A critical review of membrane modification techniques for fouling and biofouling control in pressure-driven membrane processes

A critical review of membrane modification techniques for fouling and biofouling control in pressure-driven membrane processes

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Abstract

The demand for new water resources has been increasing worldwide due to the growing global population and industrialization. Membranes play a central role in water purification processes with continuous technology improvements, new uses, and cost reductions. Phase inversion is the most used technique for preparing polymeric membranes. However, most commercial membranes are prepared from hydrophobic materials, which make them more susceptible to suffer the adsorption or deposition of molecules over their surface or inside their pores. This phenomenon, commonly termed as fouling, is one of the major operational problems in membrane processes. This review covers different membranes modification techniques to enhance permeability and to reduce fouling and the accumulation of microorganisms on membrane surface. The physicochemical properties of membrane surface can be tuned by grafting or coating to introduce functionalities like hydrophilic moieties or charged groups. Blending hydrophilic additives into the casting solution is another approach to increase membrane hydrophilicity and to improve water filtration performance. This review summarises the variety of porous materials that have been used to introduce inorganic nanoparticles into the casting solution. Porous carriers are used to introduce antimicrobial metals by preserving nanoparticle stability. Additionally, organic compounds, especially dendritic structures, have attracted considerable interest due to their highly branched structure, and their large number of terminal functional groups, which can also be used to enhance membrane properties. This work reviews the modification and functionalization techniques recently proposed to improve permeability and durability of the membranes used in pressure-driven separations.

Keywords: Fouling; Biofouling; Membrane modification; Grafting; Surface coating; Hydrophilic additives

Abbreviations

BSA	Bovine serum albumin
CA	Cellulose acetate
CF6	Fluorinated carbon chains
EC	Epoxy-containing coumarin moieties
EIPS	Evaporation-induced phase separation
EO	Ethylene oxide
EPS	Extracellular polymeric substance
GO	Graphene oxide
HBPs	Hyperbranched polymers
HNTs	Halloysite nanotubes
MF	Microfiltration
MMMs	Mixed matrix membranes
MOFs	Metal–organic frameworks
MSPs	Mesoporous silica particles
NF	Nanofiltration
NIPS	Non-solvent-induced phase separation
NPs	Nanoparticles
PA	Polyamides
PAA	Poly(acrylic acid)
PAMAM	Poly(amido amine)
PAN	Polyacrylonitrile
PD	Polydopamine
PEA	Poly(ether amine)
PEG	Poly(ethylene glycol)
PEI	Poly(ether imide)
PES	Poly(ethersulfone)
PET	Poly(ethylene terephthalate)
PO	Propylene oxide
PP	Polypropylene
PsU	Polysulfone
PVA	Poly(vinyl alcohol)
PVDF	Poly(vinylidene fluoride)
PVP	Poly(vinyl pyrrolidone)
QS	Quorum sensing
RO	Reverse osmosis
TIPS	Thermally induced phase separation
UF	Ultrafiltration
UV	Ultraviolet
VIPS	Vapour-induced phase separation

DOI: 10.1007/s41204-020-00077-x

Water resources and water scarcity

The demand for new water resources has been increasing worldwide due to the global growth population, socio-economic development, and industrialization. In 2050, it is expected that world population rises by almost 40%, thereby increasing the demand for safe, clean, and drinkable water [1]. Water resources have been assumed as abundant. However, only 2.5% to global water resources are freshwater, the rest being saline. From this 2.5%, 70% is frozen in polar regions and the rest is in aquifers of difficult access. Therefore, less than 1% of total water resources are directly available for human use. Unfortunately, most available water shows evidence of anthropogenic contamination by effluents from domestic, agricultural, and industrial activities [2, 3]. Furthermore, the uneven distribution of water over the globe leads to severe water scarcity in certain regions. According to the United Nations World Water Development report, within the next 30 years, there will be 3.9 billion people living in "water-scarce" areas. Moreover, the World Health Organization estimates that at least 1.1 billion people are lacking access to clean drinking water [4, 5]. Based on this scenario, there is a need to protect the existing water resources around the world as well as design proper strategies to reduce and reuse water to preserve the environment and support new generations.

Membrane technology

Membrane technology contributes by almost > 50% of the total world water treatment volume. Water purification involves the removal of pollutants such as organic, chemical, and biological contaminants, as well as the suspended solids present in water to obtain sufficiently clean and satisfactory sensory water [6, 7]. In recent years, the development of membrane technology has been widely applied in desalination and wastewater treatment in areas such as manufacturing, biotechnology and food processing industries favoured by due to its operational simplicity and cost-efficiency [8–12]. A membrane can be defined as "a selective physical barrier that retains unwanted materials on the surface and allows certain compounds to pass through, depending on their physical and chemical properties, when a driving force is applied across the membrane" [13]. According to membrane configuration, applied pressure and pore size, membrane processes are often classified into four different categories, which are represented in Fig. 1. Moreover, the main advantages and drawbacks of the membrane operation are summarised in Table 1.

– Microfiltration (MF) is a process in which membranes have relatively large pores, generally in the 10–0.1 µm range and a molecular weight cut-off (MWCO) greater than 100,000 Da. This process requires a relatively low operating pressure, typically in the 1–4 bar range. MF membranes are useful for removing large suspended solids such as colloids, particles, and some bacterial species from solutions [14]. MF has used in a number of industries such as food industry (i.e. wine, juice and beer clarification) [15], dairy industry (reducing microbial load of milk, casein or whey fractionation) [16], metal industry (oil/water emulsion separation) [17], and industrial processes including wastewater treatment (clarification of fermentation broths, wastewater treatment, pretreatment upstream of NF or RO to reduce fouling potential) [18, 19].

– Ultrafiltration (UF) membranes with a pore size range between 0.1 and 0.01 µm and MWCO of approximately 1000–100,000 Da. This technology separates relatively large molecules such as proteins, polysaccharides, humic material and all microbiological species, including viruses. The operating pressure is usually in the 1–8 bar range [20]. UF is implemented in the dairy industry (milk protein concentration) [21], biotech or pharmaceutical fields (endotoxin removal, antibiotic production, blood plasma processing) [22], food and beverage industry (fruit juice concentration, plant extract processing) [23], industrial process and wastewater (oil removal in wastewater treatment, dissolved natural organic matter) and can also be used as pretreatment of seawater prior to reverse osmosis [24].

– Nanofiltration (NF) is the intermediate membrane process between UF and RO. These membranes have a pore size in the 0.01–0.001 µm range which is equivalent to a MWCO of 200–1000 Da. This separation process usually operates at pressures in the 5–30 bar range [25]. NF can remove low molecular weight molecules like sugars, amino acids, and divalent ions while some monovalent ions are still able to permeate through the membrane. NF is used in many

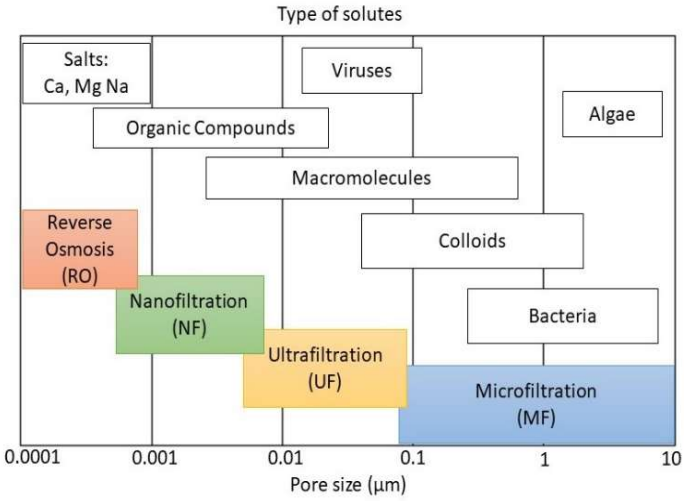

Figure 1. Different types of pressure-driven membrane processes. Adapted from Ref. [31].

industries such as dairy (concentration and demineralization of lactose) food and beverage (maple

DOI: 10.1007/s41204-020-00077-x

syrup concentration) [26], textile and dyes (dye concentration) [27].

– Reverse osmosis (RO) is the most effective technology for removing inorganic contaminants, dissolved salts, and chemical constituents from water. Membranes are dense with a pore size lower than 1 nm and molecular weight cut-off about 100 Da. This process requires higher operating pressure, usually in the 20–65 bar range [28]. RO is used in various applications including selective separation, purification, concentration, and desalination processes. In food industry, RO is applied for concentration of fruits and vegetables juices, dealcoholisation of alcoholic beverage, and preconcentration of milk or whey, and to purify drinking water [29]. RO is used in industrial process (wastewater treatment, desalination of seawater), automotive manufacturing (treatment and recycle of water used for cleaning and painting), or the treatment of landfill leachates (removal of salts and heavy metals prior to discharge) [30].

Membrane preparation methods

Membranes can be produced using several different techniques, including phase inversion, controlled stretching, interfacial polymerization, melt extrusion or electrospinning, depending of the desired membrane morphology [39, 40]. Among these techniques, phase inversion is the most used to prepare both asymmetric and symmetric polymeric membranes. Generally, phase inversion is a demixing process whereby the homogeneous polymer solution is transformed, in a controlled manner, from liquid to solid state. Additionally, the demixing process can be defined by the exchange rate between solvent and non-solvent during precipitation [41]. Therefore, phase inversion can be achieved in several ways, namely:

– Non-solvent-induced phase separation (NIPS) or immersion precipitation is a process in which a polymer is dissolved in a proper solvent until a homogeneous solution is obtained. After that, it is cast on a suitable support followed by immersion in a non-solvent coagulation bath, typically water. During this process solvent/non-solvent exchange takes place and polymer precipitation occurs. Finally, a solid polymer film is obtained with asymmetric structure [42, 43]. This method requires complex control of solvent exchange rate, which is strongly affected by dope composition, choice of solvent, coagulation bath composition, temperature, and evaporation time [6, 44]. The most important parameters affecting the structure of the resulting membrane are, the composition of the casting solution (i.e. polymer concentration, solvent and non-solvent selection, polymeric additives), the composition and temperature of coagulation bath, exposure time (i.e. evaporation rate), and air humidity and temperature [45, 46]. An exhaustive review of the different factors for controlling membrane final morphology can be found elsewhere [47]. These parameters affect the kinetics and thermodynamics of membrane formation process. Consequently, both symmetric and asymmetric membrane can be prepared by NIPS depending on the type of demixing (delayed or instantaneous): Symmetric membranes with a well-defined pore size along their entire thickness, or asymmetric membranes with a dense skin top layer supported by a porous sublayer [48]. NIPS is the most widely used method for membrane preparation in research and industry.

Table 1: Main advantages and drawbacks of pressure-driven membrane processes.

Process	Advantages	Disadvantages	References
Microfiltration (MF)	Low pressure Low energy consumption Relatively cheap, few manual actions required	Insufficient quality of treated wastewater Only suspended matter and bacteria are removed Sensitive to oxidants	[32,33]
Ultrafiltration (UF)	Cost-effective operation due to low energy consumption Processes are relatively simple to scale up Excellent chemical and thermal stability with service life up to several years	Only suspended matter, bacteria and some viruses are removed.	[34,35]
Nanofiltration (NF)	High removal efficiency Separation of organic compounds of low molecular weight and divalent ions from monovalent salts Easy operation Higher water permeability and lower energy consumptions compared to RO	Relatively high cost Limited retention for salts and monovalent ions Membranes are sensitive to free chlorine	[36,37]
Reverse osmosis (RO)	Removal of all mineral salts High efficiency Simple operation Know-how with large plants	High pressure requirements High capital and operation costs Pretreatment required in some cases	[29,38]

DOI: 10.1007/s41204-020-00077-x

– Thermally induced phase separation (TIPS). In this process, a polymer solution is prepared at elevated temperature using an appropriate solvent with a high boiling point. Then, the hot homogeneous solution is cast into the desired shape, followed by cooling to induce phase separation and membrane formation. After polymer solidification, the remaining diluent is removed typically by solvent extraction, leaving a highly porous membrane. The solvent should have a high boiling point, low molecular weight, and low volatility [31, 49]. In this method, most polymers are dissolved at a temperature higher than their melting point. Therefore, if a crystalline polymer is used to produce membranes, at high polymer fractions, (> 30%), the polymer tends to crystallize during the phase inversion process, producing high strength membranes, with a typical sphere-like porous structure. The size of the spherulites is highly dependent on fabrication conditions including cooling rate, temperature gradient, and use of additives. Conversely, at relatively lower polymer fractions, membranes exhibit a porous honeycomb-like cellular morphology. Controlling the liquid–liquid demixing kinetics is critical to finetuning the final membrane morphology [31, 50]

– Evaporation-induced phase separation (EIPS). It is one of the simplest methods for membrane preparation by phase inversion. In this process, a polymer is dissolved in a solvent or mixture of volatile solvents and a less volatile non-solvent. When the polymer solution is cast on a suitable support, the solvent evaporates resulting in demixing. Once polymer precipitation occurs, a highly porous film is formed [42]. The morphology of the casted films can be controlled by using solvent with different boiling points. By this method, dense anisotropic membranes are generally obtained. However, the final membrane morphology depends on several factors such as polymer and non-solvent concentration, casting solution thickness, relative humidity, the temperature on the final film and the rate of evaporation [51]. It was demonstrated that, at low concentration of non-solvent, delayed phase separation may lead to the formation of denser membranes. In addition, at higher drying rates, skinners membranes can be formed due to the high evaporation rate of solvent and non-solvent [52]. Polymer concentration plays an important role in membrane morphology, changing from porous anisotropic structure, at low polymer contents, to dense symmetric structure, at higher concentration [48].

– Vapour-induced phase separation (VIPS). This method is used to prepare highly porous membranes. Once the polymer is dissolved in a specific solvent, the casting solution is exposed to an atmosphere containing a non-solvent vapour, usually water, in a vapour chamber. Upon vapour absorption, precipitation occurs, yielding the membrane structure [53]. Mass transfer is slower, resulting in a highly porous surface because the polymer concentration near surface is lower than the initial polymer concentration. VIPS can be used to avoid the formation of macrovoids [54]. Phase inversion can be completed by immersion in a nonsolvent coagulation bath. In this process, different values or air humidity and temperature, or different exposure times, lead to membranes with different morphologies. Membrane surface changes from a very porous surface to a denser one depending on exposure time and air humidity. It was found that the average pore size increased with relative humidity [48, 55].

Taking immersion precipitation as an example, there are three components involved in phase separation, namely polymer, solvent, and non-solvent. Therefore, a ternary phase diagram can be used to describe the thermodynamic behaviour as shown in Fig. 2. The pure components are represented at the edges of the triangle while any points inside the diagram represent a mixture of the three components. The ternary phase diagram of a polymer–solvent–non solvent system is formed by a single-phase or homogeneous region and a two-phase or unstable region. In the first one, the three components are fully miscible whereas in the unstable region the solution separates in two phases, the polymer-rich phase, which forms the matrix of the membranes, and the polymer-lean phase that forms membrane pores. The binodal curve is the boundary between both regions [56]. The precipitation process is represented as a line through the phase diagram starting from point A and ending with point D. Point A represents the homogeneous casting solution made up of solvent and polymer, which is immersed in the coagulation bath (non-solvent). As the solvent is removed from the polymer solution the composition of polymer moves along A–B–C. At point B a transition takes place from the one-phase region to the two-phase-region, a polymer-rich phase and a polymer-poor phase appear at the upper and lower boundary of the de-mixing gap, respectively. The spinodal curve delimits the metastable region of the miscibility gap. At point C the polymer concentration in the polymer-rich phase

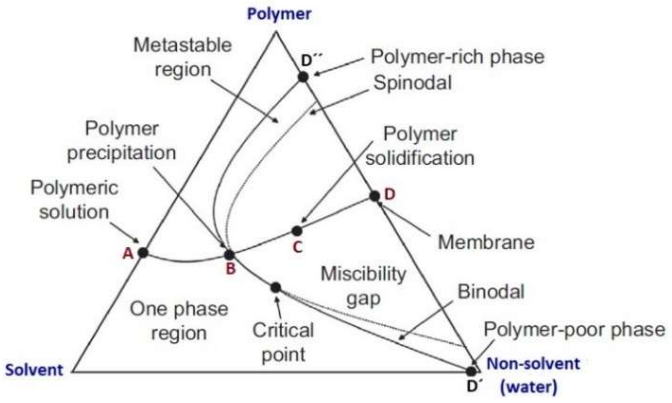

Figure 2. Schematic representation of three-component phase diagram during phase inversion. Adapted from Ref. [48].

DOI: 10.1007/s41204-020-00077-x

will be high enough to be considered solid. Further exchanges of solvent and nonsolvent lead to the final membrane composition, point D [41, 48, 57].

Effect of additives in membrane preparation

The incorporation of some additives into the casting solution plays an important role during the membrane preparation because they can affect the solution demixing process during phase inversion. Commonly used additives can be classified into the following categories: [58].

– Polymer additives such as polyvinylpyrrolidone (PVP) or polyethylene glycol (PEG).

– Low-molecular-weight chemicals including salts (LiCl), inorganic acids (acetic acid and phosphoric acid), organic acids (propionic acid).

– Weak co-solvents like ethanol, propanol, and acetone.

– Weak non-solvents glycerol or ethylene glycol.

– Strong non-solvents such as water.

During immersion precipitation, either instantaneous or delayed demixing occurs and different membrane structures can be obtained, depending on the rate of polymer precipitation in the non-solvent bath (Fig. 3). If the polymer precipitates quickly in the non-solvent bath, instantaneous demixing takes place, resulting in membranes with thin skin layer and finger-like morphology sublayer (Fig. 3a) [59]. However, if the composition path does not cross the binodal curve a delayed demixing occurs. In this case, the mem-brane formation is slow, and the separation takes a longer time. These membranes show a relatively dense top layer and a characteristic sponge-like structure (Fig. 3b) [47, 55]. Additives significantly affect membrane structure. They can accelerate coagulation exchange, enhance pore formation, improve pore interconnectivity, enhance hydrophilicity, increase viscosity, or suppress macrovoid formation [60]. Mansourizadeh and Ismail prepared poly(vinylidene fluoride) (PVDF) membranes using LiCl as the nonsolvent pore-forming additive. They demonstrated that the concentration of LiCl in the dope solution affects membrane morphology and CO_2 absorption performance [61]. At low LiCl concentration (2%), highly porous membranes with a large finger-like porous structure were obtained due to the increased phase separation rate, which significantly improved the CO_2 flux. However, at higher concentration (4%), membranes with a sponge-like structure were found owing to the increased solution viscosity, in this case results showed an approximate 30% CO_2 flux reduction that can be related to partial wetting of the membranes pores due its sponge-like structure. Lan and Wang studied the influence of glycerol, butanol and PEG-400 on the morphology and performance of PES membranes when used during membrane fabrication [62]. They demonstrated that when butanol concentration increased from 8 to 15%, the membrane structure changed from finger-like morphology to sponge-like structure. The same effect was observed increasing glycerol concentration from 2 to 6%. At higher concentration, membrane porosity significantly decreased, which confirms that glycerol contributes to produce membranes with more compact and dense structure, the membrane flux also showing a decreasing trend. Finally, at higher concentration (PEG 8%), it was observed that the viscosity of the solution increased, which delayed phase separation, thereby inhibiting finger-like pore structure. In addition, Deshmukh et al. demonstrated that the introduction of PVP as a non-solvent in the PVDF polymer dope solution, resulted in membranes with a slight decrease in mean pore size and

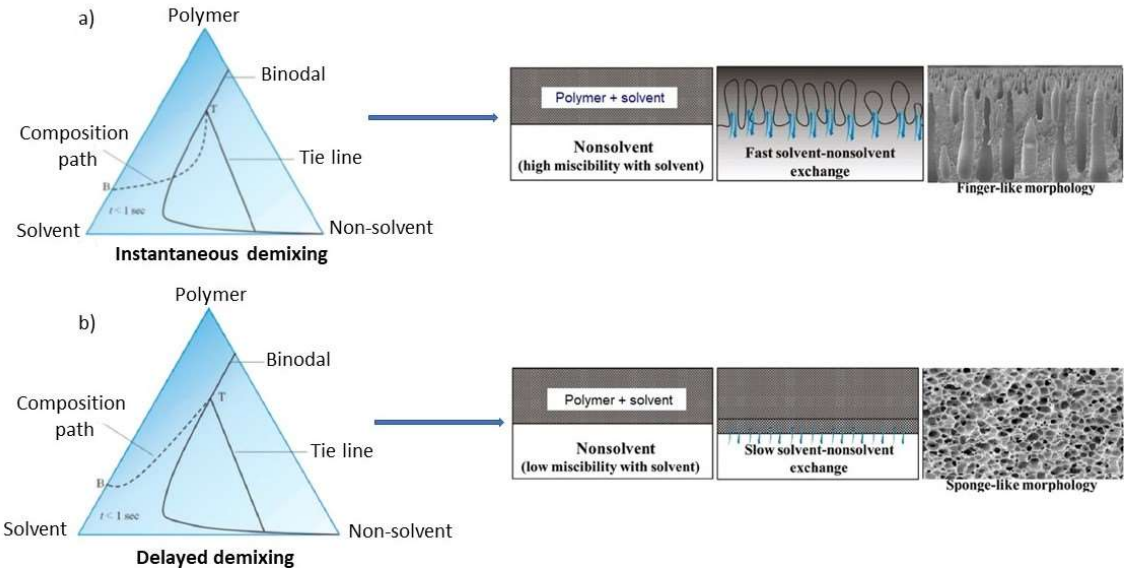

Figure 3. Different membrane morphologies depending on the rate of solvent-nonsolvent exchange. Adapted from Refs. [47, 55].

DOI: 10.1007/s41204-020-00077-x

a drastic increase in effective porosity as compared to those without addition of PVP [63]. Moreover, it was found that glycerol and phosphoric acid as additives in PVDF dope resulted in larger pore size and higher MWCO, thereby improving membrane permeability and CO_2 absorption [64].

Membrane fouling

The most common polymeric materials used for preparing MF, UF NF and reverse osmosis membranes are: polyethersulfone (PES), polysulfone (PsU), cellulose acetate (CA), polyacrylonitrile (PAN), PVDF and polyamides (PA) due to their high chemical, thermal and mechanical stability [47, 65]. However, one disadvantage of these polymers is that they suffer from the deposition of some substances on their surface or inside their porous structure leading to a decrease in permeate flux. This phenomenon is referred to as fouling, because of which expensive cleaning and periodic regeneration procedures are necessary to prevent membrane loss of performance and to mitigate the need for higher pressure and energy consumption, which would be required to maintain constant flux. Fouling also reduces the useful service life of membranes [66, 67]. According to the International Union of pure and Applied Chemistry, fouling can be defined as follows: "The process that results in a decrease in performance of a membrane, caused by the deposition of suspended or dissolved solids on the external membrane surface, on the membrane pores, or within the membrane pores" [68]. Therefore, there are four different types of membrane fouling:

– Inorganic fouling: also known as a scaling or precipitation fouling, is caused by the deposition or precipitation of inorganic particles and crystallization of mineral salts, oxides and hydroxides present in the feed [69].

– Organic fouling: Natural organic matter is a primary component of organic fouling. Organic matter includes complex organic substances as polysaccharides, proteins, nucleic acids, humic substances and fatty acids generated by the microbial decay of plants and vegetables [70, 71]. These compounds contribute to form an organic gel layer on top of the membranes and inside its pores. It is considered that adsorption is the initial precursor of such layer [72, 73].

– Particulate/colloid fouling: Colloids cover a wide size range, from a few nanometres to a few micrometres [74]. Particle matter in natural waters and wastewaters has been classified in the following categories. Settleable solids > 100 μm, supra-colloidal solids; 1–100 μm, colloidal solids 0.001–1 μm [75]. Generally, particles close to the size of membrane pores can cause pore plugging while those much larger can accumulate on the membrane surface forming a cake layer that provides an additional hydraulic resistance to water flux [76].

Depending on the type of blocking, four fouling modes can be observed, which are represented in Fig. 4 [74, 77, 78].

– Complete pore blocking: Meaning the complete sealing of pores by particles. This blocking requires foulant sizes larger than membrane pores.

– Standard pore blocking. It refers to the constriction of membranes pores due to the attachment and deposition of small particles at the internal pore walls.

– Intermediate pore blocking is a combination of the preceding ones. In it, particles block membrane pores and attach to other particles on membrane surface building up bridges between pores.

– Cake layer: Additional particles are deposited outside the external membrane surface contributing to the development of a filtration cake layer.

– Microbial fouling or biofouling: Biofouling has been defined as the undesired development of biofilms on surfaces [79]. The International Union of Pure and Applied Chemistry defines biofilms as an "aggregate of microorganisms in which cells that are frequently embedded within a self-produced matrix of extracellular polymeric substance (EPS) adhere to each other and/or to a surface" [80].

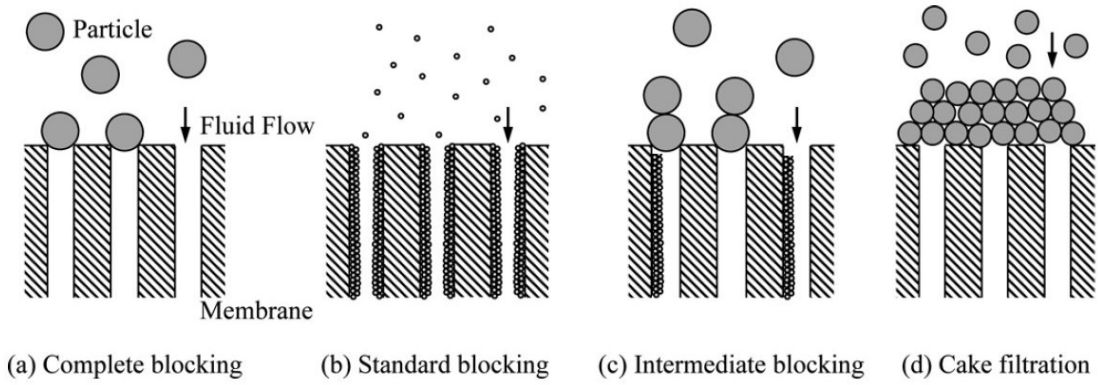

Figure 4. Schematic illustration for four different blocking filtration mechanisms. Adapted from Ref. [78].

DOI: 10.1007/s41204-020-00077-x

Biofilm formation comprises three different steps: adhesion, maturation, and dispersion. The adhesion step could be categorized as a two-stage process; initial reversible attachment and irreversible attachment [81]. Fig. 5 represents the process of biofilm formation. It begins when planktonic cells and nutrients present in the feed are transported to the surface. Initially, single cells are associated loosely with the surface via physicochemical forces. The use of fimbriae and flagella may also provide mechanical attachment to the surface [82, 83]. Then, on a time scale ranging from seconds to minutes, cells express EPS that facilitate binding to the surface leading to the irreversible bacterial attachment to the surface. Mature biofilms present increased cell density and complexity. Within the biofilm matrix, there are channels for the circulation of water and nutrient which keep cells interconnected. Consequently, interspecies bacterial can interact among themselves, sharing different metabolic substrates. Finally, some bacteria and biofilm aggregates can be released from the matrix, allowing the biofilm to expand on the surface and colonize new niches [84]. EPS can be defined as a "polymeric conglomeration generally composed of extracellular biopolymers such as polysaccharides and proteins, in various structural forms" [70]. EPS production offers several advantages for biofilm-forming microorganisms, as they provide mechanical support for the bacterial community and protection against several environmental conditions such as dehydration or salinity [85]. Biofilm formation and bacterial quorum sensing (QS) are closely interconnected processes. Quorum sensing can be defined as "a cell–cell communication mechanism that synchronizes gene expression in response to population cell density" [86]. This process is coordinated by small diffusible molecules called; autoinducers. The concentration of these signalling molecules regulates the expression of a series of genes, allowing cells to modulate surface adhesion, EPS production, maturation, and/or the dissolution of the biofilm [87, 88]. QS is a complex communication system able to detect cell density in a specific bacterial community, and as a function of it and environmental factors, regulate gene transcription to create adaptive responses [89].

It is well-known that membrane-solute interactions and membrane surface chemistry play an important role in understanding fouling. Membrane fouling is a complex phenomenon resulting from several, which can be classified into three different categories [91]:

– Physico-chemical composition of the feed solution: The physicochemical properties of individual feed molecules such as morphology, concentration, charge, hydrophobic interactions determine the formation of membrane fouling due to specific interactions between membrane and the foulants present in the water [15].

– Operating conditions: Operating parameters including transmembrane pressure (TMP), feed crossflow velocity, feed concentration, temperature, or pH play an important role in membrane performance and a direct effect on membrane fouling [92]. A compressive review of the different factors is given elsewhere [93].

– Membrane characteristics: The intrinsic characteristic of membranes, such as surface roughness, hydrophobicity or hydrophilicity, pore size and surface charge, have an important influence on solute-membrane interactions, and thus on membrane fouling. It is widely assumed that hydrophilic membranes are less susceptible to fouling than hydrophobic ones. Moreover, membrane fouling is promoted by rougher surfaces. The presence of protuberances on the surface contributes to membrane fouling by capturing suspended organic and inorganic matter, while membranes with smoother surfaces and less susceptible to fouling. Usually, membranes with larger pores are more prone to irreversible fouling because colloids penetrate more easily. In addition, most membranes

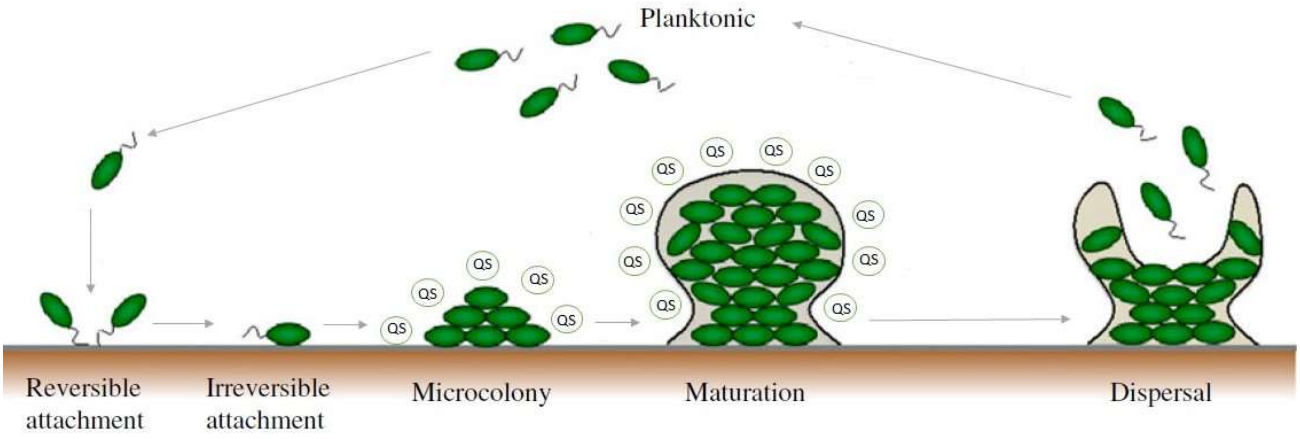

Figure 5. Biofilm formation steps: bacterial attachment to a surface, microcolony formation, biofilm maturation and bacterial dispersion. Quorum sensing molecules are responsible for cell-to-cell communication into the biofilm. Adapted from Ref. [90].

DOI: 10.1007/s41204-020-00077-x

exhibit a net negative charge under common operation conditions, that may produce electrostatic forces between the membrane surface and some charged particles present in the feed [93, 94].

Strategies to mitigate fouling and biofouling.

Many modifications have been proposed to improve the fouling and biofouling behaviour of phase inversion membranes since their early foundational discovery dated back to the 60s' [95]. The first successful strategy was to use PVP as additive, which added to the casting solution of PES UF membranes was shown to modify pore size [96]. In what follows, newly proposed grafting, coating, or blending approaches aimed at improving membrane fouling or biofouling behaviour are briefly summarized and commented.

Surface grafting

Surface grafting refers to the addition of hydrophilic chains, functional moieties or electrostatically charged groups onto the membrane surface. Surface grafting creates permanent surface changes by covalent bonding between grafted chains and membrane [97, 98]. This technique presents some advantages such as being an easy modification process allowing relatively higher chemical stability with high spatial control of grafting onto the desired surface [99]. However, it has the disadvantage of requiring energy intensive methods, resulting in an increase in membrane cost and the difficulty to scale-up. Table 2 shows a summary of recent studies using surface grafting. Membrane surface can be activated for grafting using different methods:

Plasma-induced grafting

Plasma can be defined as the fourth state of matter and consists of an electrically quasi-neutral gas partially ionized. Plasma is typically achieved when gases are excited into energetic states using microwaves or radio frequency waves [100]. When a polymeric material is exposed to it, different functional groups can be created on its surface that can be used for subsequent grafting or crosslinking reactions. In this way, plasma treatment can be classified into two categories. A schematic representation of these mechanisms is represented in Fig. 6.

Plasma functionalization or plasma activation: The substrate is struck with electrons and ions from the plasma-phase to generate surface radicals. The attachment of functional groups depends on the plasma gas used, which may be either inert or reactive. Plasma activation using reactive gases such as oxygen results in the introduction of carboxylic acid, hydroxyl, or peroxide functional groups. Jahel et al. activated the surface of polypropylene (PP) membranes used oxygen plasma treatment, which allowed the introduction of oxygen-containing functional groups, facilitating the deposition of TiO_2 nanoparticles (NPs) by dip-coating [101]. Plasma from carbon dioxide or carbon monoxide results in the introduction of carboxyl acid groups and generates hydroxyl, aldehyde, ester and ketone groups [102]. Nitrogen and ammonia plasmas generate surface primary, secondary and tertiary amines, and amides. Although treatment with inert gas plasma such as helium or argon does not result in the production of surface functional groups, it tends to be less aggressive rendering more stable membranes. Inert plasma can be used in combination with other gases or monomer precursors to produce homogeneous plasma discharge [103]. The addition of polar groups mainly occurred after treatment, when the polymer was exposed to oxygen from air [100].

Plasma polymerization or plasma deposition involves monomer fragmentation and radical site formation on the membrane surface using a plasma discharge. Reactive fragments can recombine forming polymers in the gas phase, so creating a plasma-deposited polymer coating on the substrate [104]. Therefore, instead of the attachment of functional groups, free radicals on the surface can initiate graft polymerization [105].

UV grafting

Photochemical-initiated graft polymerization, especially UV grafting, is one of the most common techniques for membrane surface modification due to its simplicity, versatility and low-cost. This method modifies the membrane surface by introducing functional groups without substantially affecting bulk properties [106]. The photo-initiated graft polymerization can be performed with or without photoinitiator. Polyarylsulfone membranes are intrinsically photo-sensitive, which means that they can produce active sites or free radicals upon irradiation without the presence of any photoinitiators. It has been reported that UV-light absorption takes place in the backbone of the PES polymeric chains due to the phenoxyl–phenyl chromophores present in its structure, resulting in a homolytic cleavage of C–S bonds at the position of a sulfonic group. This process gives rise to the formation of two radical positions, an aryl radical and the sulfonyl radical that later lose its sulfonyl group producing an additional aryl radical that induces the grafting process [107]. The modification of polyacrylic-polyethersulfone nanofiltration membranes via UV photografting was demonstrated using a solution of acrylic acid monomers, the resultant membranes showing higher water permeability and lower reverse salt diffusion [108]. Igbinigun et al. grafted allylamine monomers on the active surface of PES membranes using UV light followed by the binding of graphene oxide nanosheets. Functionalized membranes showed smooth surfaces, higher hydrophilicity, lower fouling attachment and higher water flux [109]. Nevertheless, other polymeric membranes require the addition of a photoinitiator or photosensitizer and an additional step, known as

DOI: 10.1007/s41204-020-00077-x

Table 2. Summary of selected recent studies using surface-grafting modification techniques.

Base membrane	Modification	Main results	Reference
Polyvinylidene fluoride (PVDF)	Argon-plasma treatment + Polystyrene sulfonate deposition	Average pore radius decreased Selective to divalent anions Good removal of Cr (VI) and stability	[121]
Polysulfone (PsU)	Argon-oxygen plasma treatment + Natural seed basil gum NPs	Increased pure water permeability Reduced membrane fouling Higher BSA (bovine serum albumin) rejection rate	[122]
Polypropylene (PP)	O_2 plasma treatment + Deposition of TiO_2 NPs	Significant improvement of the membrane hydrophilicity	[101]
Polysulfone (PsU)	NH_3-O_2 plasma treatment	Higher hydrophilicity and permeability Enhancement of membrane antifouling properties using BSA (bovine serum albumin)	[123]
Polyethylene terephthalate (PET)	Plasma-induced graft polymerization	Higher hydrophilicity Decreased effective pore radius	[124]
Polyvinylidene fluoride (PVDF)	Plasma induced self-polymerization of PAA + Self-assembly of ZnO NPs	Higher membrane hydrophilicity and water flux Self-cleaning and antifouling properties under UV light	[125]
Polyethersulfone (PES)	UV-grafting of acrylic acid monomers	Higher water permeability Lower salt diffusion	[108]
Polyethersulfone (PES)	Grafted allylamine monomers using UV-light	Smooth surface, higher hydrophilicity, surface ζ-potential and water flux Lower fouling with humic acid	[109]
Polyvinylidene fluoride (PVDF)	UV-grafting of graphene oxide via benzophenone-initiated crosslinking	Strong antibacterial activity No effect on membrane permeability or solute rejection properties	[110]
Polypropylene (PP)	UV-grafting polyacrylic acid (PAA) using benzophenone as photo initiator	Higher water flux and rejection rate	[111]

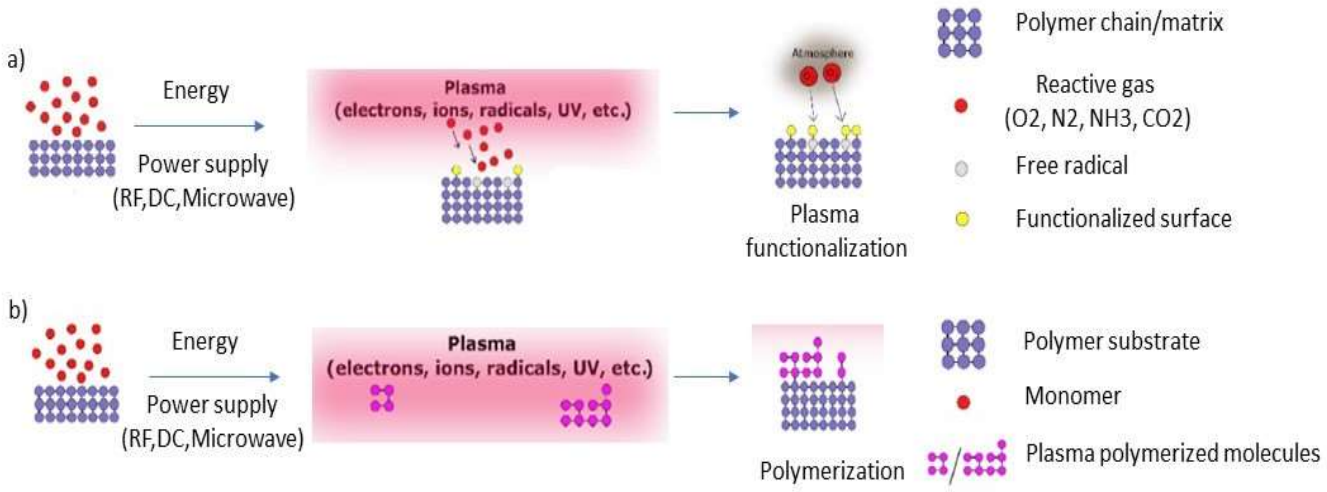

Figure 6. Schematic representation of plasma functionalization (a) and plasma polymerization (b) mechanism. Adapted from Ref. [105].

photoactivation to initiate the grafting process. Kaneda et al. modified polyvinyl fluoride (PVDF) membranes by irreversibly grafting graphene oxide (GO) nanosheets via benzophenone-initiated crosslinking reaction under UV irradiation. The resulting membranes showed higher antibacterial activity against *Escherichia coli* without compromising solute retention properties or membrane permeability [110]. Yang et al. grafted monomer of acrylic acid on the surface of PP hollow fibre membranes using benzophenone as a photoinitiator under UV irradiation to obtain membranes with higher flux and better rejection rate [111].

DOI: 10.1007/s41204-020-00077-x

Membrane surface coating

Surface coating is a simple, economical, and environmentally friendly method for surface modification, which involves the deposition of a layer on membrane surface [112]. This technique aims at reinforcing surface properties causing minimal structural effects. The major disadvantage of this method is that coating layer can be unstable. In this way, surface coatings created with strong covalent bonding at the substrate-coating interface offer enhanced performance and long-term stability [113]. Moreover, high molecular weight polymers are used to avoid the penetration of the coated layer into membrane pores [98]. Table 3. shows a summary of recent studies of different surface coating membrane modifications.

– Polydopamine coating. Polydopamine is a biopolymer, inspired by the strong adhesion property of mussels, that can easily self-polymerize under alkaline conditions (pH typically between 7.5 and 8.5) using oxygen as an oxidant, to yield a very thin layer onto many substrates [114, 115]. Polydopamine coating imparts high surface hydrophilicity and anti-organic/fouling properties. Furthermore, polydopamine presents multiple functional groups that can interact with a wide range of molecules, providing an important platform to form covalently grafted functional layers over a substrate [116].

– Electrospun nanofiber layer is a method that offers some unique benefits due to the superior properties of the nanofibers, which exhibit high interconnectivity, tunable porosity, tunable functionalization and high surface area to volume ratio [117]. Electrospun layers can also incorporate different antimicrobial agents such as metallic NPs, carbon nanomaterials or antimicrobial biopolymers, thereby contributing to reduce the biofilm formation [118]. Electrospinning is the only technique generally available to produce fibres with extremely small diameters. As shown in Fig. 7, an electrospinning system consists of three different components: a high voltage power supply, a spinneret, and a collecting plate usually a metal screen plate, or rotating device. This technique utilizes a high voltage source to inject charge of a certain polarity into a polymer solution, which is then accelerated towards a collector of opposite polarity [119].

Blending technique

Polymer blending is a process in which two or more compounds are physically mixed into the casting solution using the same solvent. Polymeric materials or inorganic nanofillers are frequently used as additives in the phase separation process to manipulate membrane properties. Since most of these additives are hydrophilic in nature, this method is considered the simplest way to enhance membrane hydrophilicity to reduce fouling. The limiting factor associated with this technique is the limited compatibility between hydrophilic additives and hydrophobic polymers as well as the leaching of blended materials after long-term use [107, 134]. Table 4 shows a summary of the recent studies of blending polymeric membranes using inorganic and organic compounds.

Blending inorganic materials

Membranes that combine the features of inorganic fillers and polymeric materials are commonly known as mixed matrix membranes (MMMs). MMMs aim to take great advantages of the processability, durability, permeability, and selectivity of polymers by offering the advantage of a unique surface chemistry [135]. Introducing inorganic fillers to the polymeric matrix enhance the thermal properties (e.g. thermal stability or thermal conductivity), and the mechanical stability (e.g. tensile strength, Young's modulus, stiffness, and hardness) of composite membranes compared to pure polymeric membranes [136]. Furthermore, uniform dispersion of inorganic materials into the polymer matrix provides significant improvement in physical

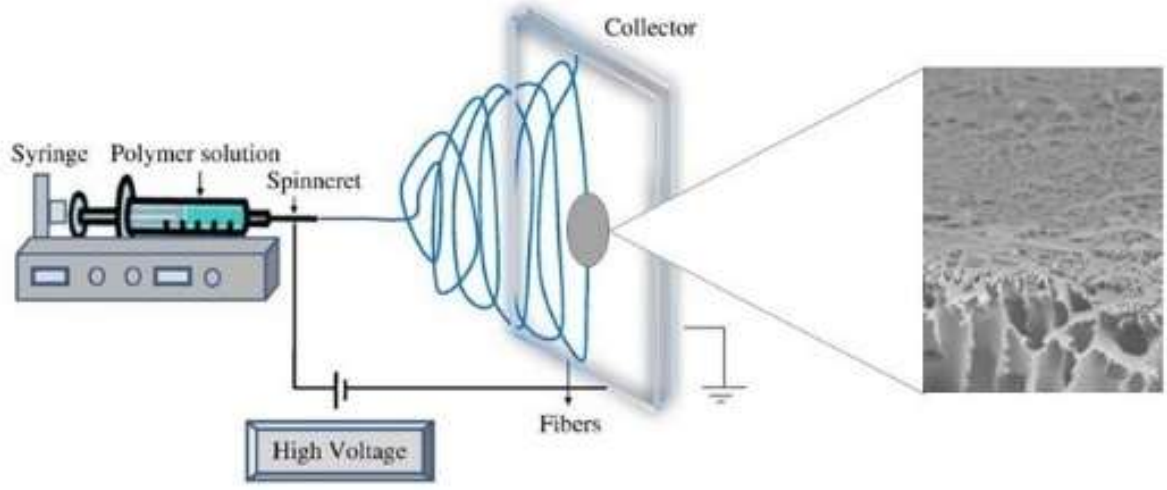

Figure 7. Scheme of the electrospinning system with major components. Adapted from Ref. [120]

DOI: 10.1007/s41204-020-00077-x

Table 3. Summary of recent studies of membranes modified using different surface coatings.

Base membrane	Modification	Main results	Reference
Polysulfone (PsU)	Polydopamine (PD)-layer	Increased membrane surface hydrophilicity Excessive deposition decreases membrane permeability, due to pore blocking and reduced pore size Optimal concentration 2 mg/mL, 15 min deposition time	[126]
Polysulfone (PsU)	Silver (Ag)-PD layer	"in situ" immobilization of AgNPs Increased pure water flux, maintaining BSA rejection. Enhanced protein-fouling resistance Good antibacterial activity	[127]
Poly(vinylidene fluoride)(PVDF) + Polysulfone (PsU)	PD-layer	Restored the properties and performance of aged membranes Low water flux reduction, higher retention Lower protein adsorption and polysaccharide accumulation	[128]
Poly(ether imide) (PEI)	PD layer + immobilized AgNPs	Improved permeation and separation Antibiofouling against gram-positive and gram-negative bacteria	[129]
Poly(vinylidene fluoride) (PVDF)	Dopamine coating + in situ immobilized CuNPs	Increased surface hydrophilicity Enhanced antibacterial activity against the gram-negative bacteria *E. coli*	[130]
Poly(vinylidene fluoride) (PVDF)	TiO_2 electrospun nanofiber coating	Higher hydrophilicity Enhanced antifouling behaviour, higher glucose rejection rate	[131]
Polysulfone (PsU)	Poly(acrylic acid) (PAA)-poly(vinyl alcohol) (PVA) electrospun layer	Increased membrane hydrophilicity Reduced organic fouling (BSA) Antimicrobial activity against *E. coli* and *S. aureus*	[132]
Poly(vinylidene fluoride) (PVDF)	Electrospun PVDF nanofibers containing SiO_2 NPs	Superhydrophobic membranes More stable flux than uncoated membranes	[133]

properties such as viscoelastic properties, crystallinity, density, conductivity, structure, or morphology. The nanocomposite membranes generally possess higher viscoelastic properties than the pristine membranes [137]. Several inorganic materials have been incorporated into the polymer matrix of MMMs membranes:

Inorganic NPs are a promising additive, which may improve membrane performance and properties. Many types of inorganic materials have been directly incorporated into the polymer solution during membrane preparation, including titanium dioxide, graphene oxide, alumina or silver and copper NPs [138]. TiO_2 is as attractive choice due to its characteristic properties such as good chemical and thermal stability, low toxicity, photocatalytic activity, superhydrophilicity, and selfcleaning capacity that can be used to mitigate fouling [139, 140]. Anvari et al. incorporated TiO_2 NPs in the PVDF/PAN casting solution to prepare UF composite membranes by phase inversion. Blended membranes exhibited higher hydrophilicity, improved pure water flux, and enhanced antifouling properties [141]. AgNPs received a great deal of attention due to their broad spectrum of antibacterial properties and low cytotoxicity [6]. AgNPs serve as a local supply of Ag+ ions, which can prevent bacterial colonization and reduce solute adhesion onto membrane surface. Rehan et al. prepared PES membranes blended with AgNPs using the immersion precipitation technique to obtain membranes with good antibacterial and antibiofouling properties [142]. However, the incorporation of inorganic materials has two important drawbacks. On the one hand, nanoparticles are prone to aggregate during dope preparation. On the other hand, there is a risk of NP release to the environment [143]. To overcome these problems, a variety of porous materials such as mesoporous silica nanoparticles, nanominerals materials (halloysite nanotubes, sepiolite, zeolite) or metal–organic frameworks (MOFs) have been proposed as supports to control the nanoparticle stability.

Mesoporous silica particles gained popularity over the last years due to their useful characteristics such as high specific surface area, uniform pore size between 2 and 50 nm and easy functionalization. Mesoporous silica can be synthesized in basic and acidic environments and normally relies on a surfactant template to generate its porous structure [144]. The most common types of mesoporous nanoparticles are Mobil crystalline material (MCM-41) and Santa Barbara Amorphous (SBA-15). Guo et al. prepared PES membranes using functionalized SBA-15 material with titanium and zirconium nanoparticles to improve membrane hydrophilicity and permeability. Antifouling behaviour was obtained using a concentration as low as 0.6 wt% SBA-15 [145]. Martín et al. functionalized SBA-15 with amine and carboxylic groups by co-condensation method thereby enhancing the surface porosity, hydrophilicity, and permeability of PES membranes.

DOI: 10.1007/s41204-020-00077-x

The antifouling properties of composite membranes improved, especially against irreversible fouling [146].

Halloysite nanotubes (HNT) are a naturally occurring form of halloysite, an aluminosilicate nanoclay mineral with chemical formula $Al_2Si_2O_5(OH)_4 \cdot 2H_2O$ [147]. Its crystalline configuration consists of a 1:1 multi-walled inorganic nanotube formed by tetrahedral (Si–O) and octahedral (Al–OH) sheets with a hollow tubular structure and regular open-ending pores [148, 138]. It is commonly used as a filler in polymeric matrixes due to its large surface area, tubular shape, well-crystallized structure, and excellent dispersity [149]. Chen et al. prepared PES UF membranes via phase inversion method containing HNTs loaded with copper ions (Cu^{2+}) as an antibacterial agent. Cu^{2+}-HNTs/PES membranes were more hydrophilic and presented enhanced permeability [150].

Sepiolite is a hydrated magnesium silicate with the theoretical formula $Mg_8Si_{12}O_{30}(OH)_4(H_2O)_4 \cdot 8\ H_2O$ [151]. Its structure can be defined as a quincunx arrangement of blocks separated by parallel channels. This configuration induces a needle-like particle shape, which possesses excellent sorptive properties and large specific surface area.[152]. Sepiolite has been employed as support to incorporate metallic NPs into the silicate matrix after magnesium lixiviation in acid conditions [153]. Díez et al. prepared composite PSU-PVP UF membranes by phase inversion including sepiolite supported nanometals. NP stability was confirmed as no nanomaterials migrated to the filtrate. The antimicrobial behaviour prevented bacterial colonization for either gram-positive and gram-negative bacteria [154].

Zeolites are microporous, hydrated aluminosilicate minerals with a general formula of $M_{x/m}[(AlO_2)_x(SiO_2)_y]$, where $M_{x/m}$ refers an ion-exchangeable cation [155]. It has been demonstrated that the incorporation of zeolites into a polymer matrix increased membrane permeability, additionally enhancing mechanical strength, thermal resistance, and chemical stability [156]. Moreover, NPs can be loaded inside zeolites for antibacterial applications [157]. Yurekli et al. prepared PsU membranes impregnated by zeolite particles to remove heavy metals from water [158]. Shi et al. prepared PVDF UF membranes containing Ag-loaded zeolite nanoparticles with long-term antibacterial capacity [159].

Metal–organic frameworks (MOFs) are crystalline microporous materials that consist of a regular network of metal ions interrelated by multifunctional organic molecules [160]. They show some special features such as a large surface area, high pore volumes, tuneable pore size and high metal content that offer valuable active sites [161]. Firouzjaei et al. explored the synergetic effect of graphene oxide NPs incorporated in a silver-based MOF into a thin film nanocomposite membrane to improve antibiofouling and antifouling properties [162]. Mohammadnezhad et al. prepared PES NF membranes by the phase inversion method, modified with nanocrystalline Ce(III) MOFs. Composite membranes showed higher permeability, hydrophilicity, dye rejection capacity and good antifouling behaviour during wastewater treatment [163]. Yang et al. fabricated composite CA UF membranes by blending graphene oxide (GO) and MOF-GO in the matrix. Modified CA/MOF-GO hybrid membranes showed larger pores size and smoother surfaces. The hydrophilicity and water flux were also improved, exhibiting satisfactory performance in water purification process [164].

Blending organic materials

Hydrophilic polymer additives such as PVP or PEG are common additives used to improve membrane performance or to facilitate membrane fabrication. PVP is an excellent pore forming agent due to its hydrophilicity. Thermodynamically, it works as a demixing enhancer that accelerates the phase inversion process, contributing to form a uniform finger-like porous structure, which results in improved membrane flux [165]. Incidentally, it has been observed that increased PVP concentration, suppresses macrovoid formation leading to a decrease in water permeability [166]. PEG is a promising hydrophilic additive used to promote pore formation and to enhance permeation properties in polymeric membranes [167]. PEG is available in a variety of molecular weights with a general formula of $H(OCH_2CH_2)_n$-OH, where n is the average of repeating oxyethylene groups [168]. PEG also reduce the thermodynamic stability of the casting solution leading to the formation of finger-like porous structures. Xu et al. found that increasing the PEG molecular weight from 200 to 10 000 Da in the casting solutions, membrane morphology changed from finger-like porous structure to spheres or ellipsoids with poorer mechanical properties [169].

Dendritic polymers constitute a family that includes dendrimers and random hyperbranched polymers (HBPs), which has received considerable attention to develop a variety of nanoscale materials [170]. A dendrimer is a polymer that contains numerous terminal functional groups in a highly branched topological structure [171]. Dendrimers are produced step-by-step in a controlled and iterative manufacturing process, growing off a central core, each subsequent step representing a new “generation” of dendrimer. Their properties can vary depending on the size (generation) and the number or density of terminal functional groups [172]. Among the various classes of dendrimers, primary amine terminated polyamidoamine (PAMAM) was the first family to be commercialized [173]. Bharali et al. prepared PsU composite membranes by phase inversion using different non-solvent additives. PAMAM-dendrimers (G0) were directly incorporated

DOI: 10.1007/s41204-020-00077-x

Table 4. Recent studies of blended and composite polymeric membranes

Base membrane	Blending Modification	Main results	Reference
PES (Polyethersulfone)	TiO_2 NPs	Mitigated membrane fouling Increased macrovoid porous structure Improvement of membrane permeation flux.	[180]
PVDF/PAN (Poly(vinylidene fluoride) (Polyacrylonitrile)	TiO_2 NPs (PVDF/PAN/TiO_2)	Enhanced membrane hydrophilicity Improvement pure water flux and antifouling properties	[141]
Polysulfone (PsU)	Graphene Oxide (GO)	Enhanced hydrophilicity, porosity, permeability and pure water flux Improved mechanical properties at low GO concentration and higher ion rejection properties	[181]
PES (Polyehtersulfone)	Ag-NPs	Antibacterial and antibiofouling properties Enhanced permeability	[142]
Polysulfone (PsU)	Silver-GO NPs	Antimicrobial activity against gram negative (*E. coli)* and gram-positive *(S. aureus)* bacteria. BSA-fouling reduction	[182]
Polyethersulfone (PES)	Ti, Zr NPs embedded in SBA-15 mesoporous silica	Improved membrane permeability and hydrophilicity Lower BSA membrane absorption	[145]
Polyethersulfone (PES)	Amine and carboxylic functionalized SBA-15 particles	Enhanced surface porosity, hydrophilicity and water permeation flux. Reduced fouling adhesion, especially irreversible fouling.	[146]
Polyethersulfone (PES)	Halloysite nanotubes loaded with copper ions (Cu^{2+}-HNTs)	Higher membrane hydrophilicity and permeability Enhanced mechanical strength Good antibacterial activity against *E. coli* and *S. aureus*	[150]
Polysulfone (PsU)	Sepiolite-loaded silver and copper nanoparticles	Enhanced surface hydrophilicity, higher nanoparticle stability Effective antimicrobial activity preventing bacterial colonization	[154]
PVDF Poly(vinylidene fluoride)	Blending Ag^{+}-Zeolite particles	Improved hydrophilicity Higher thermal stability Enhanced mechanical properties Long term antifouling capability and excellent antimicrobial activity against *E. coli*	[159]
Thin-film composite (TFC)	Graphene oxide (GO)-Ag-based metal-organic framework (Ag-MOF)	Higher hydrophilicity and water permeability Improved antibiofouling and antifouling properties	[162]
Polysulfone (PsU)	Polyvinylpyrrolidone (PVP)	Improved pure water flux and membrane hydrophilicity and higher antifouling behaviour	[183]
PVDF Poly(vinylidene fluoride)	Polyethylene glycol (PEG)	Enhanced pore formation Higher molecular weight leads to higher permeability	[184]
Cellulose acetate (CA)	Polyethylene glycol (PEG)	Enhanced pure water flux and better pore distribution Enhanced hydrophilicity	[185]
Polysulfone (PsU)	PAMAM-Dendrimers (G0)–PEG (polyethylene glycol)	Higher permeability Effective for CO_2 permeation	[174]
PVDF Poly(vinylidene fluoride)	PAMAM (G1)-dendrimer encapsulated Pt NPs	Highly active and reusable catalyst for the hydrogenation of alkenes and alkynes to the corresponding alkanes	[175]
PVDF Poly(vinylidene fluoride)	Hyperbranched polyglycerol (HPG)	Pore forming agent, enhanced pore size Higher hydrophilicity Increased pure water flux	[178]
PVDF Poly(vinylidene fluoride)	Amphiphilic hyperbranched poly(ether amine) (hPEA)	Selective adsorption of hydrophilic dyes Enhanced hydrophilicity	[179]

DOI: 10.1007/s41204-020-00077-x

into the polymeric solution, providing a selective layer for CO_2 absorption in gas separation processes [174]. Furthermore, dendrimers can be used as a template to encapsulate different compounds in their inner void spaces or attached to their surface. Kotte et al. demonstrated an easy route to prepare catalytic PVDF membranes with *in situ* synthesized PAMAM (G1) dendrimers that were used as hosts and containers for platinum NPs [175]. Li et al. used PAMAM dendrimers for preparing dendrimer encapsulated AgNPs, which were grafted onto the surface of PVDF membranes via interfacial reaction, showing good solubility, permeability, and antibacterial properties [176]. Hyperbranched polymers (HBPs) are highly branched polymers, which, like dendrimers, contain numerous terminal functional groups, spatial cavities, and some unique physical and chemical properties [177]. In contrast to dendrimers, HBPs can be easily synthesized in one step polymerization processes, which makes them a cheap analogue of dendrimers and excellent candidates for large scale applications [172]. Zhao et al. prepared PVDF membranes via phase inversion using the hyperbranched polyglycerol as additive. Membranes with higher hydrophilic character, and surface pore size were obtained with increased water flux [178]. Ji et al. developed a novel amphiphilic hyperbranched poly(ether amine) (hPEA) by introducing epoxy-containing coumarin moieties (EC) and fluorinated carbon chains (CF6) through epoxy/amine click chemistry. The blended solution was prepared by dissolving PVDF and the resulting copolymer (hPEA-EC-CF6), and membranes prepared by phase inversion showed good adsorption properties to hydrophilic dyes in aqueous solutions [179].

Conclusions and recommendations

This article presents a critical review of recent articles dealing with modification procedures aimed at reducing membrane fouling and biofouling. The attention is focused to membranes produced by phase inversion that incorporate different organic and inorganic additives as well as to techniques aimed at modifying membrane surface. The rationale of the studies covered in this review is that the introduction of new substances of functional groups, either in the bulk of modifying membrane surface may contribute to the reduction of flux due to the deposition of organic and inorganic substrates. Similar arguments have been proposed to modify membranes to make them resistant to bacterial attachment and biofouling. Polymer additives such as PVP and PEG are well known to induce pore formation and to improve the characteristic asymmetric structure of membranes prepare by phase inversion. With the same motivation, a plethora of inorganic and organic blending materials have been proposed to enhance hydrophilicity, modify pore structure or to create a reservoir of different substances, notably metals with antibacterial activity. Surface modification may take place by inducing specific moieties via the post-functionalization strategies of grafting or coating. This includes plasma and irradiation for surface activation of the relatively inert membranes used in most commercial separations. Most of the studies mentioned in this work cited correspond to a laboratory stage of development, with little insight into practical aspects like long-term stability, behaviour under real operating conditions or limitations associated to the cost of sophisticated membranes. Future work should emphasise the testing of membranes in continuous semi-pilot scale during times at least in the order of magnitude of the life span or commercial membranes. Operation in relevant conditions include the use of real effluents or streams and the testing of cleaning procedures. Membrane viability in terms of mechanical properties and stability should be encouraged in studies proposing newly created membranes. Finally, an estimation of production cost and the adequacy of current industrial production methods to implement the proposed innovations would be acknowledged.

Acknowledgements. BD thanks the University of Alcalá for the award of predoctoral grant. Moreover, we would like to acknowledge the original sources from which we have adapted the figures used in this work; Elsevier's: Fig. 1, Kim et al. [31]; Fig. 2, Figoli et al. [48]; Fig. 6, Nageswaran et al. [105]; Fig. 7, Bhardwaj and Kundu [120]. American Chemical Society: Fig. 3; "Adapted with permission from Guillen et al. [47]. Taylor & Francis: Fig. 4, Iritani [78]; Fig. 5, Toyofuku et al. [90].

Funding. Financial support was provided by the Spanish Ministry of Science, Innovation and Universities (CTM2016-74927-C2-1-R).

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DOI: 10.1007/s41204-020-00077-x