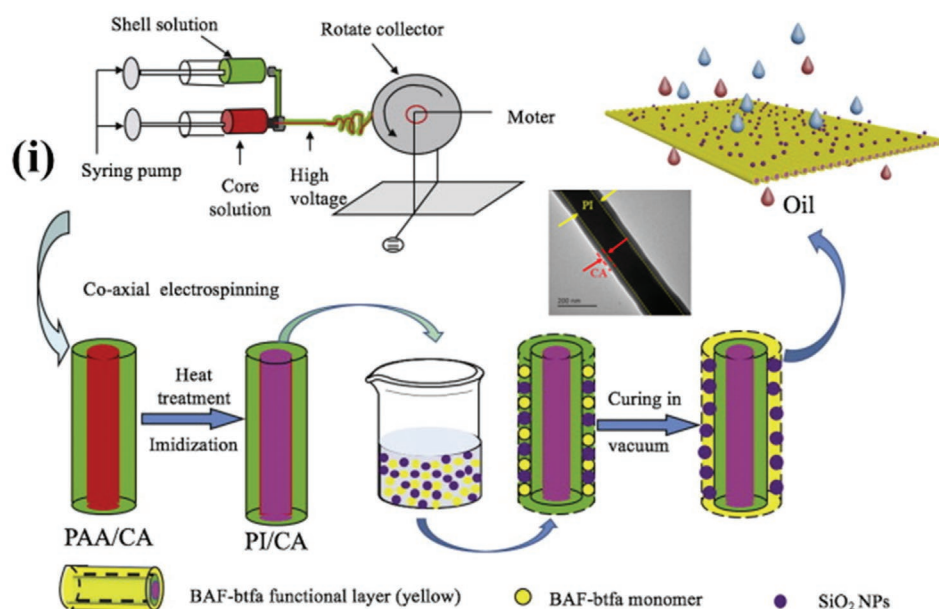
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Figure 7. i) Scheme illustrates co-axial electrospinning setup for FPB/SNP nanofiber membrane modified with PI/CA: membrane designed as cellulose-acetate (CA-shell), polyamide acid (PAA-core), polyimide (PI-core). Adapted with permission.[57] Copyright 2016, Elsevier B.V. ii) SEM images of PES-G-PANCMI membranes and cross-section view and membranes optical image. Adapted with permission.[75] Copyright 2016, Elsevier Ltd.

(G-PANCMI). PES/SPES (sulfonated PES) blended membranes used the phase separation technique through sulfonation of PES by chloro-sulfonic acid and concentrated sulfuric acid for 2 h at 10 °C. The resulted membrane showed pure water flux of (2374.1 L m^{-2} h^{-1}) and antifouling of 77.4%, which was mainly attributed to membrane hydrophilicity and porosity.[106] Figure 7ii shows morphology of the developed membrane with characteristics of water and oil contact angle, water permeability, liquid entry pressure of oil, and finally subjected to a continuous 8 h filtration test of oil emulsion in water. The treatment of PES membrane with G-PANCMI improved WCA and oil contact angle from 63.7 ± 3.8° to 22.6 ± 2.5° and 43.6 ± 3.5° to 112.5 ± 3.2°, respectively.[75] These changes correspond to a 158% higher contact angle compared to that of the PES membrane. These observations indicated that G-PANCMI plays a role in the PES membrane's hydrophilicity, permeability, and selectivity.

Nylon-6 is a versatile polymeric membrane with several advantages: high thermal/chemical stability, good mechanical strength, and good abrasion resistance. However, the limitations of the nylon 6,6 membrane are mainly fouling. Nylon 6,6 nanofiber mats were improved via solvent vapor treatment using formic acid vapor to overcome this limitation. After 5 h of exposure, the tensile strength improved from (738 to 1950 MPa), and membrane fouling occurred after 220 min compared to the untreated mats.[107] Moreover, Nylon was used as a substrate with a favorable film-forming property of starch made the coating materials easily and stably adhere to the nylon membrane.[108] A polyvinyl acetate (PVAc)-coated

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nylon 6/silica microfiltration membrane was prepared using an electrospinning technique for separation of emulsions wastewater, i.e., oil/water (O/W). The developed MF membrane showed high flux and antifouling with robust mechanical properties (i.e., the tensile strength of 23.3 MPa); the MF membrane had porosity and average pore size of 78% and 170 nm, respectively. A strong interaction between the electrospun nanofiber mat and the PVAc coating supports the membrane's structural stability. Moreover, membrane achieved a water permeability of 4814 LMH/bar and oil rejections of 98.8, 99.0, and 99.2% from oil concentrations of 250, 500, and 1000 mg L^{-1}, respectively, at four psi of applied pressure during MF of O/W emulsions with antifouling properties and a water flux recovery of 85%. PA is high-performance material with good properties, such as excellent mechanical strength, high fatigue resistance, good toughness, abrasion resistance, superior high thermal resistance, and low coefficient of friction.[109,110] Given these properties of PA, it is usually used as an active layer on the surface of membranes for O/W separation. PA layer (with nanosized thickness) is commonly fabricated via the interfacial polymerization (PI) technique of m-phenylenediamine (MPD) and trimethyl chloride (TMC) on nanoporous membrane supports.[111,112] Adding a polyamide layer is the best and most common industry among the various surface modification methods (i.e., plasma technique, chemical vapor deposition) for improving antifouling. Zhang et al.[113] developed a PSF membrane substrate fabricated by the phase separation method. Thin-film composite (TFC) from the PA layer was fabricated via the reaction between MPD and TMC; the TFC membranes surface was modified by grafted of amine-terminated fully disulfonated poly (arylene ethers) (NH$_2$-BPSH100). It was also found that TFC membrane-modified TFC membrane enhanced fouling resistance to emulsified oil. Commercial polyamide membrane surfaces can provide few terminal functional groups, restricting the initiator stabilization.

3.3. Inorganic-Based Membranes

Inorganic compounds are another membrane materials class that show rapid progress in membrane technology. Inorganic-based membranes are developed using either metals or ceramics materials. Thus, these categories have excellent wear resistance, high working temperature (up to 500 °C), chemical stability, and long lifetime. On the other hand, limitations of the inorganic membrane due to different thermal expansion led to sealing problems, and besides that, brittleness is another challenge that needs careful handling. Such kinds of membranes can be classified into two types based on porosity; nonporous (i.e., dense) membranes (e.g., metals) and porous membranes (e.g., metal or ceramic). Several inorganic membranes have been applied in water treatment, desalination, and wastewater mainly used to treat oily wastewater.[66,114,115] Ceramic membranes are used most widely in wastewater treatment, such as silica (SiO$_2$), alumina (Al$_2$O$_3$), zirconia (ZrO$_2$), titania (TiO$_2$), graphene oxide (GO), and carbon nanotube (CNT).[116] There are many efforts focused on the progress of ceramic membranes with various techniques.[117–120] Furthermore, modifying the surface of the ceramic-based membrane to improve the wetting

behavior is one way to enhance the membrane's efficiency and antifouling performance during oil/water separation. For example, alumina was used as a matrix to synthesize ceramic MF membranes treated with silica nanoparticles to enhance hydrophilicity properties and increase water flux (350 L m^{-2} h^{-1}) for oil/water separation with high oil rejection 93%.[121] Using phase inversion technique to manufacture a β-SiAlON membrane followed by graphene oxide (GO) to modify the β-SiAlON membrane surface by dip-coating was used for the separation process. The surface modification of the membrane leads to an increase in the surface hydrophilicity of the β-SiAlON membrane through the WCA decrease from 73° to 0° and an increase in the antifouling of the β-SiAlON/GO membrane.[122] Ceramic membranes are usually fabricated from metal oxides, characterized by superhydrophilic surfaces so that hexadecane thiol (HDT) is grafted on Al$_2$O$_3$ membrane surface was modified to superhydrophobic HDT-Ag-PDA-Al$_2$O$_3$ membranes, which modified membranes preparation steps with oil and of 155° WCA.[123] Zhang et al.[124] reported an inorganic membrane made from cupric phosphate (Cu$_3$(PO$_4$)$_2$) with nanosheets structure, as shown in **Figure 8**i. The developed membrane showed superhydrophilic and underwater antifouling for oil and anti-biofouling properties, thermal stability, and salt tolerance. The Cu$_3$(PO$_4$)$_2$ nanosheets possessed an extreme water absorption energy higher than other inorganic materials. Subsequently, Cu$_3$(PO$_4$)$_2$ nanosheets wrapped mesh membrane showed superhydrophilic with superior low oil adhesive force. The strong hydration of Cu$_3$(PO$_4$)$_2$ nanosheets ability endowed the membrane with good antifouling properties and underwater superoleophobic properties. The superhydrophilic properties of Cu$_3$(PO$_4$)$_2$ nanosheets are due to the 3D porous structure, surface functionality, and relatively weak mechanical strength.[124] These properties promote viscous oils with high separation efficiency and long-term stability during separation process of oily wastewater. In addition, Cu$_3$(PO$_4$)$_2$ nanosheets-wrapped mesh membrane has excellent potential for purifying natural oily wastewater and treating high-viscosity oil-polluted water. An economical and environmentally friendly membrane was developed from ZnO-Co$_3$O$_4$ overlapped for oil/water mixture separation using the hydrothermal method, as shown in Figure 8ii.[125] A metallic-based membrane is being used as an inorganic membrane modified with other ceramic materials. For example, pure inorganic MnCO$_2$O$_4$ coated stainless steel mesh (MnCo$_2$O$_4$-SSM) was developed through the annealing method.[126] The MnCo$_2$O$_4$-SSM showed excellent properties, including antifouling, ultrahigh flux (>63 L m^{-2} s^{-1}), high-efficiency oil/water separation ability (>99.9%), and superior recycling stability. Furthermore, the membrane showed robust mechanical properties, chemical stabilities, and thus long-term durability.

3.4. Metal-Organic Framework Membranes

Metal-organic frameworks (MOFs) are a new membrane material class composed of organic–inorganic composite.[127–131] They are formed via the self-assembly of metal ions in the center and organic ligands with porous and periodic network structures.[132] MOFs membranes attract much attention due to their advantageous properties, including large specific
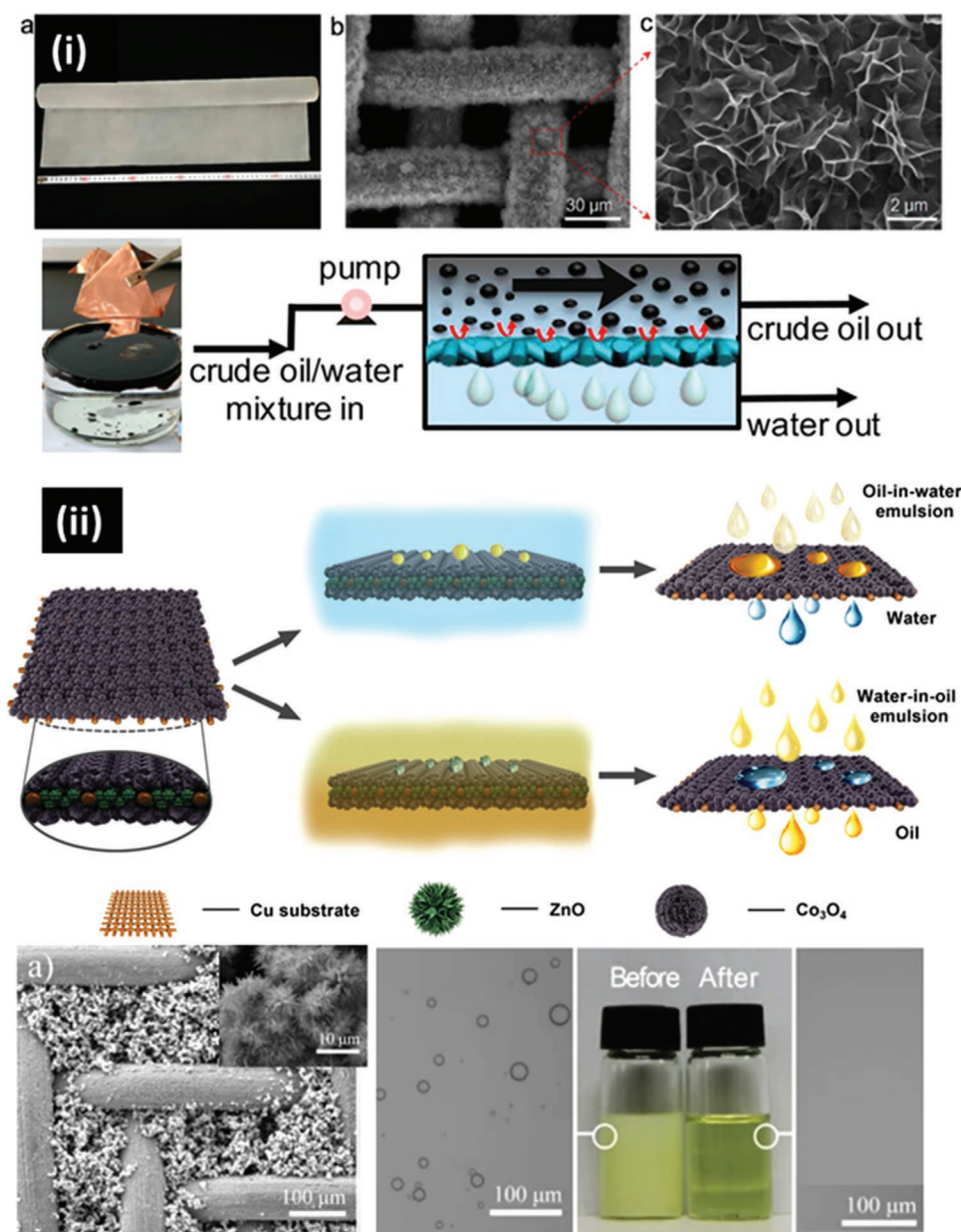
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Figure 8. The inorganic-based membranes; i) membrane structure characterization with photographic images and SEM images beside crude oil used to demonstrate the anti-oil-adhesion and self-cleaning performance and crossflow oil/water separation. Adapted with permission.[124] Copyright 2018, American Chemical Society. ii) Schematic description of inorganic ZnO-Co$_3$O$_4$ overlapped membrane structure and switchable wettability when immersed in different media and the corresponding separation capacities of oil/water emulsions, as well as the SEM images of the inorganic membrane and the results of the span80-stabilized water-in-diesel emulsion. Adapted under the terms of the CC-BY 4.0 license.[125] Copyright 2015, The Authors, published by Springer Nature.

surface area, controllable pore structure, and good thermal & chemical stability. MOF-based membranes have been successfully developed and widely used in different applications such as separation, sensing, optics, catalysis, and metal protection.[133] Numerous different synthetic approaches, including hydrothermal (solvothermal),[128] slow diffusion,[129] mechanochemical, electrochemical, heating, microwave-assisted, and ultrasound, can be applied to produce structures and features MOFs membrane.[130] **Figure 9**i displays the synthesis and modification of MOF@GO nanocomposite UiO-66-NH$_2$ (N-UiO-66), a hydrophilic stable MOF was grown on the GO nanosheet,

then poly (acrylic acid) (PAA) was used to modify of nanocomposites. A vacuum-assisted self-assembly process was reported to fabricate N-UiO-66@GO-PAA nanocomposites MOF intercalated GO membranes into adjacent GO nanosheets. Moreover, the membranes achieved significantly increased water permeability (5067 L m^{-2} h^{-1} bar^{-1} under optimum conditions) in addition to high rejections for oil/water emulsion (>99.9%) due to low membrane thickness and extended interlayer distance.[132] On the other hand, because of the hydrophilic chemical structures and typical micro/nano-topological structures, the MOF-intercalated GO membranes showed that underwater
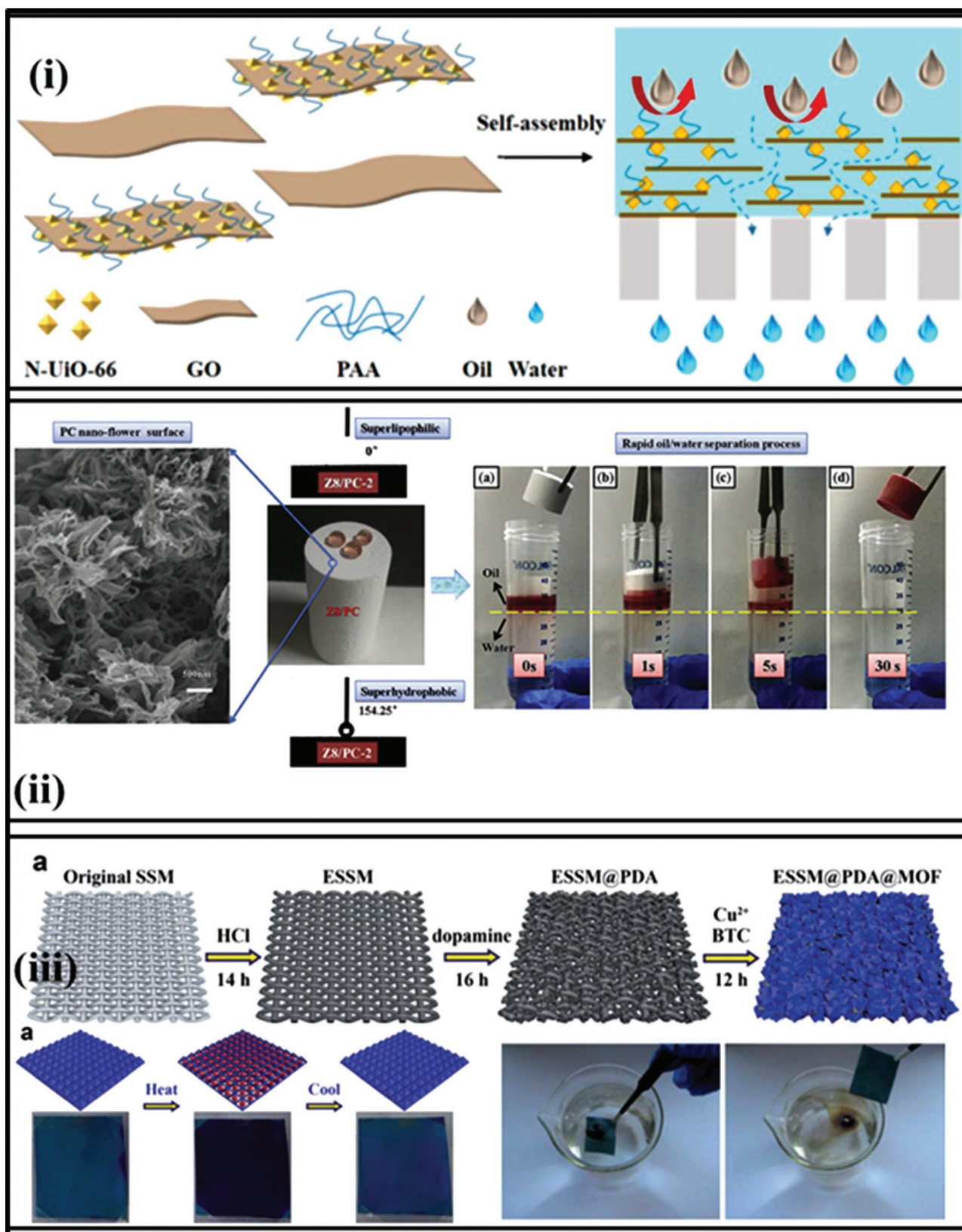
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Figure 9. i) Metal-organic framework (MOF)@GO nanocomposite. Adapted with permission.[132] Copyright 2020, American Chemical Society. ii) separation process of diesel oil with Z8/PC-2 monolith MOF membrane, Adapted with permission.[47] Copyright 2020, Elsevier Ltd. iii) MOF-based film procedure and color change of ESSM@PDA@MOF after the heat and cold treatments and antifouling tests with self-cleaning ability against crude oil of the MOF-based film. Adapted with permission.[135] Copyright 2018, Royal Society of Chemistry.

superoleophobicity and fouling resistance property with a flux recovery ratio (FRR) above 80% after the three-cycle oil/water emulsion separation. The membranes accomplished satisfying operational and structural stability caused by multiple interactions in respect of electrostatic interactions and covalent and hydrogen bonds.[132] Another example is shown in Figure 9ii. A metal-organic framework (Z8/PC) was used to modify a series of superhydrophobic polycarbonate porous monoliths that were first manufactured via a simplified thermally affected nonsolvent induced phase separation technique for efficient selective oil/water separation.[47] The methods for evaluating the performance of the monoliths for oil/water separation include selectivity, corrosion resistance, kinetics, equilibrium adsorption capacity, and circulation. MOFs materials such as zeolitic imidazolate frameworks (ZIF-8) significantly compensated for the shortage of pure monolith. The hydrophobic angle of the pure PC and Z8/PC-2 monolith upgraded from 136.18° to 154.25°, respectively, because of the micro-nanoflower surface. Meanwhile, the Z8/PC-2 monolith exhibited a more complex and continuous interconnected 3D hierarchical structure containing micro–nano regimes, which had the monolith with a higher specific surface area and porosity of 146.84 m^2 g^{-1} and 89.5%, respectively. A more superior oil/water separation ability of Z8/PC-2 monolith was demonstrated through the selective removal of oil or organic solvent from water within 30 s, a highly balanced adsorption capacity, and outstanding corrosion resistance. In addition, the ten-cycle regeneration of porous monoliths by centrifugation or evaporation showed extra attractiveness.[47] In oil, highly hydrophilic surfaces are usually challenging to be achieved due to the high surface tension of water. Example from nature, sarcocarps such as Chinese yams can retain water to maintain freshness. Inspired by the distinctive wetting properties of sarcocarps, MOFs can be spontaneously detention moisture from the atmosphere have been proposed as building blocks for underoil super-hydrophilic surfaces. A popular HKUST-1 (MOF-199; $Cu_3(BTC)_2$, BTC = 1,3,5-benzenetricarboxylate) was tightly coated on stainless-steel meshes via a mussel-inspired preparation method, as shown in Figure 9iii. The MOF surfaces can achieve high efficiency and display outstanding self-cleaning properties to crude oil underwater, on-demand separation by selective water filtration and adsorption. MOFs water adsorption can be comprehensive for other extreme wettability and interfacial issues.[134] High energy consumption, low efficiency, and difficulties in recycling and reprocessing a developed MOF-coated super wetting membrane for multichannel oil/water separation and collection of floating oils are considered complicated problems that traditional oil/water separation methods suffer from it. The most important feature of this process is that it is very flexible in manipulation and can be accomplished for one hour under a low temperature without using high pressure. This strategy offered to surface super-wetting membrane properties due to the introduction of interlayers of $Cu(OH)_2$ nanowires, which build the appropriate hierarchical structures and act as templates that were partially sacrificed to further growth hydrophilic MOF nano-whiskers. The as-prepared mesh has high flexibility, so this super wetting membrane can be used for multichannel oil/water separation applications, involving continuous oil/water separation, gravity-driven oil/water separation, and floating oil collection.

Moreover, the separation efficiency and flux of the super wetting membrane preserve high and stable under several separation cycles.[135]

3.5. Hybrid Materials-Based Membranes

Hybrid membranes incorporate different materials so that each substance's distinct properties and specific function to the membrane properties. The substance properties depend on the degree of dispersion, adhesion in the load–matrix interface, the orientation of the load in the matrix, and the aspect ratio.[136] Hybrid membranes fabricated from organic, and inorganic are easy to prepare and enhance membranes' properties. For example, metal oxide nanoparticles are used as nanofillers such as ZnO, TiO_2, ZrO_2, and SiO_2 to improve membrane performance such as salt rejection, thermal resistance, and chemical and mechanical stabilities.[137,138] Hybrid membranes are composed of organic and inorganic materials to develop and improve membrane separation and eliminate membrane defects by enhancing membrane properties, including fouling resistance, permeability, and wettability.[139] For example, an economical and straightforward superhydrophobic hybrid membrane for effective oil/water separation was developed using a cellulose membrane (CM) filter. The developed membrane is ecofriendly and resistant to fouling for oil/water separation and extended its application to waste particle filtration and to block thiol-based odor materials. A reversible addition–fragmentation chain transfer technique was used to improve the superhydrophilic nature of The CM into a superhydrophobic surface.[140] A hybrid $PVDF/Al_2O_3$ membrane was fabricated via a casting solution, and the role of Al_2O_3 is to enhance membrane wettability, and further surface modification with polyacrylamide (PAM) was performed. The $PVDF/Al_2O_3/PAM$ membrane was characterized by superhydrophilic/underwater superoleophobic and improved flux compared to the pure PVDF membrane.[136] Moreover, $PAN-GO-SiO_2$ hybrid membranes for oil/water separation showed good mechanical, chemical stability, and antifouling properties. The flux of separated water was enhanced from 2600 L m^{-2} h^{-1} for pristine PAN to 3151 L m^{-2} h^{-1} for $PAN-GO-SiO_2$.[141] Incorporation of nanoparticles significantly affects the membrane; for instance, PES/ZnO NPs incorporation followed by UV irradiation treatment showed a unique property. The modified PES/ZnO NPs membranes had improved flux from 16 to (25–28) L m^{-2} h^{-1}; membrane hydrophilicity was enhanced by achieving a contact angle value from 62° to 58°.[142] A membrane of $TA/PEI@TiO_2$ hierarchical hybrid nanoparticles (TPTiHHNs) was prepared via a simple strategy combining several catechol chemistries of phenolic tannic acid (TA) with the biomimetic mineralization chemistry of titania. The nanoparticles of $TA/PEI@TiO_2$ were added as nanofillers to prepare PVDF/TPTi hybrid membranes. $TA/PEI@TiO_2$ offers a membrane with good hierarchical structure, good roughness, higher porosity, underwater superoleophobicity, and greater hydrophilicity. Upon TPTiHHN loading, the PVDF/TPTi hybrid membranes show antifouling performance. The flux recovery ratio in oil-in-water emulsion separation was 92%. The PVDF/TPTi membrane showed high flux recovery ratio of about 85% up to three cycle of separation of oil-in-water emulsion at higher concentration.[139]

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3.6. Membranes Materials Additives

During fabrication, additives materials to membranes play an essential role in preparing polymeric membranes and improving membrane performance. Additives have a different role and effect on the preparation of the membrane and enhance the desired properties. Hydrophilic polymers as macromolecules additives blended with membranes polymeric material in the preparation process can achieve special wettability and fouling properties. Most hydrophilic polymers are applied as pore-forming agents (i.e., porogen) to form porous structures and hydrophilic membranes.[143] For example, poly-vinyl pyrrolidone (PVP) is a common hydrophilic polymer used for pore-forming because it can enhance membranes' hydrophilicity properties[144,145] and increase the antifouling with high water flux.[146,147] PVP increases different properties such as pore density and improves the membrane's water permeability and mechanical properties when blended with the PSF.[148] On the other hand, polyetherimide (PEI) has excellent properties as a hydrophilic polymer, such as asymmetry structure forming ability, high tensile strength, thermal resistance, appreciable hydrophilicity, and outstanding chemical resistance.[149] Further enhancement can be achieved when PSF membranes are coated with PEI and improved mechanical properties with strength 604–2257 N m^{-2}, and pure water flux of 5773–91398 L m^{-2} h^{-1} increased membrane pore density to 0.75 nm.[150] Amphiphilic copolymers additives consist of hydrophobic and hydrophilic, which are used as additives incorporated with the host polymer. During membrane preparation, amphiphilic additives hydrophilic polymers are inclined to deposit on the membrane surface or the pore to decrease the interfacial energy of the membrane surface. However, a hydrophobic polymer combined with the chains of membrane polymer leads to depositing a hydrophilic polymer.[151] These materials' most interesting advantages are improving membrane performance toward flux and rejection for the protein.[152] Amphiphilic polymers additives used to develop membranes are potential oil/water separation.[153–156] For example, HP(AN-MA) copolymer prepared using emulsion polymerization was successfully used as a matrix to fabricate and modify the PAN membrane via TIPS with excellent super-hydrophilicity and high water fluxes, which is up to 4341 L m^{-2} h^{-1} under low operating pressure (0.02 MPa).[157] Additionally, poly(AA-co-ACMO) copolymer synthesized by precipitation polymerization as an additive to modifying the PSF membrane prepared by the phase inversion method improves hydrophilicity. The contact angle decreased from 59.3° to 44.4°, and the flux ratio increased from 0.61 to 0.92.[158] Amphiphilic PSF-b-PEO additives with PSF homopolymer to prepare UF membranes via the nonsolvent-induced phase separation method.[154] Inorganic materials such as nanoparticles are common additives for polymeric membranes in novel membrane preparation or surface modification. Nanoparticles have advantages such as reducing membrane fouling and improving membrane permeability and solute rejection. Various nanoparticles were used to introduce nanocomposite membrane in separation applications such as TiO$_2$,[159] SiO$_2$,[160] ZnO,[161] CuO,[162] CNTs,[163] GO,[164] Al$_2$O$_3$,[165] and Fe$_3$O$_4$.[166] These nanoparticles can be used alone or dispersed in other materials such as polymeric membranes. For example, 1D metal oxide such as Al$_2$O$_3$ nanowires were dispersed onto 2D materials such as GO nanosheets. GO and GO–Al$_2$O$_3$ were incorporated into the poly(vinylidene fluoride) (PVDF) via the vacuum filtration method. These nanoparticles improved the hydrophilic properties of PVDF membranes.[167]

3.7. Nanogel Materials-Based Membrane

Nanogels have a great potential in membrane materials due to their unique properties such as improving nanofibers surface roughness underwater in harsh water environments including high salinity, acidic and basic solutions. In addition, membrane neutral electrically helps effectively separate surfactant in oil/water emulsions at low-pressure-driven filtration. Bioinspired superwetting materials enhance antifouling properties and thus improve separation efficiency.[168] Sulfobetaine-based zwitterionic nanogels modified by polyacrylonitrile (ZPAN) nanofibrous membrane offered super wetting surfaces (**Figure 10**). Zwitterionic nanogels grafted the surface of electrospun polyacrylonitrile (PAN) nanofibers to create a hierarchical structure that can improve the membrane's fouling resistance performance and surface roughness. Under widespread salt concentrations and pH values, the ZPAN membrane has super-hydrophilic and underwater superoleophobic properties. The ZPAN membrane can successfully separate oil and water emulsions under driven solely pressure by gravity with separation efficiency of > 99.6% and permeation fluxes of 32 800 L m^{-2} h^{-1} bar^{-1}. Moreover, the ZPAN membrane indicated a high potential for effective low-pressure separation of oil-in-water emulsions because of its solid mechanical property and stable reusability.[169]

3.8. Bioinspired Based Membrane Design

A bioinspired design structure obtained low water and high oil contact angles and a superhydrophilic membrane.[170] For example, hydrogel hybrid coating on hydrophobic PVDF can boost the oil/water separation process;[171] furthermore, advanced membrane mixed matrix membranes are efficient for CO$_2$ separation strategies.[172] Covalent organic framework (COF) exhibited ion removal in both aqueous and organic solvents with ultrafast precision sieving for molecular separation.[173] A membrane of PAN/TiO$_2$ can efficiently separate the oil–water mixture. Furthermore, PNIPAM/PAN/TiO$_2$ membrane has a bionic fish scale structure and wettability to comply at lower critical solution temperatures. The membrane's design decreased the water flux from 10013 ± 367 to 7713 ± 324 L m^{-2} h^{-1}, and hence can be used in the purification of reclaimed water and separation of oil-in-water.[174] It is expected that incorporating electrospinning/electrospraying with the bioinspired technique will result in the fabrication of super wetting sphere surface membranes with interconnected pores for other selective separation applications.[175] To adapt surface wettability, an intriguing property is inspired by nature. Using PDA and an amphiphilic synthetic copolymer, an innovative separation membrane was coated via functionalized steel mesh by the mussel-inspired dip-coating method.
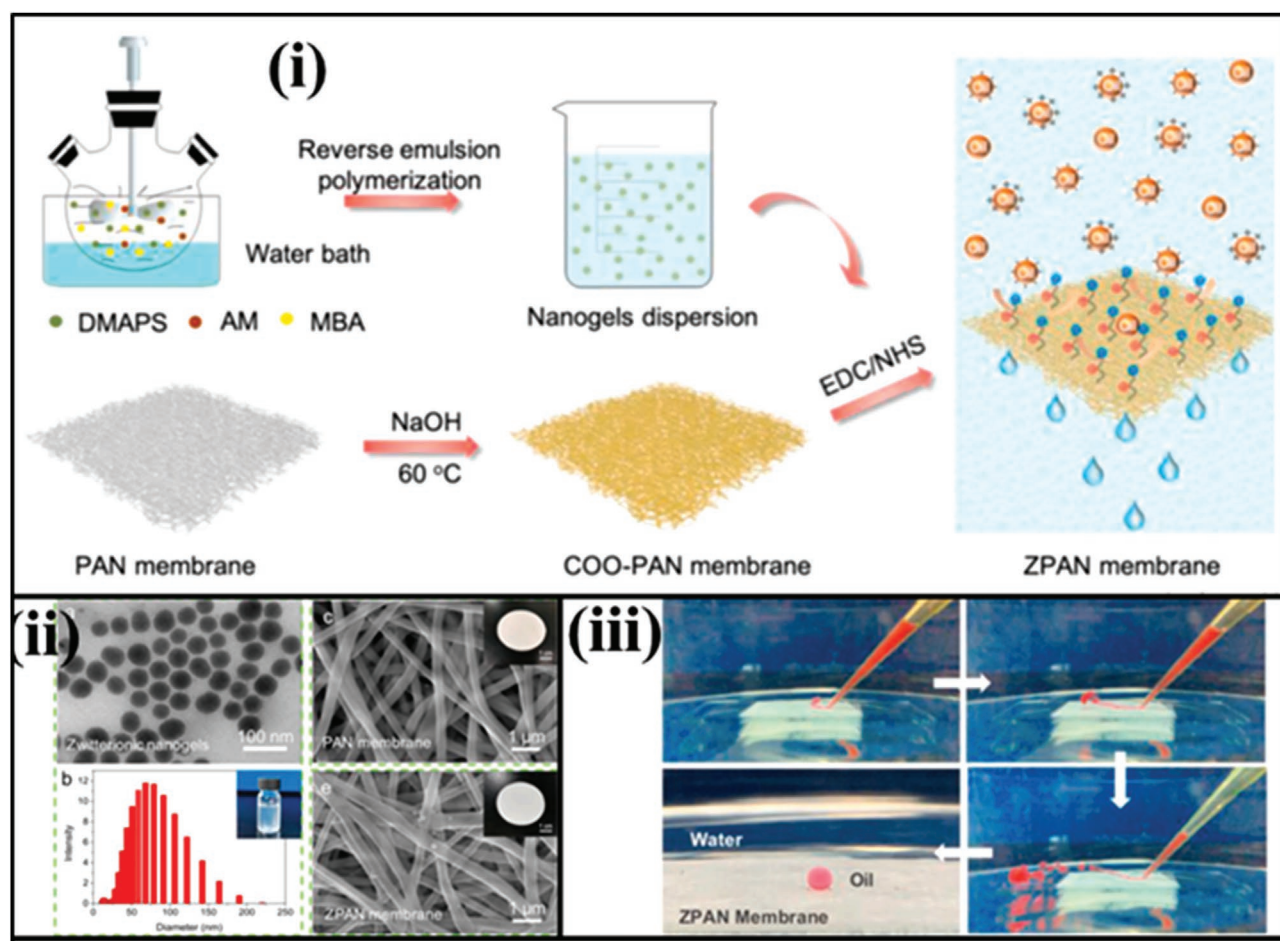
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Figure 10. i) Schematic diagram of the synthesis procedure of zwitterionic nanogels dispersion and PAN nanofibrous modification. ii) TEM and b) SEM images of the zwitterionic nanogels iii) underwater anti-oil-adhesive performance of the ZPAN membrane. Adapted with permission.[169] Copyright 2020, Elsevier B.V.

The nature inspired was distinguished excellent adjustable wettability and can change their special oil/water wettability to selectively activate "water-removing" or "oil-removing" modes for on-demand collection of the desired component (water or oil) from the oily water, i.e., upon prewetting the surfaces with water or oil as shown in **Figure 11**i.[170] Bioinspired PDMS sponges have appreciated means for isolating spilled oil. Figure 11ii shows improvement in the absorption of PDMS sponges with bioinspired design. 3D printing was used to achieve the production of negative designs templates, and that was filled with PDMS, then obtained templates were selectively dissolved. The sponge has achieved the highest hydrophobicity and oleophilic, fabricated with a line width of 200 μm and pore size of 400 μm. This design has a capacity of more than 3.7 times that of the sponge and provided a valuable advance in oil-water separation.[176]

3.9. Natural Materials-Based Membrane

Natural sponges and vegetable waxes were used to prepare a superhydrophobic and superoleophilic oil/water separation membrane via a fast and straightforward emulsion

immersion.[177,178] Wang et al.[179] developed fast, and straightforward natural renewable sponges and vegetable waxes membrane. It has the ability to absorb different oils from water with a fast oil absorption rate, high oil absorption capacity, fast oil absorption rate, and good recyclability and apply to squeeze to realize the reuse of the absorbed oil. In addition, the as-prepared natural material demonstrates excellent resistance to corrosive aqueous solutions contacting, strong stability against cyclic compression, and organic solvents immersing. The recovered sponge can be used repeatedly for 10 cycles with a separation efficiency exceeding 92% for hexadecane.[179] A prepared flat sheets cellulose membrane with hollow-fibers from solutions in 1-ethyl-3-methylimidazolium acetate showed an interesting separation process. The membrane performance for oil-water separation was evaluated, and the effect of anionic, cationic, and neutral surfactants added to emulsions with different oil contents was analyzed. Several filtration tests were applied to the membranes to investigate the effects of pH range, the oil concentration, and the addition of surfactants.[180] Bioinspired chitosan CS-based mesh has oil-fouling repellency, a high separation efficiency, and stability in a complex liquid environment. The CS coating surface in pure water and hyper-saline solutions preserves
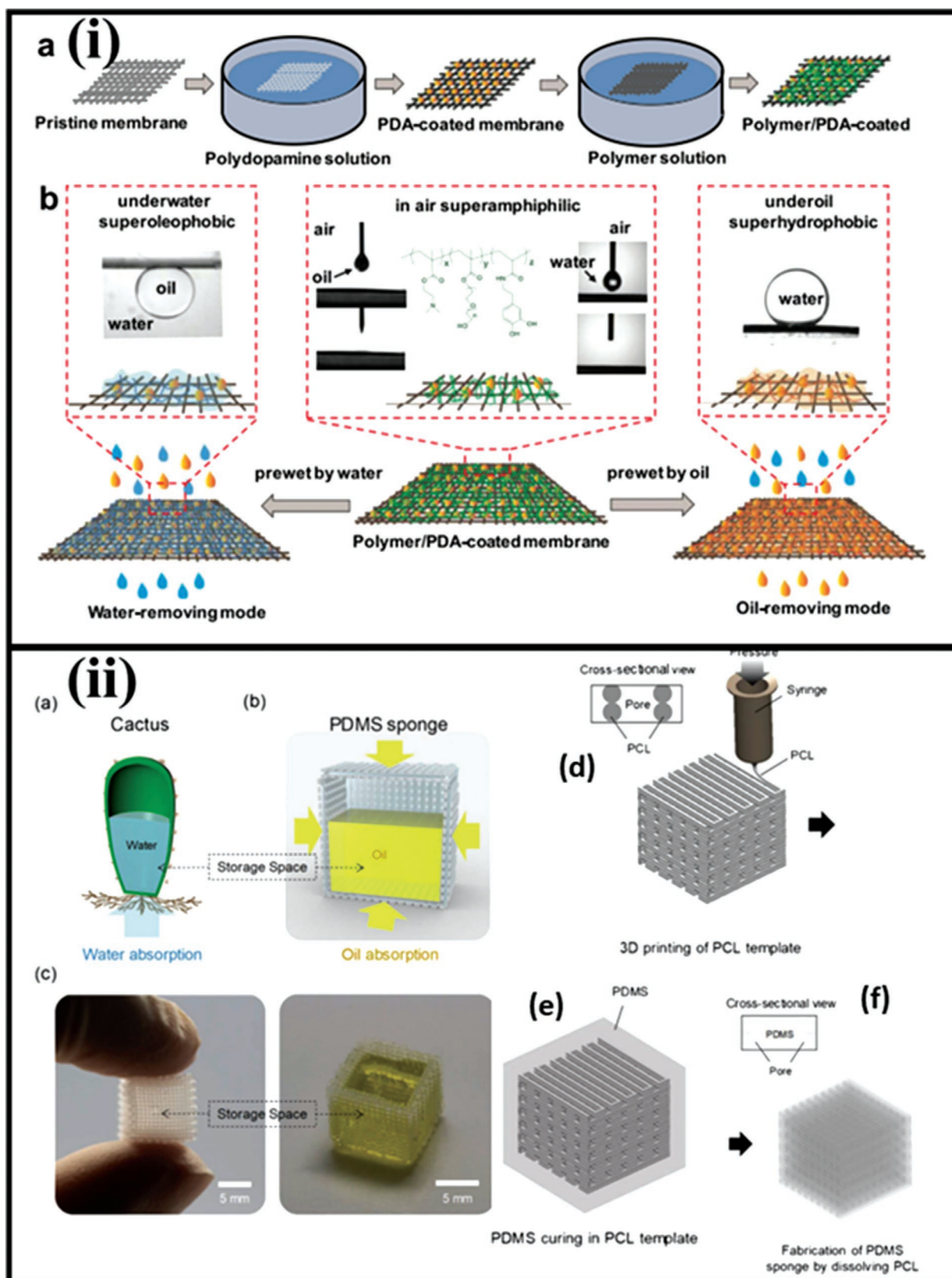
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Figure 11. (i) fabrication of polymer/PDA-coated membrane and illustration of the polymer/PDA-coated membrane. Adapted with permission.[170] Copyright 2019, Elsevier B.V. ii) Bio-inspired hollow PDMS sponge, cactus water absorption and storage space schematic, and optical image of oil absorption in hollow PDMS sponge and schematics showing PDMS sponge fabrication. Adapted with permission.[176] Copyright 2018, Elsevier B.V.
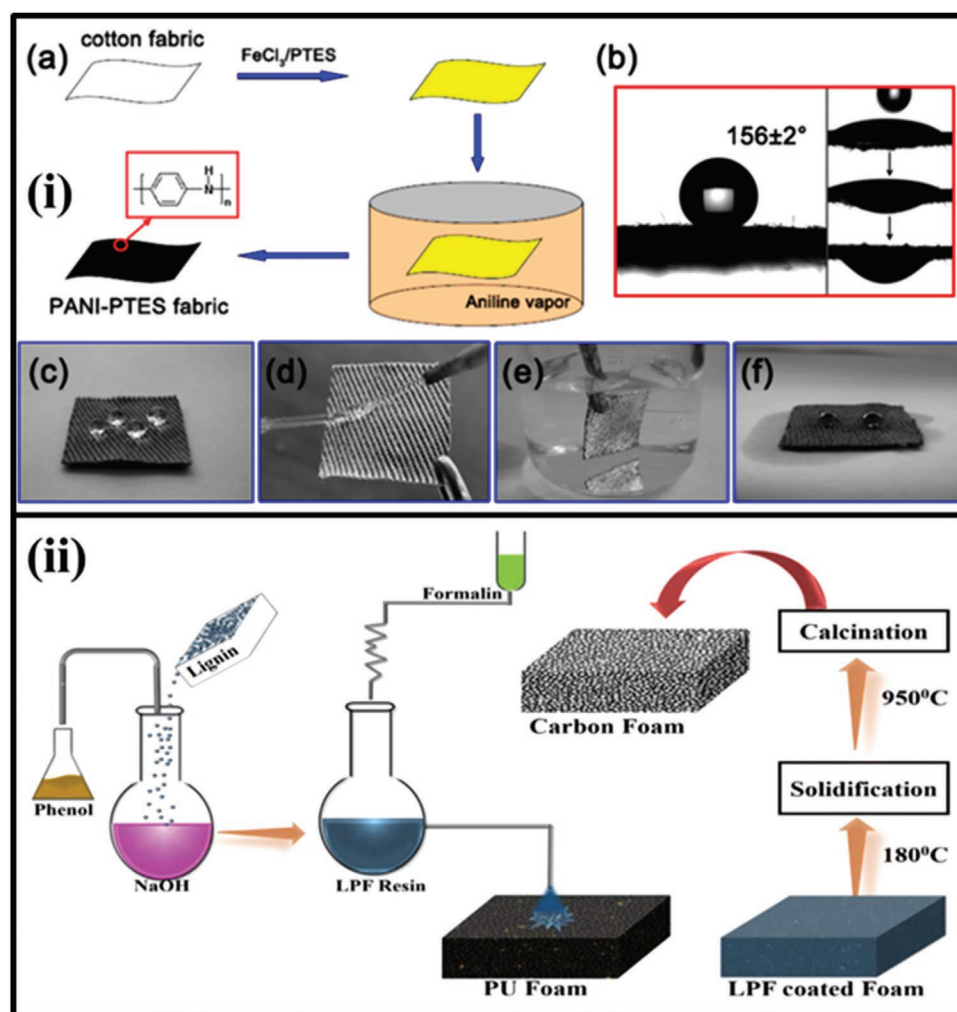
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Figure 12. i-a) Schematic of the fabrication of superhydrophobic cotton fabrics through vapor phase deposition process. b) Water droplets on the super-hydrophobic cotton fabric and hexadecane droplets spread and permeate through the fabric. c) Water droplets on the coated textile, d) a jet of water bouncing off the surface, e) the textile immersed in water by an external force, f) water droplets on the oil-contaminated textile, Adapted with permission.[182] Copyright 2013, American Chemical Society. ii) Synthesis of carbon foam using LPF resin. Adapted with permission.[183] Copyright 2021, Elsevier.

underwater superoleophobicity and low oil adhesion (<1 μN), and fully cross-linked the CS mesh with glutaraldehyde (GA) can stabilize special wettability in broad pH range environments and then be reduced by sodium borohydride to form a stable carbon–nitrogen single bond. The separation process is only driven by gravity, and the mesh can separate a range of different oil/water mixtures with a separation efficiency of more than 99% in hyper-saline and wide pH ranges.[181] In addition, the cotton fabric can be used as an effective material with separation efficiency as high as 97.8% for separating water and oil mixture (**Figure 12**i).[182] The obtained fabric preserved high separation efficiency and stable superhydrophobicity under extreme temperature, high humidity, alkaline solutions, or strongly acidic and mechanical forces.

3.10. Membrane Foamed Materials

Foamed materials are honeycomb-shaped structures with a porous network exhibiting considerable compressive

strengths and excellent efficiency for oil-water separation. Udayakumar et al.[183] reviewed in detailed foamed materials. Foamed membrane-based materials offered several advantages, such as high tailorable, highly porous 3D structure, rendering them maximum oil/-solvent absorption capacity for the targeted water treatment application. Several synthesized and function-alized foamed materials were reported based on polyurethane (PU), carbon, graphene, nickel, and other polymeric mate-rials. Qu et al.[184] developed carbon foams that used PU foam as the template for manufacturing with lignin-phenol-formal-dehyde (LPF) resin as the carbon source (Figure 12ii). Sedge grass was used to extract lignin and replaced 25 wt% of the phenol to prepare LPF resin in an alkaline medium. The carbon foams have low bulk density, unique open macropores, efficient oil absorption, and good water repellency. Carbon foams have absorption capacities ranging from 12 to 41 times their weight for various oils and organic solvents. Burning the oil inside their pores is a direct method to recycle the foams, whose absorp-tion capacity remains above 83% after 10 absorption test cycles,

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indicating good recyclability for the carbon foams.[183] The foam absorption was studied by immersing the foam in the liquid solution until the saturation limit. After that, differences were recorded in foam weights before and after immersion. The repulsion characteristics of the foam were estimated by monitoring its wettability against water and oil. The carbon foam described high oil absorbency (WCA of 0°) and, at the same time, water repellence (WCA of 149°). The carbon foams exhibited 12–41 times higher absorption capacity for high-density liquids, including oils. The developed foam showed greater than 83% oil absorption efficiency in 10 reusable cycles.[184]

3.11. Nanocomposite-Based Membranes

Nanocomposite membranes by mean of two and more than two materials mixed showed excellent antifouling and multifunctional properties such as high flux and super hydrophilic property.[185] The need to make a nanocomposite membrane was early in 1990s in the field of gas separation to overcome the Robeson upper boundary.[186] Examples of nanocomposite membranes include polymeric/ceramic membranes and hybrid polymeric materials. Carbon polymer nanocomposite membranes have recently received much attention among nanocomposite membrane materials because of their outstanding chemical, mechanical stability, and antifouling features against oil deposition/adsorption.[187] The standard blade coating and phase inversion technique were used to develop nanocomposite membranes, including polybenzimidazole (PBI), GO, and reduced GO (rGO) nanocomposite membranes for oil separation. Nanocomposite membranes were synthesized via dip-coated polydopamine (PDA), identified for their fouling resistance properties. Incorporating low weight percent GO into the PBI matrix resulted in superior oil-removal efficiency up to 99.9% and water permeability of 91.3 ± 3.4 L m^{-2} h^{-1} bar^{-1}.[188] Membranes can be used under high salinity emulsions with outstanding reusability, indicating the increased potential for treating water under harsh industrial conditions. The following section introduces smart membranes mainly designed as composite materials with innovative response stimuli.

3.12. Smart, Responsive/Switchable Membrane Materials

The next generation of membrane technology is an intelligent membrane that can sense a particular external stimulus as responsive materials.[16] External stimuli are used to transform/change the wettability properties of membrane materials from wetting (superwetting) to antiwetting (super-antiwetting) or vice versa because of responsive/switchable wettability materials behaviors.[189] **Figure 13** shows stimuli-responsive membranes for various stimuli, including pH, ions, hydro/solvent, light, temperature, electric, and pressure response. Besides, dual stimuli are exciting smart membranes separation processes.[187] Stimuli-responsive membranes have received significant attention and progress in oil/water separation.[116,117,190] For example, pH-responsive is the standard stimuli for superhydrophobicity/hydrophilicity cotton fabric was achieved when switchable wettability was achieved by coated cotton fabric via the crosslinking

reaction between pH-responsive copolymer RC, PUF NPs, and HDMI, a resulted in super wetting materials used for oil/water separation.[191] Additionally, a thermoresponsive membrane in which temperature-sensitive such as PVDF/PSMA composite membranes were fabricated with hydrophobic surfaces by phase separation method and used to separate oil–water mixtures. A range of temperatures (from −20 to 200 °C) was applied on PVDF/PSMA composite membranes for water in oil mixtures in which the oil flux increases with increasing temperature, and the change of temperature influences the pore size.[192] Furthermore, dual or multi stimuli-responsive surfaces with switchable wettability can be achieved. A reported dual pH and ammonia-vapor-responsive membrane were designed by dipcoating an electrospun polyimide (PI) membrane in decanoic acid (DA)-TiO$_2$ and SNPs. The SNP/DA-TiO$_2$/PI membrane achieved a high flux (6500 ± 100 L m^{-2} h^{-1}) and separation efficiency (>99%) during oil/water separations. In addition, high thermal and abrasion resistances are essential for applications in oil/water separation.[193] Recent investigations showed that a dual response membrane is a pH and thermo-responsive PSF membrane was manufactured by additive materials of copolymers of P(AA-NIPAm) on the membrane surfaces via a vapor-liquid phase separation method. The dual responsive membranes respond weakly to solution pH but strongly to the operation temperature.[194]

The functionalized cotton fabric membrane used perfectly alternating polydimethylsiloxane (PDMS) and poly(N,N-dimethylaminoethyl methacrylate) (PDMAEMA) blocks and displayed underoil superhydrophobicity and underwater superoleophobicity as shown in **Figure 14**a. The adaptive permeability gives functional cotton fabrics the ability to separate heavy oil-water-light oil triple mixtures.[195] A photothermal-responsive ultrathin Au nanorods/poly(N-isopropylacrylamide-co-acrylamide) hybrid with single-walled carbon nanotube (SWCNT) nanoporous membranes were fabricated, as shown in Figure 14b. The resulted membrane can separate oil-in-water nanoemulsions with a maximum flux up to 35 890 L m^2 h^{-1} bar^{-1} due to the feature of hydrophilicity, underwater oleophobicity, and nanometer pore sizes. Meanwhile, it shows super separation efficiency of 99.99% and suitable antifouling and recyclability properties.[196] Figure 14c shows an intelligent surface with responsive wettability to external stimulus for oil/water/solid interface, whereas there remain challenges of response in situ, low voltage, and stable substrate. The micro/nanoscale hierarchical-structured polyaniline (PANI) mesh resulted in superhydrophobic and underwater superoleophobic. At the same time, its superhydrophobicity can turn to hydrophilicity at 160 V and can further selectively filter water at 170 V. Furthermore, the developed membrane can work as a low underwater oil-adhesion force anti-corrosion of PANI. Overall, these features can work under a severe environment during the practical application and promise oil/water separation and microfluidic devices.[197]

4. Membrane Design

Membranes performance and high separation efficiency are the most important criteria considered in membrane design. Permeability and selectivity must be regarded as two major design
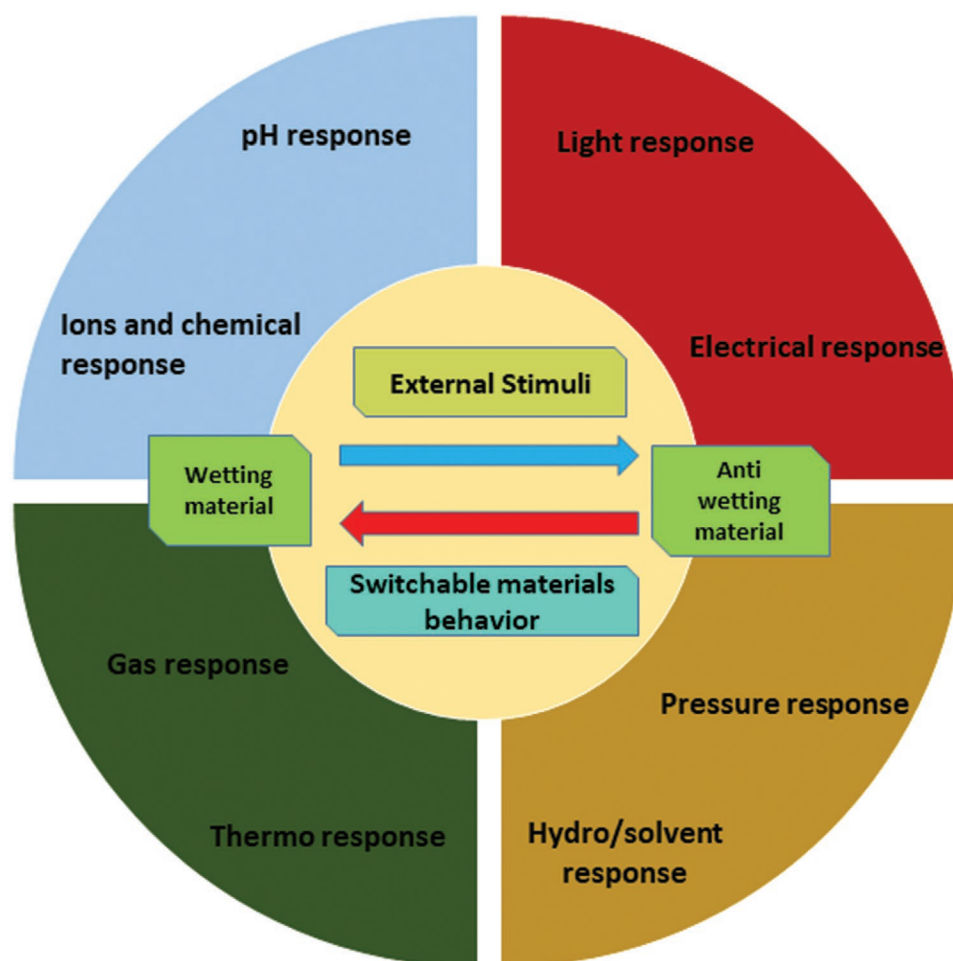
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Figure 13. Different types of external stimuli on membrane materials.

factors.[198] The trade-off relationship between permeability and selectivity can be achieved according to the direction set by Robeson. Thus, permeability determines the permeate quantity, while selectivity illustrates its quality.[199] The high separation performance is in the top right corner of the Robeson plot. Accordingly, there is a great challenge to obtain a high-performance membrane design to avoid problems and find new techniques for membrane modification.[200] Several membrane separations have a hydrophobic surface caused by the macromolecules on the surfaces, thus leading to membrane fouling.[201] To eliminate or reduce the present defects and limitations of membranes, membrane design through preparing novel materials and suitable fabrication techniques. Nanocomposite membranes have excellent properties toward desired separation efficiency. It can also be classified based on the position of membrane materials preparation, either mixed matrix nanocomposites or multilayers.

4.1. Mixed Matrix Nanocomposite Membranes

Mixed matrix nanocomposite membranes (MMNMs) are the essential generation of novel design membranes, where

nanofillers are introduced into a polymeric matrix to obtain membranes with improved properties. As a result, MMNMs have wide application, including gas–gas, liquid–liquid, and liquid–solid separations.[202] MMNMs are not limited to separation but have several applications such as sensor applications, lithium cell batteries, pervaporation (PV), proton exchange membrane fuel cells, organic solvent NF, methanol fuel cells, and water treatment. Moreover, MMNMs are heterogeneous nanofillers dispersed in a polymer matrix and shaped as flat sheets or hollow fiber. Among MMNMs preparation process, the dense region of the MMNMs layer depends on the molecular sieve of fillers inside the polymer layer.[203] Incorporating nanofillers into the polymer matrix improves membrane properties and performance, such as thermal, mechanical, chemical, catalytic, permeability, and antimicrobial properties. MMNMs are vital in advancing membrane fouling mitigation, separation efficiency, and enhancement without changing mechanical stability.[204] Nanofillers should have good quality and be free of defects to exhibit exceptional performance with good processability of polymers matrix.[205,206]

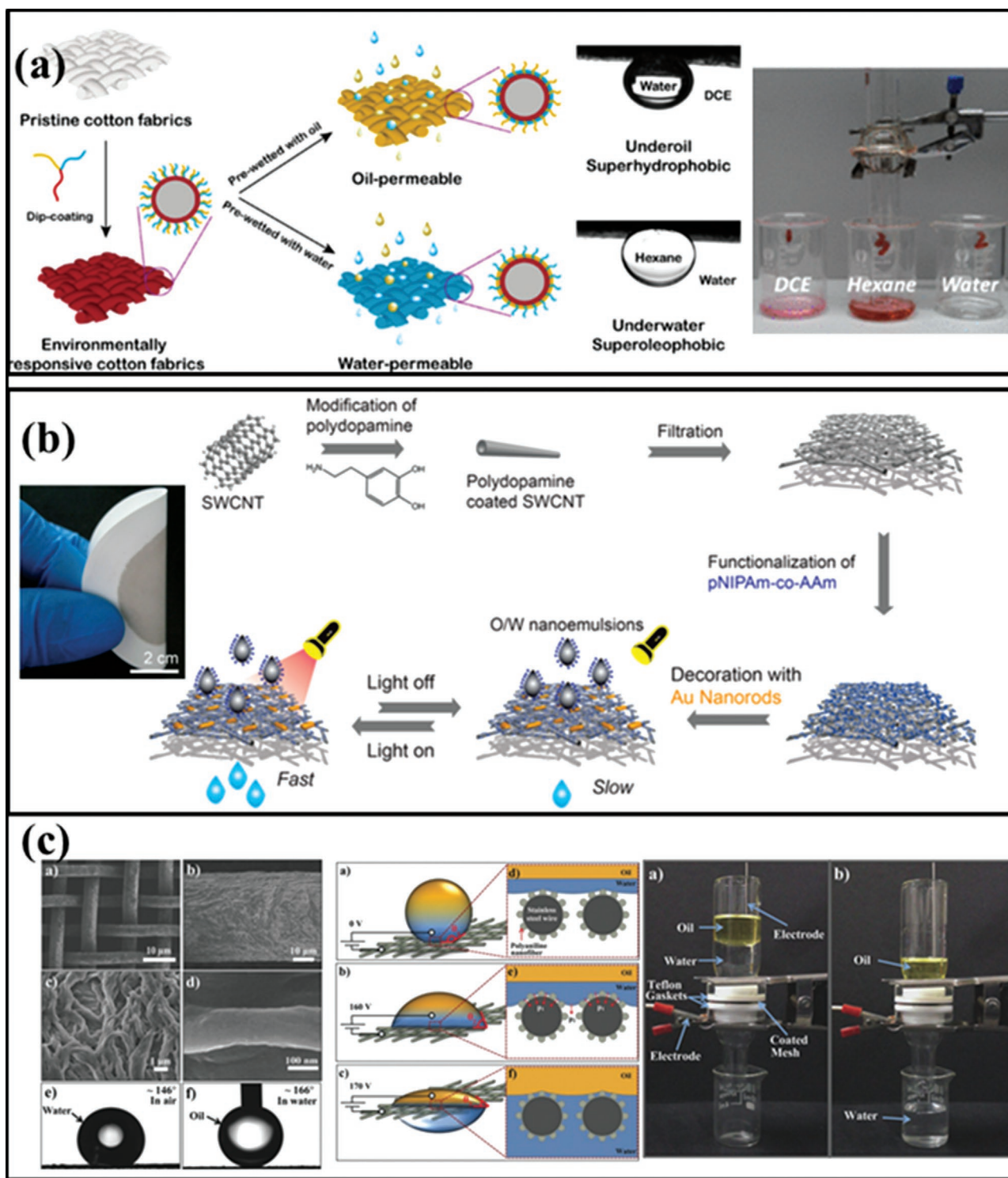Additionally, nanomaterials in the polymeric matrix fabricate inorganic–polymer composite membranes.[207]

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Figure 14. a) Schematic of an environmentally responsive cotton fabric prepared by coating a μ-PDMS-b-PDMAEMA-b-PIPSMA ABC miktoarm terpolymer and its permeability after contact with oil or water, and photographs of continuous separation of DCE water–hexane triple mixtures. Adapted with permission.[195] Copyright 2019, American Chemical Society. b) Schematic illustration of fabrication process of photothermal responsive Au nanorods/pNIPAm-co-AAm hybrid SWCNT ultrathin membranes. Adapted with permission.[196] Copyright 2015, American Chemical Society. c) Characterization of morphology and wettability of water and oil on the PANI mesh, and graphic mechanism diagrams of the electric field induced oil/water separation process based on the PANI mesh. A small amount of water in the device proved that the mesh film could not permeate the oil/water mixture, and electric field-induced oil/water separation. Adapted with permission.[197] Copyright 2016, Wiley-VCH.

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Fabrication of MMNMs can be alternatively used polymer/polymer mixed matrix from two different kinds of polymers.[208] Moreover, blending is the most common method to prepare the polymeric membrane that can acquire properties desired via incorporation. Further, additives into the polymer matrix as nanofillers are hydrophilic polymers,[209] inorganic nanoparticles,[210] and amphiphilic copolymers.[211] These are considered essential additives in polymers matrix that enhance the membrane wettability, permeability, and fouling resistance properties. Nanoparticles (NPs) improve membrane separation properties such as MOFs and offer good characteristics, including excellent compatibility with the soft polymeric matrix. For example, hybrid PVDF/NH$_2$-MIL-88B(Fe) (PVDF/NM88B) membranes have high separation performance and increased permeation flux in oil/water separation. As a polymeric membrane, PVDF was developed, and NH$_2$-MIL-88B(Fe) was added to improve self-cleaning capacity using the induced phase conversion technique. It was reported that NH$_2$-MIL-88B(Fe) offered good photo-Fenton activity and high hydrophilic amino groups.[207] One of the limitations of the blending method is that the interaction between the additives and the polymer matrix is weak, and the additives are inclined to secede from the membranes during long-term use; therefore, academic research is constantly evolving on this issue.[9]

4.2. Multilayers Nanocomposite Membranes

Membranes containing multilayers structures improve the membrane properties, such as permeability, wettability, fouling resistance, and oil rejection. Accordingly, a multilayer membrane makes it capable of acquiring sophisticated performance traits.[212] Hence, according to fabrication layers with different materials, many membranes may reach up to three layers: thin-film, dual, and Janus membranes.[213–215] Membrane layers can be manufactured via several techniques, such as layer-by-layer (LbL), which is the most common method to prepare membranes layers,[212] and the electrospinning technique for manufacturing layers which is significant for practical applications.[216,217] Multilayers can be categorized as functional materials such as nanoparticles, proteins, and DNA that are easily fabricated by hydrogen bonding or electrostatic interactions between different materials.[218]

4.2.1. Dual-Layer & Thin-Film Composite Membranes

Thin-film composite (TFC) membranes are prepared via the polymerization process, followed by two different hydrophilic materials introduced onto the membrane surface to react. TFC membranes are synthesized via dip-coating or grafting, either hydrophilic polymers or nanomaterials.[215] Thin film of aromatic polyamide (PA) formed by interfacial polymerization from TMC and MDP possesses a thin (≈100 nm) polyamide selective layer. Surface properties of the thin-film layer are different according to layer thickness, especially the dense layer, due to polymer density being not uniformly distributed.[219] TFC membranes were deposited on NF and

RO membranes widely used in water treatment. Moreover, TFC membranes with an ultrathin PA layer with the highest water flux. It is considered that most surface modification techniques are used to treat the antifouling property of membranes for oil/water separation.[220,221] **Figure 15**i shows a dual-superoleophobic membrane, prepared via one-step tailored growth of mussel-inspired PDA nanoparticles on biomass surfaces of membranes, exhibited efficiencies of oil/water separation of more than 99.98% and fluxes ranging from 4000 to 22 200 L m^{-2} h^{-1}.[222] TFC pervaporation membranes have received attention for alcohol dehydration since a high permeation flux through the thin selective layer has been achieved. LbL interfacial polymerization (IP) method was used to synthesize ultrathin multi-layer polyurea or polyamide pervaporation membrane using the hydrolyzed polyacrylonitrile (HPAN) substrate membrane Figure 15ii. The composite membrane presented a high separation factor of 844.6 with a desirable permeation flux of 1284.3 g m^{-2} h^{-1} for the pervaporation dehydration of 95 wt% ethanol aqueous solution at 80 °C (10-times higher separation factor compared with the control membrane). This method can be applied in other membrane separation processes based on solution-diffusion mechanisms, including gas separation and reverse osmosis membrane.[223]

4.2.2. Janus Membranes

Janus membranes are a particular dual membranes class with a double face and different properties. The heterogeneous structures of membranes have opposing properties on each side; however, this does not prevent them from performing both functions with outstanding performance. Janus membranes attract attention in many applications, especially for unidirectional oil/water separation. The surface properties of Janus membranes may be hydrophilic/hydrophobic and positive/negative charges.[224] Several techniques have been used to fabricate Janus membranes. The electrospinning technique synthesized nanofiber membranes with excellent percentage surface-to-volume, low surface energy, and high wettability properties with multiporous structures.[95,225] Each layer can be manufactured separately with different properties and assembled in this technique. For example, the hydrophilic PAN nanofibrous layer with hydrophobic PSF was used as a Janus membrane for oil/water separation application.[226]

Furthermore, the Janus membrane from PVDF/PDA/PEI was manufactured based on in situ mussel-inspired chemistry,[227] as shown in **Figure 16**i. Examples of these applications include the following: water collection, lossless transportation, decontamination, and on-off control.[228–230] Figure 16ii reported a Janus nanocomposite membrane that was fabricated via incorporating organosilane functionalized carbon nanotubes (f-CNT) layer onto the crosslinked hydrophilic electrospun polyvinyl alcohol (c-PVA) membrane through simple vacuum filtration for oil and water separation capability.[231] A cotton fabric with the aid of mussel-inspired chemistry and grafting of the amphiphilic octadecylamine (C$_{18}$-NH$_2$) molecules, as shown in Figure 16iii, was reported. By employing C$_{18}$-NH$_2$ molecules on the fabric surface of

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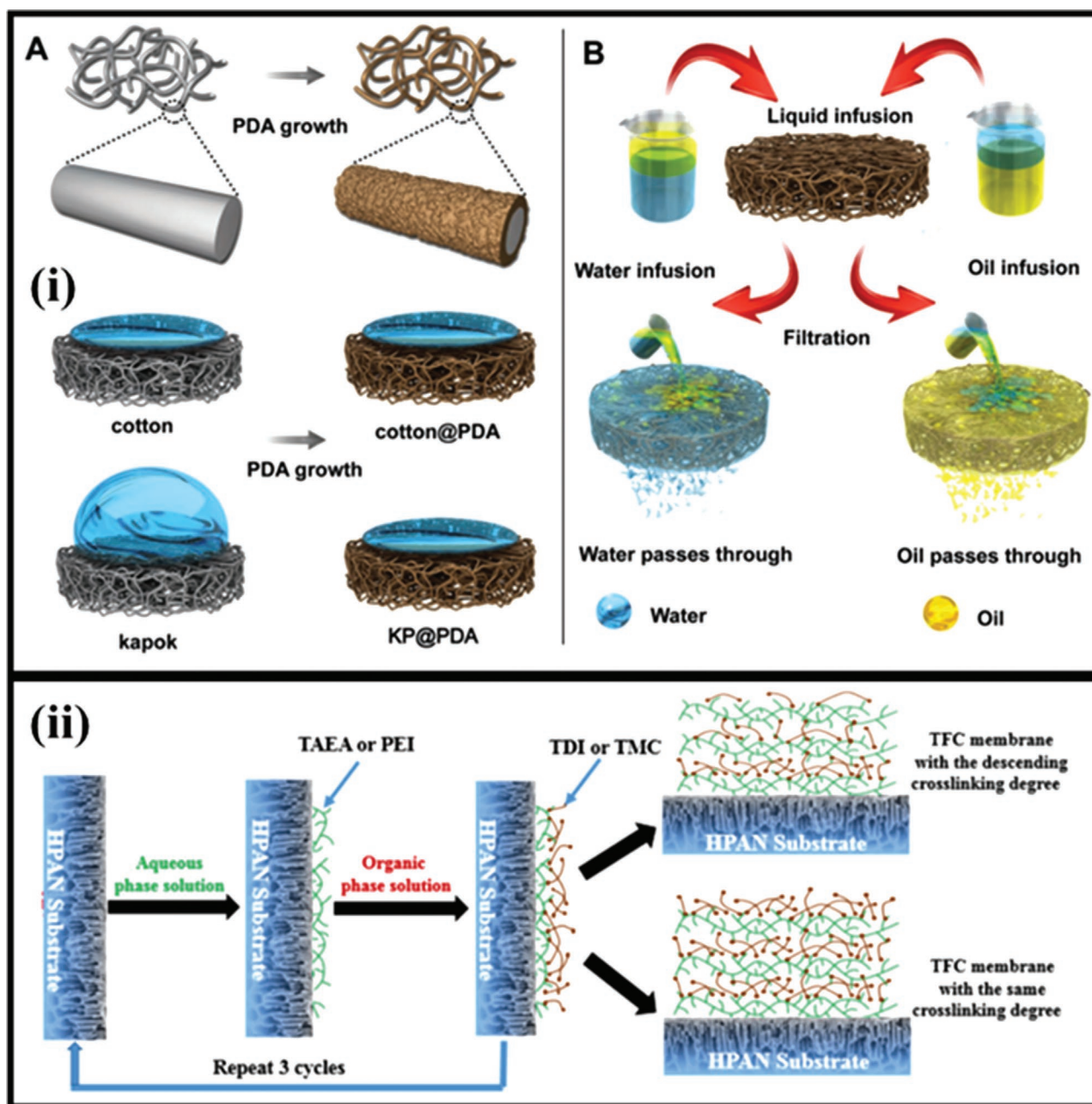INTERFACES**

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Figure 15. Schematic illustration i-A) manufacture of PDA modified cotton fabric and kapok fabric. B) Modified fabrics were applied to selectively separate oil and water. Adapted with permission.[222] Copyright 2020, Wiley-VCH. ii) The fabrication and design of TFC pervaporation membranes via LbL interfacial polymerization. Adapted with permission.[223] Copyright 2019, Elsevier B.V.

the oil side, change surface wettability from the hydrophilic character to the hydrophobicity features with WCA of 119° on one side and the other side has WCA of 0°. The surface sides have opposing properties that are hydrophobic/hydrophilic. In the case of Janus, membranes are applied for the oil/water separation, and water droplets spread on the hydrophilic side but have no penetration on the surface. In contrast, the other hydrophobic side water droplet was spherical and could penetrate the surface and block the penetration of the oil phase.[232]

5. Membranes Fabrication and Modification

5.1. Fabrication Techniques

It is essential to understand and optimize the development of membrane techniques for an efficient separation process. Separation processes are most likely through contact with the surface of the membrane; thus, the performance of the membrane is influenced by technique and further surface modification. Several methods are used for membrane fabrication and
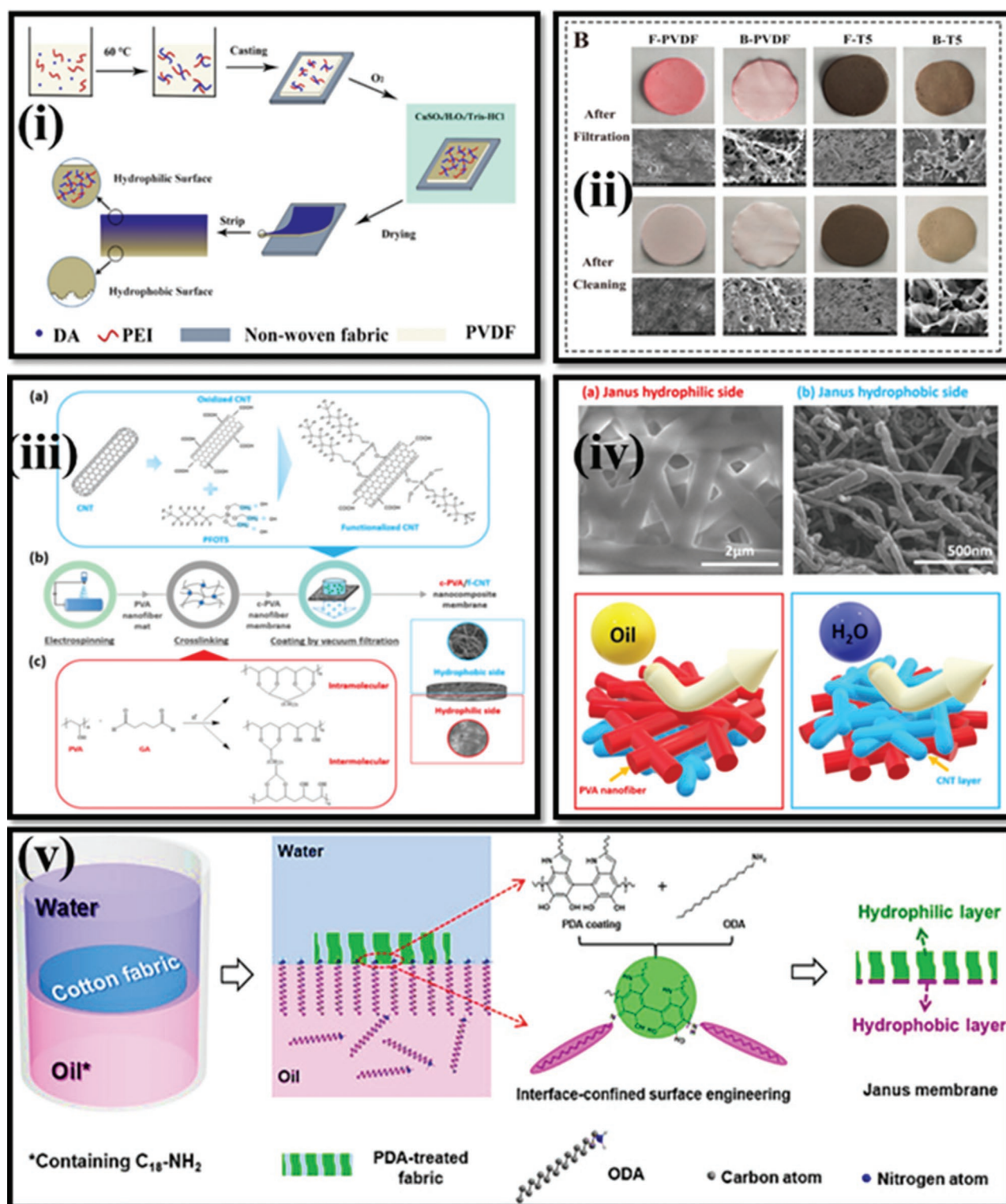
Figure 16. i) Schematic of preparation Janus fabric membrane and ii) water droplets on surfaces of Janus fabric at the oil/water interface. Adapted with permission.[227] Copyright 2018, Elsevier B.V. iii) Schematic representation of the a) PFOTS/CNT functionalization, b) the c-PVA/f-CNT nanocomposite fabrication process, and d) the crosslinking details of the c-PVA. iv) SEM Images of Janus c-PVA nanofiber membrane. Adapted with permission.[231] Copyright 2019, Elsevier B.V. v) Janus fabric membrane fabrication using liquid/liquid interface-confined surface engineering strategy and possible mechanism. Adapted with permission.[232] Copyright 2019, Elsevier B.V.

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modification.[233] The following section will describe several most common procedures, including electrospinning, phase inversion, stretching, and 3D printing. Moreover, membrane surface modification is essential to enhance membrane properties for the separation process via coating or grafting techniques.[65]

5.1.1. Electrospinning Technique

Electrospinning is a versatile technique employed to produce nanofibers membranes using mainly polymeric solution, hybrid polymers, and nanocomposite. Nanofiber membranes are applied in various fields, such as, photocatalytic membranes for wastewater treatment,[29] and oil/water separation.[25,57,234,235] Moreover, electrospun nanofibers using polymer solutions have been used since the 1930s. Recently, this technique has received considerable attention as it can fabricate nanofibers with excellent properties such as high surface-to-volume ratio, small diameter, and high porosity.[236] Several factors influence nanofibers generation using electrospinning. These factors include solution properties such as polymer concentration, solvent, viscosity, and solution conductivity.[237] The electrospinning setup should be considered, including the applied voltage, the working distance between the metallic needle and collector, and the flow rate. Other requirements include the surrounding environment, such as humidity and temperature.[237] Additional factors can control fiber formation, such as the fibers' orientation, cross-sectional configuration, fineness, and surface morphology, mainly on needle and collector setup.[238] Moreover, electrospinning can fabricate nanofibers with complex structures, including full, hollow cross-section, core–shell nanofibers with porous nanofibers, dual, multicomponent nanofibers, composite, and hybrid nanofibers.[239] It can be considered an efficient technique for oil/water separation applications due to filtration membrane and nanofibrous absorbent.[240,241]

5.1.2. Phase Inversion Technique

The phase inversion technique is commonly employed in polymeric membranes for oil/water separation.[92,185,242,243] Developed membranes via phase inversion have excellent mechanical strength, high volume ratio, and different configurations. It offers several advantages, including low cost, high production, and easy preparation and setup.[244] There are four phase inversion processes classifications: thermally induced phase separation, immersion precipitation, vapor induced phase separation evaporation, and induced phase separation. Among these, immersion precipitation is the first commercially well-known membrane fabrication technique and has various membrane morphologies.[245] Immersion precipitation is a simple way to prepare polymeric membranes. There are different ways to precipitate the polymer solution during the phase inversion process, such as immersion in a coagulant bath, cooling, vapor adsorption, and evaporation. Furthermore, this process converts the liquid phase of a polymer solution by casting onto suitable support into the solid phase by immersed into a coagulation bath.[246] Several polymers are used to fabricate membranes by

phase inversion process, which is used to synthesize MF, UF, NF, and reverse osmosis membranes, using PSF, PVDF PES, CA, and PAN membrane materials.[247]

5.1.3. Stretching Technique

The stretching technique is one of the most suitable techniques for polymeric membrane fabrication. It is a solvent-free process, where the polymer is heated above the melting point and extruded, then stretched to make it porous. This technique is preferred for highly crystalline polymers.[59] Stretching is one of the cheapest methods of fabrication membrane in which the polymer is generally stretched either uniaxial or biaxial in the direction of the extrusion to produce an extruded membrane.[248] This technique has many advantages, such as high membrane performance due to increased pore aspect ratio and thus high permeate flux.[249]

5.1.4. 3D Printing Technique

3D printing (also known as additive manufacturing) is leading innovation in many industries and research fields, including membranes for water separation, desalination, and water treatment.[250] The main advantage of 3D printing is to manufacture devices with specific shapes and functions to satisfy desired requirements of the water separation process. **Figure 17**i shows 3D printing of spherical oil skimmers with hydrogel coatings that could remove the floating oil. A superhydrophilic and underwater superoleophobic mesh (S-USM) with hydrogel coatings was fabricated using fused deposition modeling (FDM) with 3D printing of Fe/polylactic acid (PLA) composites with a separation efficiency of 85%.[251] Hydrophobic nanosilica filled PDMS ink was used to fabricate a superhydrophobic membrane with an ordered porous structure for oil/water separation using 3D printing with an efficiency reached ≈99.6%. Figure 17ii shows an environmentally friendly 3D printing membrane with superhydrophobic properties and ordered porous structure using hydrophobic nano-silica filled with polydimethylsiloxane (PDMS) ink for oil/water separation.[252] The developed 3D printed membrane integrated the superhydrophobic surface into the porous framework. The membrane is mechanically durable and superhydrophobic and can avoid the weak interface adhesion and has the advantage that the pore size can be controlled using a computer program and thus optimize both water flux and separation.

5.2. Membrane Surface Modification Techniques

The interstitial polymerization (IP) of a thin layer on the surface of the membranes by depositing two reactive monomers that occur in reaction and copolymerization between them is commonly used. Furthermore, IP exhibited excellent value because of the thin-film and surface modification, selectivity, and decreased fouling in oil/water separation. The barrier membranes layer and structural morphology can be prepared by controlling factors like the concentration of monomers,
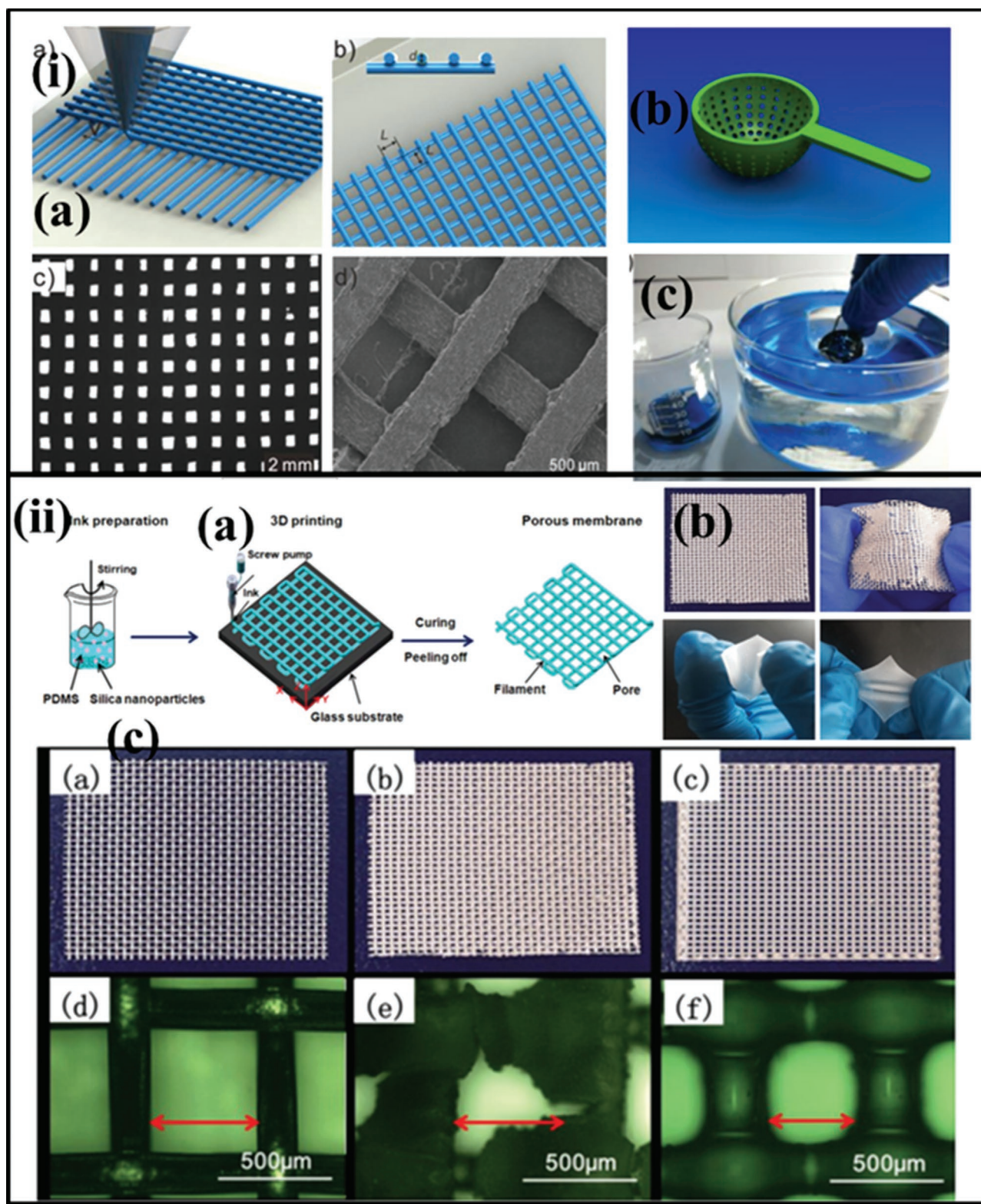
Figure 17. i) 3D printing process via FDM. a) 3D printing orthogonal mesh considering diameter, spacing, and layers. b) Floating oil removal by 3D-printed spherical oil skimmer and (c) diesel dyed in green with barrel skimmer. Adapted under the terms of the CC-BY 4.0 license.[251] Copyright 2019, The Authors, published by MDPI. ii-a) 3D printing of a porous membrane using nanosilica filled PDMS ink and images of b) pristine steel mesh and the printed membrane under bending and stretching, c) image of membrane pores and costed with superhydrophobic PDMS/silica coating. Adapted with permission.[252] Copyright 2017, Royal Society of Chemistry.

reaction time, solvent type, and post-treatment conditions. Monomers are the most common method that has been applied in IP processes such as m-phenylenediamine (MPD), tannic acid, bisphenol A (BPA), and polyvinyl amine reacting with isophthaloyl chloride or trimesoyl chloride (TMC) to form a thin active film layer.[59,253] LbL assembly is an alternative way for versatile surface modification of membranes.[212,254–256] It is a beneficial and effective technique to prepare ultra-thin films that suits composition and discipline characteristics to obtain high-performance separation membranes. The idea of this method depends on the alternating electrostatic intermixture of positive and negative on the charged surface by hydrogen bonding, electrostatic attraction, and chemical bonding, cause to the attachment of multiple layers on the membrane's surface.[202,257] The LbL method can control the nanometer-scale of film thickness during deposited layers and manage electrostatically deposited layers on the membranes' surface, leading to improved fouling resistance for membrane performance.[258,259] Membrane surface modification using the plasma technique is another technique applied in the separation process.[260,261] Plasma treatment occurs by changing the surface chemistry of membranes without changing their bulk properties. Inert or reactive gas plasma exertion onto a material surface to modify surface properties via plasma treatment. Surface treated using many molecules to react with the formed free active as neutral particles, ions, free radicals, electrons, and photos on the membrane surface.[253,262] Plasma surface activation improves the wettability surfaces of the membrane through copolymerization of the active surface of the membrane with monomers for plasma grafting.[263] Monomer concentration and grafting time conditions are the most critical factors during the polymerization to control the grafting density and length of the grafting chains.[264] Chemical vapor deposition (CVD) is another common facial surface modification technique that plays an essential role in fabricating 2D nanomaterials and thin films on the membrane surface. CVD is a chemical process used to deposit thin layers on the porous membrane at high temperatures. Reaction conditions and the choice of reactants can be optimized to control the pore size and thickness of the membrane. The CVD method offers denser and more regular surface modifications than those prepared by the liquid-phase technique.[265] CVD offers advantages such as basic coverage and the possibility of the selective deposition of the method and reactivity, volatility, and molecular thermal stability of the precursor to deposit growth material.[266] Membranes modified via CVD can achieve excellent water repellence, breathability, and oil/water separation properties.[267] **Table 2** shows the most reported membrane advances (basic materials, additives, treatment, fabrication technique, observed changes, and flux) for oil/water separation applications.

6. Membrane from Materials Development to the Device Level

Over the past 20 years, membranes materials for oil/water separation have been continually innovated. Several research efforts and attention have been paid to take advantage of the mentioned materials' characteristics in oil/water separation applications. During this interval, the development trend of the membranes varies depending on whether the modification process was either a blending or coating process and the use of different additives. Over time, several papers reported improving polysulfone-based membranes as the most common industrial polymeric membrane for oil/water separation with various modification methods. Between 2000 and 2005, Kim et al.[287] blended PSF with P(VP-AN) copolymers to prepare membranes via the phase inversion process. Furthermore, PSF/PVP membranes and PSF/P(VP-AN) membranes change solute rejection from 62.2% to 90.7% and water flux from 7.5 to 17.9×10^{-5} L m^{-2} h^{-1} Pa^{-1}. In the period between 2006 to 2010, the effect of operating conditions, transmembrane pressure, initial feed concentration, and pH on UF of PSF membranes were modified by PVP and PEG as additives of hydrophilic polymers with increasing molecular weight.[288] Yan et al.[289] reported one of the best modification methods for PVDF membranes which is inorganic materials blending, where Al_2O_3 nanoparticles are used as additives to prepare Al_2O_3/PVDF composite membranes via the phase-inversion method. This led to improving membrane hydrophilicity by decreasing of the contact angle from 83.64° to 57.42°, and consequent antifouling performance. A route to manufacture a high flux thin-film nanofibrous composite (TFNC) ultrafiltration membrane was achieved using a hydrophilic barrier layer and a nanofibrous substrate. A nanofibrous composite membrane from poly(vinyl alcohol) (PVA)/polyacrylonitrile (PAN) was fabricated via electrospinning. A PVA nanofibrous layer was employed to PAN nanofibrous substrate. This sequence was followed by remelting PVA nanofibrous layer to form a barrier PVA film via chemical crosslinking in glutaraldehyde water/acetone solution for further composite membrane development.[290] Chen et al.[291] reported the incorporation of CA casting solutions with tetraethyl orthosilicate (TEOS) by the phase inversion method to fabricate CA/TEOS membranes. The pure water flux of CA/TEOS membranes increased from 1.7 to 435.2 L m^{-2} h^{-1} and increased oil antifouling through a high oil rejection ratio (about 99.8%). Cui et al.[292] prepared PVDF porous membranes by combining inorganic SiO_2 particles /dibutyl phthalate (DBP) via TIPS method. The resulted PVDF membranes showed increasing permeability and water flux.

Between 2011 to 2015, many studies were published regarding the modification of polymeric membranes with different techniques. PSF polymer was blended and functionalized with inorganic SiO_2 nanoparticles via a phase inversion process to improve the membrane's permeate flux from 1.08 to 17.32 L m^{-2} h^{-1}, wider pores size, antifouling properties, and decrease the flux decay ratio (DR) from 98.28 to 86.55%.[293] Zhang et al.[294] prepared a superhydrophobic/superoleophilic PVDF membrane by adding ammonia into a PVDF solution and fabricating it via a phase inversion method. The membrane's characteristics were observed to change through increasing WCA from 85° to 158°. High mechanical strength was observed with a tensile strength of 2.0 MPa and elongation of 29.2% with different fluxes in the range of 700–1000 L m^{-2} h^{-1} of water-in-oil emulsions. Makaremi et al.[236] reported that PAN nanofibrous membranes prepared by PAN reinforced with halloysite nanotubes (HNTs) were successful in improving their mechanical properties (i.e., tensile strength 13.9 ± 0.9 MPa when the addition of HNTs 1% w/w),

Table 2. Recent advances of membrane for oil/water separation application.

Basic materials	Additives	Treatment	Fabrication technique	Observed changes	Flux	Refs.
PVDF	TEA	Hydrothermal	Electrospinning	Modification high hydrophobic of electrospun nanofiber PVDF membrane to be super hydrophilic with very high flux	$20\,664$ L m^{-2} h^{-1}	[268]
PVDF	PEGDA	Low-pressure plasma	Phase inversion	Membrane after modification had high separation efficiency 97% and higher flux	3773.7 L m^{-2} h^{-1}	[269]
PVDF/PPTA	PEG, LiCl	In situ blend	Phase inversion	The positive effects of different additives materials on the surface of the PVDF/PPTA membrane increased hydrophilicity and enhanced the water flux	–	[270]
PSF	MPD, TMC	Interfacial polymerization (IP)	Electrospinning	The IP reaction on the surface of nanofibers mat improves superhydrophilicity with oil rejection of 99.97%	8 to 12.21 m^3 m^{-2} day^{-1}	[271]
PSF	PVP PEI PEG PES	–	Phase inversion	Adding different polymeric additives to PS led to improve flux to the highest value by addition PVP	33.66 L m^{-2} h^{-1}	[76]
PES	SPEEK	–	Electrospinning	The membrane can be used in different types of oil/water mixtures with excellent performance and can be used in different fields	–	[272]
Nylon-6	GO, TiO$_2$	Hydrothermal	Electrospinning	Improvement nylon-6 by the photocatalytic activity of TiO$_2$(P25)/ RGO composite membrane by using electrospinning and hydrothermal techniques	–	[273]
PSF/LDH-NPs	PA	Interfacial polymerization	Casting	The blending PSf/LDH-NPs then thin film by PA layer improved porosity, surface pore diameter, thermal stability, and mechanical strengths	18.1 L m^{-2} h^{-1}	[274]
CA-PI	F-PBZ/SNP	In situ polymerization	Coaxial electrospinning	The treatment of CA-PI nanofiber membrane surface made it superhydrophobic and superoleophilic	1136 ± 50 L m^{-2} h^{-1}	[275]
GO	SiO$_2$	–	One-step vacuum filtration	The dispersion SiO$_2$ between GO layers led to expand the vertical interlayer nanochannel and enhance the water permeation oil rejection (>99%)	4550 L m^{-2} h^{-1} bar^{-1}	[276]
Cellulose/PAN		Hot roller presses at 80 °C	Electrospinning	The hydrophilic composite fiber was fabricated	–	[277]
PA/PVDF/CA	PA	Interfacial polymerization	Casting	Multilayers lead to reduce foulant dispersion on the surface membrane and high hydrophilicity	–	[278]
Nylon6	CSS	Coating		Nylon membrane had high flux after using CSS coating and excellent hydrophilicity	31847 L^{-1} m^{-2} h^{-1} bar^{-1}	[108]
Nylon6/SiO$_2$	PVAc	Coating	Electrospinning	Good mechanical strength and high porosity with high water flux and almost 99% oil rejection	4814 L^{-1} m^{-2} h^{-1} bar^{-1}	[63]
PVDF	CNW	–	Electrospinning	The PVDF nanofiber was embedded CNW on the improved surface and oil-water filtration performance with 99% oil rejection	–	[279]
PVDF	PDA, NiCo-LDH	Hydrothermal		High surface energy, antifouling performance, and high superhydrophilicity/underwater superoleophobicity by novel grass-like structured composite membrane	–	[280]
PVDF	SiO$_2$/PVDF microspheres	–	Simultaneous electrospinning and electrospraying	The hierarchical structure on the nanofiber surface developed good flexibility, superhydrophobicity/superoleophilicity and higher oil adsorption capacity with the separation efficiency 97%.	–	[281]
PVDF	PDA	Self-polymerization	TIPS	The coating enhanced permeation with 98.5% oil rejections.	2600 L m^{-2} h^{-1} bar^{-1}	[282]

Table 2. Continued.

Basic materials	Additives	Treatment	Fabrication technique	Observed changes	Flux	Refs.
PAN	HH	–	Phase inversion	PAN membrane fabricated to enhance permeate flux and a high separation efficiency	2200–3806 L m^{-2} h^{-1} bar^{-1}	[92]
PAN	PANI	–	Phase inversion	Composite PAN with PANI high hydrophilic membranes properties	350 L m^{-2} h^{-1}	[283]
PES	SiO$_2$	Dope solutions	Phase inversion	The addition of SiO$_2$ in the PES membrane led to higher water flux achieving and 85% oil rejection	73 L m^{-2} h^{-1}	[284]
PES	APTES-SiO$_2$	–	Dry-wet spinning	The permeate flux increase and oil removal from 97.23% to 99.87%	134.63 to 159.88 kg m^{-2} h^{-1}	[285]
PAA	CNTs, Pd with Pt	–	Vacuum filtration	Pd with Pt /PAA-CNTs composite membranes are formed by the catalyst layers resulting in good superhydrophilic/ underwater superoleophobic and separation efficiency approach to 99%	–	[38]
PES	PET nonwoven	Heat treatment	Electrospinning	PES electrospun nanofibrous mat supported by a PET sublayer, the nanofibrous layer was prevented from delamination and deformation		[286]

and water filtration performance (i.e., rejection ratio of 99.5% and high flux 92 L m^{-2} h^{-1}).

The reported progress work from 2016 to 2022 focused on the combination of two nanoparticles which gained significant attention due to their synergistic effect on improving the properties of polymeric membranes. Wang et al.[295] introduced a new concept of membrane manufacturing with green solvent toward polyvinylidene fluoride membrane. The developed membrane showed a high performance with pure water permeation flux and superior mechanical properties compared to that membrane using a conventional phase-separation process. Modi and Bellare[55] reported carboxylated CNT and GO nanosheets (CNTs/GO) as a CG nanohybrid was incorporated in polysulfone hollow fiber membranes that resulted in the improvement of wettability with a WCA of ≈51.4°, superior water permeation flux (487.9 ± 25.4 mL m^{-2} h^{-1} mmHg), high mechanical strength with Young's modulus of 510.2 ± 12.4 MPa, and high antifouling property (flux recovery ≈ 90.5%), as well as high oil rejection (98.7 ± 1.2%). PVDF-based membrane's oil/water separation performance has attracted much attention due to its featured properties. Recently, Wang et al.[296] coated a PVDF membrane, depositing sodium alginate suspension onto a PVDF membrane with a vacuum pump as a facile, cost-efficient, environmentally friendly, multifunctional, and green method. This is achieved by separating various oil-in-water emulsions with separation efficiencies up to 99.6%, super-oleophobicity with oil contact angles more than 150°, and flux as high as 910.4 L m^{-2} h^{-1} bar^{-1}. A study by Zhang et al.[235] used GO sheets deposited on aminated PAN (APAN) membrane fibers to enhance the rejection ratio by around (98%) and also high flux (10000 LMH). Arslan et al.[297] modified electrospun CA nanofibers (CA-NF) membrane with perfluoro alkoxysilanes (FS/CA-NF). This modification allows the formation of the superhydrophobic nanofibrous membrane with a WCA of 155° and excellent separation efficiencies.

These efforts and progress in membrane materials to overcome oil leakage and its mixture with water and other emulsions by domestic and industrial consumers. Due to their environmental pollution and a massive waste of oily water. Many efforts were made to develop a membrane with unique materials and a device level for the continuous separation process. An oleophilic array of conical needle structures for the supply of micron-sized oil droplets was used to fabricate a continuity and high throughput device. This design was inspired by the collection of similar-sized water droplets on conical cactus spines and the structures mimic cacti underwater and can capture micron-sized oil droplets.[298] Then oil droplets swelled through constant coalescence and stayed in the base of the cone. A PDMS replica (size, 0.8 × 0.8 cm^2) was employed for more extensive oil collection and drenched in water at an angle then sprayed with an oil-in-water emulsion.

Figure 18i shows device for oil/water separation using two antagonistic polymer brush-functionalized meshes. This device separated large volumes of oily wastewater with the continuous flow at high-speed. The developed device showed a high purities (≈99.9% mol/mol) and large volumes (≈1000 L) of n-hexadecane/water mixture with a continuous process at high flow rates (≈5 mL s^{-1} cm^{-2}).[299] Inspiration of membrane design can mimic water skipper float on water via its superhydrophobic legs with fibrous structures. Figure 18ii shows a fabricated device membrane from stainless-steel mesh (SSM) coated with a sol-gel technique with ZnO nanocrystals along the chain of 1H,1H,2H,2H-perfluorooctyl trichlorosilane (PFOTS) with exceptional superhydrophobicity. In addition, it exhibited permeation flux of (>22 000 L m^{-2} h^{-1}) and separation efficiency of (>98.5%) based on gravity-induced oil-water separation with more than 20 cycles in a continuous separation process.[300] The application of the oil-water separation process needs a device that can perform complete separation and operate at a range of suction pressures. Figure 18iii shows an efficient membrane with a superhydrophobic material. This filter membrane was developed by the zeolitic roughening and hydrophobic surface treatment of silica. The membranes are a mounted syringe and selective collection of oil was achieved under suction applied pressure. The membranes possess small pores of 0.7 μm; as a
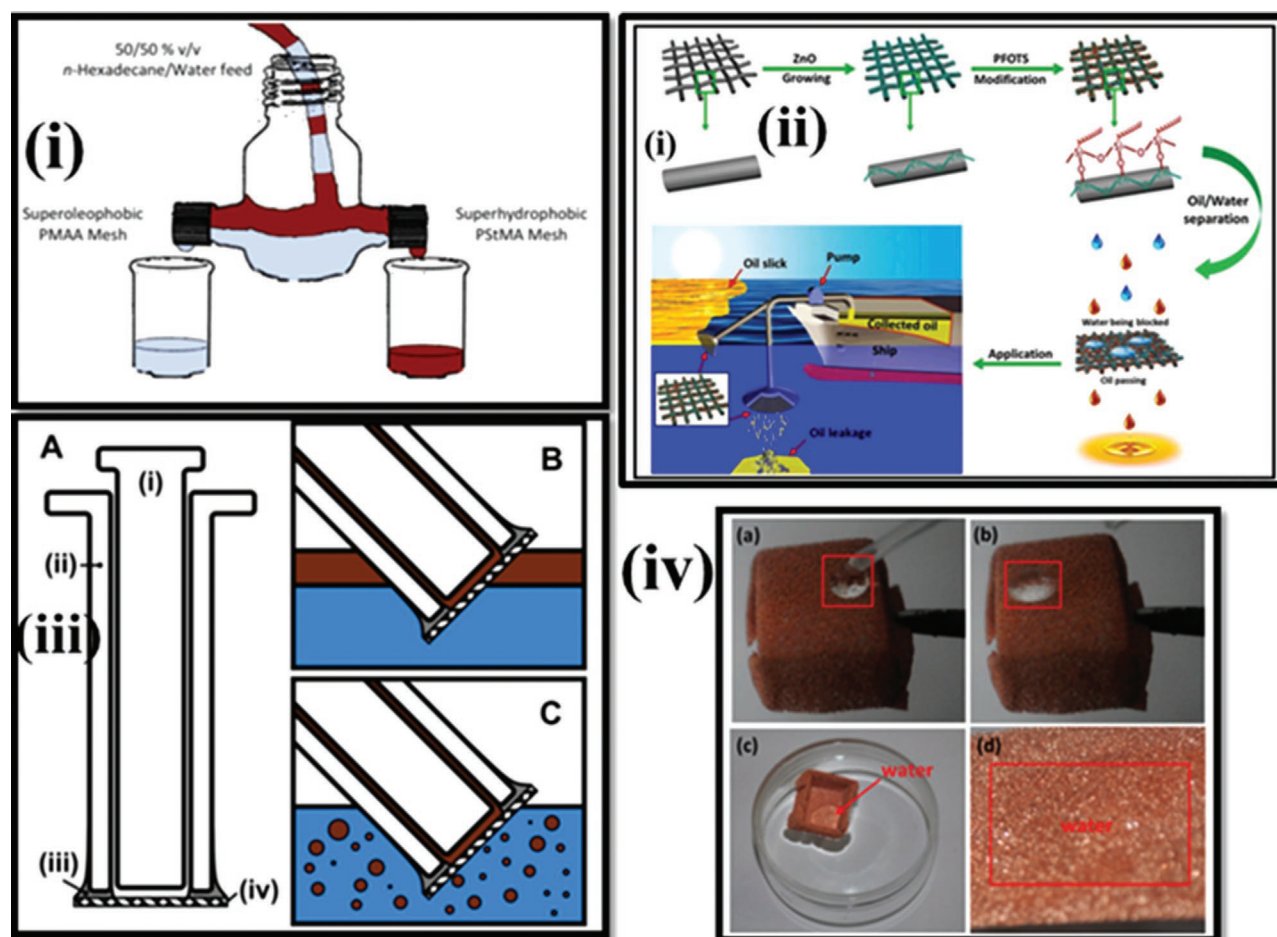
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Figure 18. i) This figure illustrates oil/water separation device using two antagonistic polymer. Adapted with permission.[299] Copyright 2015, American Chemical Society. ii) Spilled oil collection via SSM@ZnO@PFOTS membrane prepared with inspiration design to mimic water skipper float on water. Adapted with permission.[300] Copyright 2019, Elsevier B.V. iii) Figure shows device through the targeted treatment of silica meshes. The inset panel (A) shows device components of (i) glass syringe plunger, (ii) shortened glass syringe barrel, (iii) silicone adhesive, and (iv) superhydrophobic silica membrane. Panel (B) shows the device collects a range of oils from water and pannel (C) dispersed oil droplets. Adapted under the terms of the CC-BY 3.0 license.[301] Copyright 2015, The Authors, published by Taylor & Francis. iv) Shows a functionally integrated device for oil/water separation, (a) a drop of water placed on the outer surface of the device. b) Upon tilting the device, the drop of water slid off quickly. c) A drop of water is placed inside the device. d) Water drop placed on the surface of the untreated copper foam. Adapted with permission.[302] Copyright 2015, Royal Society of Chemistry.

result, these devices can operate at a range of suction pressures and perform a complete separation process and used in a real application such as oil spill clean up and industrial filters.[301] Figure 18iv shows a functionally integrated device with a multifunction of the oil-containment boom, oil-sorption material, and water/oil separating film. During the simple immersion process in an ethanol solution of stearic acid, superhydrophobicity, surface roughness, and low-surface-energy coatings were achieved simultaneously. The as-prepared functionally integrated device could float on water and act as an oil containment boom, an efficient oil-absorbing property, which was attributed to the capillary effect caused by micrometer-sized pore structures and used as oil-sorption materials; which was suitable for water/oil-separating film. This device can be integrated and functionalized for oil collection, absorption, and water/oil separation into a single device. Interestingly, these functions are independent, thus reducing the cost of energy consumption.[302]

7. Conclusion

Membrane technology has been substantiated as an outstanding solution for treating the world-class oily-water treatment problem, which involves a small scale and promising results on a large scale. In contrast to traditional membranes for oil-water separation, membranes surface with superwetting has several features, including a high permeability rate, high separation efficiency, and low energy requirements. Recent research progress led to finding several techniques to improve the performance of conventional materials in oil/water separation applications. Yet, there are still many challenges in developing oil/water separation membranes. Membrane materials including metallic, organic, inorganic, MOFs, hybrid membranes, nanogel, natural, foamed, innovative responsive/switchable, and additives materials were reported. Most important for separating oil/water emulsion is high membrane performance design to avoid fouling limitations and novel membrane modification techniques. On the

other hand, it is possible to fabricate the desired membrane surface with decreasing membrane fouling through enhancement of membrane properties such as wettability, permeability, antifouling, and mechanical properties. The membrane fabrication can be improved via modern technologies such as electrospinning, 3D printing, and phase inversion.

8. Disclaimer

The research presented was not performed or funded by EPA and was not subject to EPA's quality system requirements. The views expressed in this article are those of the author(s) and do not necessarily represent the views or the policies of the U.S. Environmental Protection Agency.

Conflict of Interest

The authors declare no conflict of interest.

Keywords

antifouling, membrane design, membrane materials, membrane technologies, oil/water separation

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