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Membrane separation as a pre-treatment process for oily saline water

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Abstract

Oil and gas industry generate large quantities of oily wastewater effluents. This wastewater has a major impact on the environment and human health. Hence, a suitable separation method is applied to treat oily wastewater to not only meet the environmental regulations but also to promote water recycling and desalination. Many studies were performed in the literature to investigate the best technologies for treating oily saline water such as the traditional technique of gravity sedimentation and dewatering. Among all, membrane separation processes have been receiving extra attention in the past decades. This is due to their high separation efficiency, low energy requirements and easy operation.

Additional research activities were also directed to utilize membranes in pre-treatment separation processes of oily water ahead of the desalination units. This paper presents a comprehensive review for the recent treatment processes available in the literature for oily wastewater with the concentration on the use of various membranes to accomplish this target. The paper also reviews the recent findings in membranes' development and emerging modification techniques such as interfacial polymerization, nanoparticles incorporation, and surface grafting. A special emphasis was given for ceramic membranes, their operation and their preparation techniques. Moreover, the paper compares and discusses the effect of different operating conditions such as trans-membrane pressure and cross flow velocity on membrane separation performance in oily water.

Keywords: Oily wastewater, Produce water, Oily saline water, Oil/water separation, Oil removal, Desalination, Membrane Technology, Polymeric membranes, Ceramic membranes, Membrane modifications.

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1. Introduction

Oil water separation has been receiving a great interest recently. The main reasons are the increase of the environmental and health consciousness as well as the increased demand for clean water. Oily wastewater is a common product of various chemical industries such as oil and gas, food and steel industries. This wastewater also results from oil spills to open water bodies during crude oil exploration, and crude and petroleum products transportation [1]. The scarcity of fresh water is becoming a severe problem worldwide primarily due to the rapid increase of industrial activities and steadily population growth [2,3]. The severity is well noticed in certain developing countries [4,5]. Thus, desalination of saline seawater is one of the key solutions available to secure freshwater supplies [6].

There are several techniques that are typically used for oily wastewater separation. Examples are gravity or centrifugal, electrostatic precipitation, cyclones, floatation, demulsification, heat treatment, adsorption and membrane separation technologies [7–9]. Padaki et al. provided a through comparison between several physical and chemical techniques that are mostly used for the separation of oily wastewater [10]. Membrane separation technologies, in particular, are efficient and more effective in removing oil droplets from oil water emulsions when compared to conventional methods [11–16]. Although, membrane separation methods possess several advantages such as high selectivity, low energy requirement, simple operation, reliability, low maintenance cost and small space [17–19], however, the major drawback of membrane separation methods is the fouling of the membranes. Hence, the objective of this article is to provide a comprehensive review of the recent advances in membrane separation techniques for saline oil water emulsions with a focus on the use of ceramic membranes.

2. Constituents of oily wastewater

The pollutants in oily wastewater are classified into two categories, organic and inorganic. The organic pollutants are mainly petroleum hydrocarbons (PHCs) and can be further classified into four major categories. These categories are aliphatic, aromatic, asphaltenes and the compounds that contain oxygen, nitrogen and sulfur [10,20]. These compounds are often accompanied with some nickel, cadmium, lead and vanadium organometallic complexes [21]. The organic pollutants can also be polar or non-polar with several functional group alternatives such as alcohol, carboxyl,

phenol and amine groups [22]. These pollutants typically either disperse, emulsify or dissolve within the oily wastewater.

In general, aliphatic and aromatic compounds count for up to 75% of petroleum hydrocarbons in oily wastewater [23,24]. Moreover, the oily wastewater may contain some processing residuals such as defoamers, demulsifiers, inhibitors, glycols and sulfur scavengers [25]. Table 1 shows an example of actual oily wastewater composition [26]. Releasing massive quantities of oily wastewater to water bodies causes further consumption of oxygen by the microorganisms [27]. This eventually leads to hypoxia (< 2 mg O₂/L) or even anoxia [28]. Oxygen is essential for eliminating colors, tastes, and odors through the chemical and biological reactions in aerobic wastewater treatment units [29].

Table 1: Example of actual oily wastewater composition [26].

Compounds	Conc. (mg/L)	Compounds	Conc. (mg/L)	Compounds	Conc. (mg/L)
COD	270 - 230	Chlorine	8475 - 9219	Stronyium	68 - 72
TOC	45 - 71	Sodium	5462 - 5836	Sulfte radical	61 - 68
TDS	14890 - 16237	Potassium	10 -12	Total Phosphorus	1.7 - 1.8
pH	7.3 -7.4	Calcium	356 - 372	Total Nitrogen	23 -26
Oil	96 - 112	Magnesium	114 - 118	Suspended solids	98 - 116

3. Processes for oil-water separation

There are several processes that are typically used in oily wastewater separation. In the following section the main practices will be discussed.

3.1 Gas flotation

In this process, the gas bubbles adhere to the surface of suspended oil droplets in oil/water mixture to form fine agglomerates. These fine agglomerates float to the surface due to the difference in density and can be eventually collected from the upper surface [30]. The success of this method

depends mainly on the contact and the attachment between gas bubbles and oil droplets and the floatability of bubble-oil aggregates. Floatability of the aggregates depends on the difference in density between water and the bubble-oil aggregates [31]. The higher the density difference, the more successful the separation is. The flotation process is proposed for the removal of oil from oil/water mixtures and it consists of four fundamental steps [32]. Firstly, the generation of gas bubbles. Secondly, making a contact between these floated gas bubbles and the suspended oil droplets in the oil/water mixture. Thirdly, the gas bubbles and oil droplets are attached together to form aggregates, and finally, these aggregates float to the surface where they are skimmed off. Figure 1 shows a conventional dissolved gas flotation system [31].

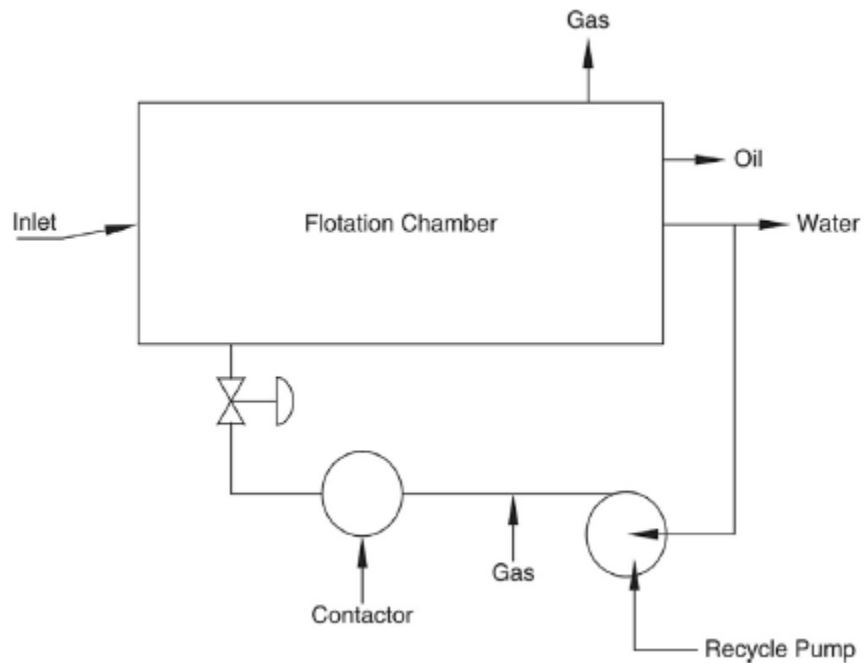


Figure 1: Conventional dissolved gas flotation system [31].

Several gases could be used in floatation, however, due to the availability and cost, air is the most commonly used gas [33,34]. Nevertheless, in certain applications where the presence of oxygen is unfavorable, other gases could be used such as methane, carbon dioxide and nitrogen [35]. The most popular floatation techniques are the dissolved gas floatation and the induced gas floatation [36]. They both have a very high efficiency in removing oil from oil/water mixtures and they are commonly used to separate oil when the oil concentrations are lower than 1000 mg/L [31].

3.2 Electrostatic precipitation

Frederick Gardner Cottrell invented the first electrostatic precipitator in 1907 [37]. The objective was to collect the dust of the blast furnace gas, the oxides from the fumes of lead and copper smelters and sulfuric acid mist from sulfuric acid production plants. Thereafter, the electrocoalescence technique was further developed. This technique involved the application of an electrical field for the separation of liquid phases such as oil/water emulsions [38]. The electric field is used to enhance coalescence rate by bringing the small droplets closer and enhance their agglomeration to form larger droplets [39]. Hence, the generated large droplets settle down easily through gravity force. The presence of the electric field improves the phase separation by increasing the droplets speed toward the electrodes [40]. This technique can be used to separate oil from saline water or water from crude oil. Both direct current (DC) and alternating current (AC) electric fields can be used. The strength and the frequency of the electrical field depend on the extent of the aqueous phase [41,42]. The high electrical fields are often utilized to separate water from crude oil emulsions [43]. A schematic of electrocoalescer is shown in Figure 2.

The coalescence process takes place in three major steps [44–46]. Firstly, oil droplets, if the water is the continuous phase, brought together while separated by a water film. Secondly, the water film will get thinner until it reaches a critical thickness. Any disruption at that critical thickness breaks the water film. This step is the controlling step, and the electrical field is used to fasten the film thinning process. Finally, coalescence occurs.

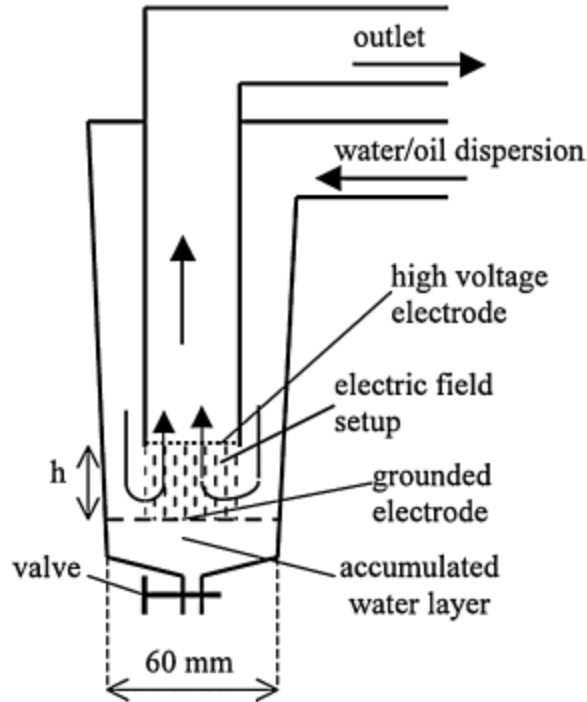


Figure 2: Schematic of electrocoalescer [39].

3.3 Solvent extraction

Solvent extraction technique has been extensively used to separate oil from oil/water emulsions. In this technique, oil is separated from water by adding specific organic solvent that is selectively miscible to the oily phase, while being immiscible to the aqueous phase [47]. Appropriate amount of the solvent is used to guarantee a complete miscibility of the oil. After the phase separation process, the immiscible water is settled down the extraction column. The oil is then separated from the solvent/oil mixture using a distillation unit [48]. Several solvents have been utilized such as, n-heptane, toluene, cyclohexane, propanol, butanol, methyl ethyl ketone, methylene dichloride, ethylene dichloride, diethyl ether, naphtha cut and kerosene cut [49–55].

There are several factors that determine the efficiency of the extraction process. These factors are the oil content, the type of solvent, solvent to oil ratio, mixing speed and duration, temperature and pressure [56]. The solvent extraction processes are easy to operate, utilize high diversity of solvents, they are distinguished by their high extraction efficiency and they operate at moderate

temperatures and pressures [57]. However, they have major drawbacks such as, the need of considerable volume of solvents, the substantial solvent losses and specially of the high volatile solvents, and the energy intensive distillation process that is used to separate oil from solvent [58,59]. These drawbacks restrain the large-scale applications of solvent extraction technique. Figure 3 shows a flow diagram of solvent extraction process.

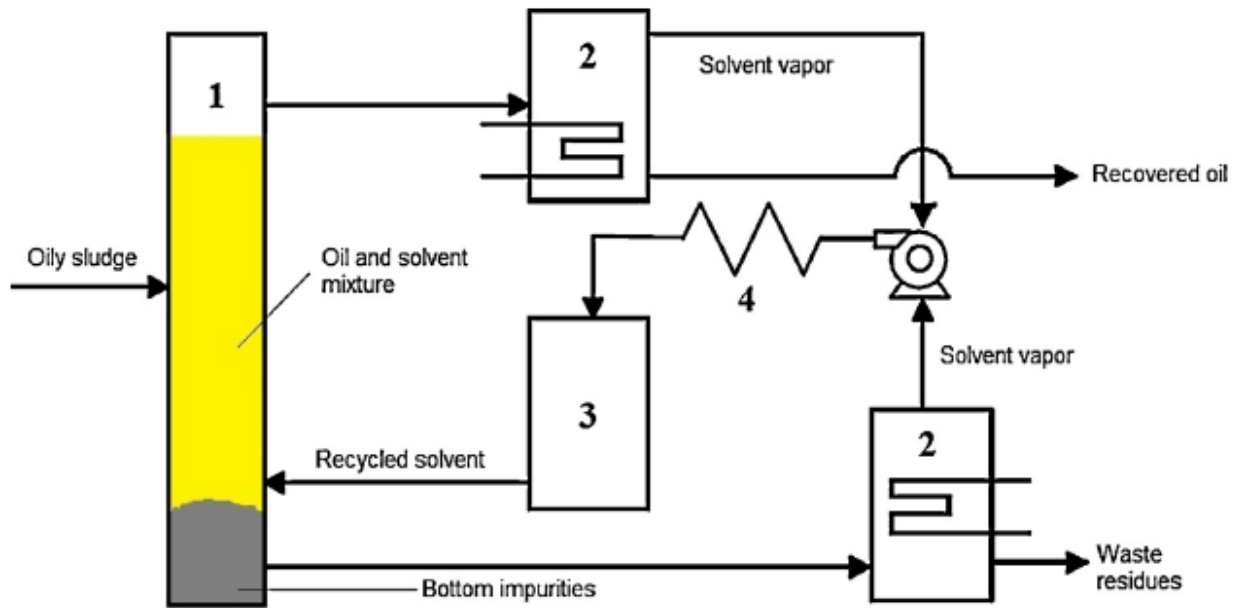


Figure 3: Flow diagram of solvent extraction process (1: reactor column; 2: distillation system; 3: solvent recycling tank; 4: compressor and cooling system) [53].

3.4 Centrifugation and Hydrocyclones

In centrifugation, a high-speed rotating device is used to create a centrifugal force. This centrifugal force is employed to separate constituents based on the differences in their densities. The centrifugal force required, and consequently the energy required are proportional to the density difference of the constituents involved [60]. The difference in density between oil and water is small, hence, for oil/water separation, the centrifugation process demand a strong centrifugal force and consequently a large amount of energy [61]. A possible approach to lower the energy

requirements is by reducing the feed mixture viscosity. This could be done by adding demulsifying agents [62,63], adding coagulant reagent [64], heat treatment [65], or steam injection [66].

Liquid-liquid Hydrocyclones are used to separate mixtures utilizing the differences between the centrifugal and the drag forces [67]. They are highly efficient and attractive for oil/water separation. Hydrocyclone is simply vertical pipe that have one inlet and two outlets. The feed, oil/water mixture in this case, is pumped through a tangential inlet and the product is separated to two streams. The dense aqueous stream is forced toward the outer side of the cyclone and drained to the lower outlet and the light oil stream suspense at the cyclone center before it is drawn to the upper outlet by vortices [68,69].

Hydrocyclones operate without moving parts, hence, it is easy to install and operate [70]. They require low operational and capital costs, and low operation time and space [71]. Therefore, they are capable of separating large volumes. However, hydrocyclones have several drawbacks such as the low separation efficiency at low flowrates and high viscosities, the breakage of the droplets at high velocities, and they require a high feed pressure in order to achieve a high centrifugal force [72,73]. Although, it is impossible to achieve a perfect separation of oil from water using the hydrocyclone technique, however, employing multi hydrocyclones in series would significantly enhance the separation efficiency [74].

3.5 Surfactant enhanced oil recovery (EOR)

Surfactants are simply amphiphilic compounds, i.e. they consist of hydrophobic and a hydrophilic tails. The latter promote the dissolution of surfactants in the aqueous phase and increase the solubility of some oil compounds, whereas the former allows the surfactants to accumulate at the interface [75,76]. This decreases the water surface tension between aqueous and oily phases; hence, increases oil compounds mobility [77,78]. Surfactants could be used to separate oil from oil/water mixture or to separate water from oily sludge. Separation processes using chemical surfactants are quite fast, economically feasible, able of treating huge volumes with separation efficiency of water from oil that can exceed 80% [79,80]. However, most of these surfactants are toxic and not biodegradable [81]. In contrast, biosurfactants are biodegradable with lower toxicity, more efficient with high emulsion activity, and they can be produced from various raw materials

[82–84]. In spite of that, the production cost for biosurfactants is still high which affects their economic feasibility and limits their commercialization [85].

3.6 Freeze/thaw process

Freeze/thaw process is used to separate oil/water emulsions into two phases. It is highly effective especially in cold places where it is possible to utilize the free frostiness [86–88]. This method relies on the fact that the volume of water expands when converting from liquid phase to solid phase. The volume expansion promotes the coalescence of water droplets and reduces the interfacial tension between the aqueous phase and the oil phase. There are two main mechanisms for the Freeze/thaw separation process. In the first mechanism, the aqueous phase freezes before the oil phase, hence, the water droplets volume expands and then coalesce. This leads to the emulsion instability. Thereafter, the freezing started in the oil phase and due to the interfacial tension the oil coalescence, and consequently the oil/water emulsion separated into two layers by gravity [89,90]. In contrast, in the second mechanism, the freezing starts in the oil phase surrounding the water droplets, hence, the frozen oil encapsulates the water droplets. Afterwards, the freezing proceeds to the encapsulated water droplets, leading to volume expansion and consequently, a rupture of the solid oil surrounding the water droplets. As a result, the unfrozen water parts connect together as a broad network. This network thaws and create small water droplets that coalesce, thus, generate an unstable emulsion that can be separated into two isolated layers by gravity [86,91,92].

The performance of the freeze/thaw process depends mainly on the freezing temperature, the freezing time, freezing method, water content, water constituents and the pH [92–97]. This process can be used to treat different types of wastewater and competent even with very stable and viscous emulsions and with the presence of numerous solid particles [97,98]. It is worth to mention that this process is environmentally friendly with no pollution generation and the separated oil can be collected and reused [20, 86,99].

Microwave irradiation [100,101], ultrasonic irradiation [102–104], sludge pyrolysis [105], electrokinetic methods [106], and membrane filtration, are other methods that could be also used to treat oily water. In the next section the membrane filtration will be discussed in more details. In

addition, remediation methods are utilized to degrade the oily compounds from oily wastewater. The most commonly used remediation methods are chemical oxidation and enhanced oxidation processes [107–109], bioremediation processes [110–112], solid-phase processes [113–116], Bio-slurry processes [117], and incinerations [118–121]. Table 2 provides a comparison between various oily wastewater treatment techniques [10,53].

Table 2 Comparison of various techniques for oily wastewater treatment [10,53]

Technique	Status	Efficiency	Advantages	Disadvantages
Froth flotation	Lab scale	50-75%	Easy to apply, not energy intensive	Low efficiency, not suitable for oil sludge, unable to treat heavy metals
Electrokinetic	Lab scale	50-75%	Fast, efficient, no additional chemicals	Not easy to apply, low treatment capacity
Solvent extraction	Field scale	50-90%	Easy to apply, fast, efficient	High cost, massive organic solvent is needed unable to treat heavy metals
Centrifugation	Field scale	< 50-75%	Easy to apply, fast, efficient, no additional chemicals	High capital and maintenance costs, energy intensive, noisy, unable to treat heavy metals
Surfactant EOR	Field scale	75-90%	Easy to apply, fast, efficient	High cost, surfactants could be toxic, surfactants must be removed
Freeze/thaw	Lab scale	50-75%	Easy to apply, fast, suitable for cold regions	High cost, low efficiency, unable to treat heavy metals, energy intensive
Microwave irradiation	Field scale	> 90%	Very fast, efficient, no additional chemicals	High capital and operation costs, energy intensive, low capacity, unable to treat heavy metals
Pyrolysis	Field scale	50-90%	Fast, efficient, large treatment capacity	High capital and maintenance cost, energy intensive, not suitable for oily sludge with high moisture content

4. Membrane Treatment

Membrane can be defined as a selective barrier between two phases that restricts the transport of various chemical species [122]. The feed to the membrane is separated into two streams: the retentate and the permeate. Either the retentate or permeate could be the product stream. Membranes are usually synthesized from any material that can form stable thin films. Hence, polymers, ceramics, glasses, metals and monolayer liquids can be used as membranes [123,124]. Membrane separation depends mainly on three mechanisms, molecular sieving, electrostatic interactions and adsorption effects [125,10]. Membranes have several classifications mainly according to their structure, the material used for their fabrication and pore size [126]. Based on their structure, membranes can be either dense homogenous asymmetric membranes or porous membranes [127]. Most of the dense membranes nowadays are porous with a dense top film [128–130]. According to their material of fabrication, membranes can be organic, inorganic or hybrid [131]. Industrial organic membranes are manufactured mainly from polymers [132]. Inorganic membranes, on the other hand, can be either metallic, ceramic, zeolite or elemental carbon [133]. Inorganic membranes can bear high temperatures and pressures and they are very stable even under harsh environments [130,134]. Their permeability is significantly high compared to polymeric membranes with adequate selectivity [135]. However, their selectivity is greatly effected by the separation process variables such as temperature and pressure [136]. Due to the fact that inorganic membranes operate under harsh environmental conditions, they can endure the harsh washing processes used to remove fouling, hence, they are suitable for oil/water separations [137,138].

According their pore size, membranes could be macroporous (> 50 nm), mesoporous (2 – 50 nm), microporous (< 2 nm) or dense membranes (< 0.5 nm) [136,139,140]. Although, liquids are not able to permeate through microporous and dense membranes. However, they can transfer through mesoporous and macroporous membranes by viscous flow mechanism [141–143]. Table 3 shows oily water treatments using membrane technology compared to other techniques [9].

Table 3: Oily wastewater treatment using membrane technology and other techniques [9].

Technique	Treatment results
Flotation	Oil removal is more than 90%
Peeling flotation	Oil removal is 81.4%
Dissolved air flotation	COD removal rate is 92.5%
Dissolved air flotation	Oil removal is more than 90%
CAX Coagulation	Oil removal is more than 98%
Aggregation zinc silicate and anionic polyacrylamide Coagulation	Oil removal is 99%
Poly-aluminum zinc silicate chloride Coagulation	COD removal is 71.8%
Membrane bioreactor	COD removal is 97%
Upflow anaerobic sludge blanket	COD removal is 74%
Biological aerated filter reactor	COD removal is 97%
Yarrowia lipolytica W29 immobilized by calcium alginate	COD removal is 97%
UF	Oil content is below 1 mg/L
MF (Carbon membrane with pore size of 1.0 μm)	Oil removal is 97%
MF (NaA zeolite membrane with pore sizes of 1.2 μm)	Oil removal is 99%
MF ($\alpha\text{-Al}_2\text{O}_3$ membrane with 50 nm pore size)	TOC removal is 92.4%
Dynamic membrane	Oil removal is 99%
Nano-porous membrane	COD removal is 76.9%
Nano-porous membrane-powdered activated carbon	TOC removal is 71.5%

4.1 Membrane treatment processes

Membrane separation processes are primarily driven by concentration difference, temperature difference, electrical potential difference or pressure difference [144]. The pressure difference or pressure driven processes are classified according to the pore size of the utilized membrane to four processes. These four processes are microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) [145,146]. The main characteristics of pressure driven membranes are shown in Figure 4 [145,147] and Table 4. Taking pore size and the required pressure in

consideration, it is convenient to utilize the sieving effect of MF and NF membranes for oil/water separation [148]. According to their membrane pore size (2 – 50 nm), UF membranes are more efficient than MF membranes [149,150] and in spite the high permeate flux of MF, oil might squeeze through their relatively large pores with the permeate [151]. RO and NF membranes can be also used to separate oil from water especially for high salinity water [152]. Compared to the traditional separation processes, membrane processes are compact and able to achieve the desired separation without any chemical additives with lower energy requirements [149, 153,154]. However, their vital disadvantages are the low permeate flux, concentration polarization and fouling which lead to persistent decrease of the permeate flux [155–159]. Furthermore, polymeric membranes, in particular, possess a limited capability to separate volatile organic compounds (VOCs) from water [60] and are very sensitive to polar and chlorinated solutions [160].

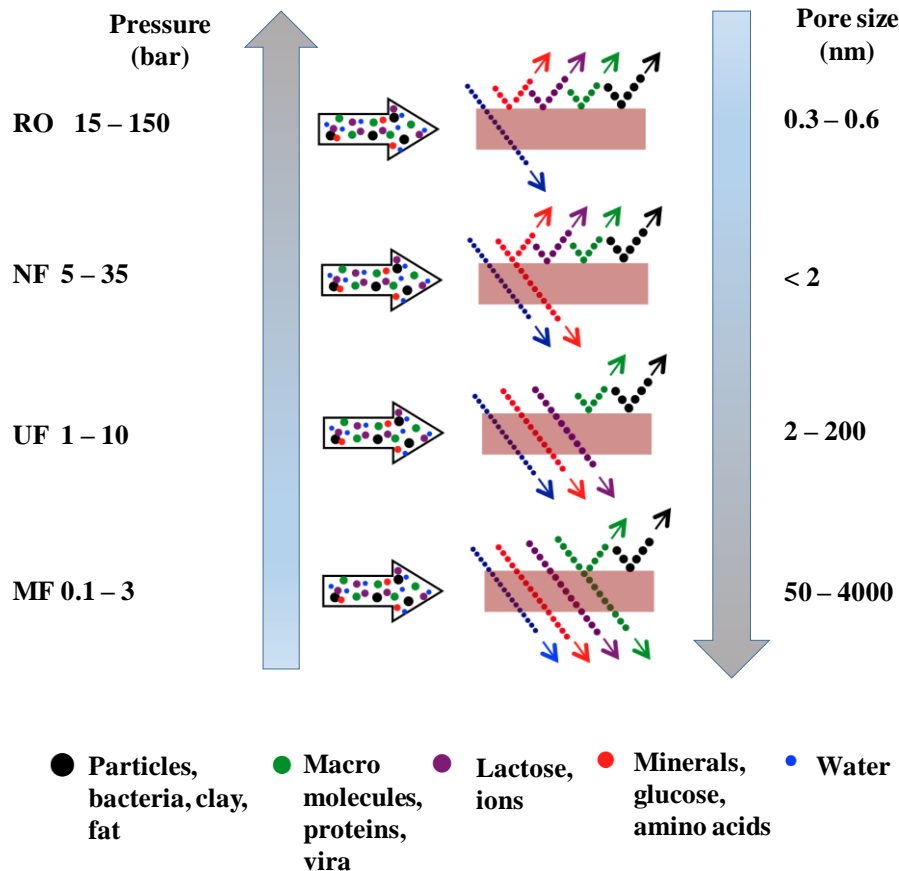


Figure 4: Membrane separation processes and its separation characteristic [145,147].

Table 4: Comparison between the four pressure driven membrane processes [16,145,160].

	MF	UF	NF	RO
Membrane	Symmetric	Asymmetric	Asymmetric	Asymmetric
Thickness (mm)	10 - 150	150 - 250	150	150
Thin film Thickness (nm)	-	1000	1000	1000
Pore size (nm)	50 - 4000	2 - 200	< 2	0.3 - 0.6
Applied pressure (bar)	0.1 - 3	1- 10.0	5 - 35	15 - 150
Flow Mechanism	Convective pore flow (Darcy's law)	Convective pore flow (Darcy's law)	between pore flow and diffusion	Solution diffusion (Fick's law)
Permeability (L/h.m².bar)	> 1000	10 - 1000	15 - 30	0.05 - 1.5
Rejection	Particles, bacteria, clay, fat	Macro molecules, proteins, vira, polysaccharides	Lactose, mono-, di-, oligosaccharides, ions, HMWC	Minerals, glucose, amino acids, HMWC, LMWC
Energy Consumption (kWh/m³)	0.4	3	5.3	10.2
Materials	Ceramic, PP, PSO, PVDF	Ceramic, PSO, PVDF, CA, thin film	CA, thin film	CA, thin film
Module	Tubular, spiral wound, plate and frame	Tubular, spiral wound, plate and frame	Tubular, spiral wound, plate and frame, hollow fiber	Tubular, hollow fiber

HMWC: High Molecular Weight Compounds, **LMWC:** Ligh Molecular Weight Compounds, **PSO:** Polysulfone, **PVDF:** Polyvinylidenedifluoride, **CA:** Cellulose acetate, **PP:** Polypropylene

Membrane processes are being applied for treating wastewater when possible and depending on the type of oily waste water. According to the oil dispersion, oily water can be classified to three main kinds, free-floating oil, unstable oil-water emulsions and stable oil-water emulsions. [161,162]. In free-floating, oil can be easily removed mechanically [31] and the oil from the

unstable emulsions can be removed either mechanically or by adding specific chemical additives [58,59]. However, it is quite difficult to remove the oil in stable emulsions using conventional methods since the size of the oil droplets is in microns. Stable emulsions are usually produced from a variety of industries such as, oil and natural gas exploration and processing, metallurgical industrials, textile manufacturing, food industries. Table 5 summaries the sources and nature of oil emulsions from various industries [9,152].

Table 5: Sources and nature of oil emulsions from various industries [9,152].

Sources of Oily Wastes	Industries	Nature/treatment
Petroleum oil from exploration, spills and tanks washing	Oil exploration and refining	Both free-floating and emulsion oils/difficult to treat
Alkaline/ acidic cleaning materials	Metal manufacturing and processing	High emulsion oils due to surfactants usage/difficult to treat
Floor washing	Use in most industries	A mixture of different types of oils: hydraulic and cutting fluids, along with paints
Cooling machines materials	Metal manufacturing and processing (e.g. steel and aluminum rolling)	Normally mixture of emulsions/difficult to treat
Natural fats and oils from plants and animals	Cooking oil, detergent industry, fish and leather processing	Both free-floating and emulsion oils/difficult to treat

Membrane separation efficiency is normally identified by the oil rejection coefficient (R_o) and it is defined as:

$$R_o = \frac{\text{oil concentration in feed} - \text{oil concentration in permeate}}{\text{oil concentration in feed}} \times 100 \quad (1)$$

Rejection coefficient is influenced by several variables. These variables include: the size distribution of the contaminations in the solution, operating conditions such as, temperature, trans-membrane pressure (TMP), pH, and cross flow velocity (CFV), capillary pressure of oil droplets in the membrane pores, and the membrane material surface characteristics such as, surface tension, surface energy, surface charge or polarity [163,164]. These characteristics are strongly connected to the surface adsorption capacity [165]. Usually, an effective membrane has high rejection coefficients for total organic carbon (TOC), total surface charge (TSC) and chemical oxygen demand (COD) [166].

Another important parameter in membrane separation is the permeate flux. Permeate flux is defined by:

$$J = \frac{V_P}{At} \quad (2)$$

where J is the permeate flux, V_P is the permeate volume, A is the membrane effective area, and t is the permeation time. The permeate flux is highly influenced by the operating conditions [164,167], in particular by the TMP and that is due to the concentration polarization. Normally the permeate flux increases by increasing the TMP until it reaches a maximum value. Due to concentration polarization, any further increase of TMP leads to decrease of the permeate flux. However, once the balance between the mass transfer flow to and from the membrane is achieved, the permeate flow reaches steady state [168–170]. The permeate flux depends also on the membrane properties such as the pore size, porosity and hydrophilicity [59,169]. Higher permeate flux is achieved using large pore size membranes, however, small pore size membranes provide a higher oil rejection coefficient and consequently improved demulsification efficiency [171], but demand a higher TMP [172].

As mentioned earlier in this context, membrane fouling is one of the major drawbacks of membrane separation processes which leads to persistent decrease of the permeate flux. The main cause of fouling is the pore plugging of membrane surface pores by the adsorbed components of the feed solution [173]. When compared to new membranes, fouled membranes are characterized by higher surface tension and lower hydrophilicity [174]. The lower surface tension is due to the adhesion of oil and/or surfactants to the surface of the membrane. The fouling is classified into

two types, reversible fouling and irreversible fouling. Reversible fouling can be removed by backwashing the membrane. However, irreversible fouling can't be removed by hydraulic washing methods [175]. Irreversible fouling can be further classified to hydraulically and chemically irreversible fouling. Chemically irreversible fouling persists even after using chemical cleaning methods [176].

4.2 Polymeric membrane

Polymeric membranes are low in cost compared to inorganic membranes and very efficient in oil/water separation [177]. However, oil and particulates accumulate within their pores leading to a significant reduction in their separation performance [178,179]. The most utilized polymers to prepare MF and UF membranes are polysulfone (PSO) [151], polyethersulfone (PES) [180], polyvinylidene fluoride (PVDF) [181,182], polyacrylonitrile (PAN) [183] and cellulose acetate (CA) [184,185]. However, due to the fouling propensity of these membrane, many modifications attempts were conducted to promote the antifouling and raise their hydrophilic characteristics. Oily compounds adhesion on hydrophobic surfaces is high, hence, fouling can be significantly reduced when hydrophilic membranes are employed. Enhancing the hydrophilicity of polymeric membranes can be achieved by two main methods. Either by chemically and/or physically modifying the membrane surface or through incorporating hydrophilic additives to the polymer structure [186]. Nevertheless, even after modification, the membrane performance is easily affected by the operating conditions such as temperature, trans-membrane pressure (TMP), pH, and cross flow velocity (CFV). Table 6 shows the main effects of various operating conditions [10,187] and Table 7 summarizes established polymeric membrane separation industries [188].

Table 6: Main effects of various operating conditions on membrane performance [10,187].

Operating condition	Main effects
Temperature	The higher the temperature, the higher the diffusion rate and the mass transfer rate
Trans membrane pressure (TMP)	The higher the TMP, the higher the driving force
pH	Affects the membrane material

Cross flow velocity (CFV)	The higher the CFV, the lower the concentration polarization and the higher the permeate flux
---------------------------	---

Table 7: Established polymeric membrane separation industries [188]

Process	Selective layer type	Structure	Selective layer Thickness (μm)	Polymer
MF	Macroporous	Symmetric	6 – 35 50 – 300 50 – 500 100 – 500	Polyethylene terephthalate Polycarbonates, aromatic Cellulose acetates Polyethersulfones Polyvinylidene fluoride Polytetrafluoroethylene Polyethylene Polypropylene Cellulose nitrate Polyamide, aliphatic
UF	Mesoporous	Asymmetric	~ 0.1	Cellulose acetates Cellulose, regenerated Polyacrylonitrile Polyetherimides Polyethersulfones Polyamide, aromatic Polysulfones Polyvinylidene fluoride
NF, RO, GS	Nonporous	Asymmetric	< 0.1	Cellulose acetates Polyphenylene oxide Polytetrafluoroethylene Polyamide, aromatic, in situ synthesized Polycarbonates, aromatic Polyimides Polysiloxanes Polysulfones

ED	Nonporous	Symmetric	50 – 500 100 – 500	Perfluorosulfonic acid polymer Poly(styrene-co-divinylbenzene), sulfonated or aminated
PV		Asymmetric /composite	< 1 – 10	Polysiloxanes Polyvinyl alcohol, crosslinked

Several improvements are required in order to elevate polymeric membranes economical and technical feasibility. The main improvements are increasing the separation efficiency, decreasing membrane and reinforcing the chemical resistance of the membrane [189]. These improvements are achieved through several preparation and/or modification techniques. Furthermore, improving the membrane against fouling, in particular, received a major interest. Hence, several preparation and/or modification techniques to enhance the membrane fouling resistance were investigated in the literature. Membrane tendency for fouling is strongly related to its hydrophilicity. Hence, improving membranes hydrophilicity reduces their fouling significantly [190,191]. Membranes Hydrophilicity can be improved via several physical and chemical methods. Examples of these methods are interfacial polymerization [192], nanoparticles incorporation [193], and surface grafting. The latter can be initiated by ultra-violet (UV) irradiation [194], electron beam irradiation [195], plasma treatment [196], layer-by layer modification [197], or Grignard and phosphoric acid grafting [198]. Interfacial polymerization, nanoparticles incorporation and surface grafting methods will be further discussed in the next sections.

4.2.1 Interfacial polymerization

Interfacial polymerization (IP) technique was invented by Emerson Wittbecker in 1959 [199]. IP was commonly used to prepare polyamide nanofiltration membranes [200,201]. However, it can be used to prepare polyurethanes, polysulfonamides, polycarbonates, and polyesters. In IP, the polymerization reaction occurs at the organic side and near the interface between the immiscible aqueous/organic solutions [202,203]. This is due to the fact that the solubility of the monomer is good in the organic phase and negligible in the aqueous phase [204]. This difference in solubility created a chemical potential difference, which consequently, drives the monomers diffusion to the interface, hence, initiating the polymerization reaction there [205]. The main advantage of IP is

the self-inhibiting of the polymerization reaction caused by the created thin film. This film acts as a barrier, thus, hindering the supply of reactants and producing a substantially thin film of thickness between 50 nm and 100 nm [206]. The thin film thickness, porosity, pore size composition, and thermal properties are determined by the several factors. These main factors are the solvents nature, the monomers type, the monomers concentration in both the aqueous and the organic phases, the reaction time, and the reaction temperature [192,207,208]. Moreover, there are certain additives such as, inorganic salt, carbon nanotubes and silica and titania nanoparticles, that can be added to the aqueous phase. These additives play a remarkable role in enhancing the monomers dissolution and diffusion rates, hence enhancing the performance of the prepared membranes [209–213]. The prepared membranes eventually showed significant improvements for both the rejection capacity and the antifouling capability.

4.2.2 Nanoparticles incorporation

Nanoparticles incorporation (NI) is another technique that is utilized to improve the membrane fouling resistance. In this technique, nanometer-sized particles are incorporated within a polymeric membrane structure. This incorporation affects the characteristics of the original polymeric membrane such as, permeability, selectivity and hydrophilicity [214]. Hence, influences the performance of the prepared membrane. Recently, numerous types of nanoparticles were incorporated into polymeric membranes. The most commonly incorporated nanoparticles are silica [215], zirconium dioxide (ZrO_2) [216], titanium oxide (TiO_2) [217], ferric oxide (Fe_3O_4) [218], hydrous manganese oxide ($MnO_2 \cdot nH_2O$) [217], iron (II) [219], graphene oxide (GO) [220], carboxylated graphene oxide (cGO) [221], carbon nanotubes (CNT) [216], polyethylene glycol-functionalized polyhedral oligomeric silsesquioxane (PEG-POSS) [222], and polymeric nanoparticles [223]. Typically, one or more of these nanoparticles types may be incorporated into any of the known polymeric materials. The properties of the prepared membrane are the result of the interactions between the nanoparticles and the polymeric material. Therefore, the specifications of the sought membrane rely on the amount, size and type of the incorporated nanoparticles [224]. The incorporation of the nanoparticles into polymeric membranes can be done mainly via in-situ solution-casting [225,226], immersion coating [221], aerosol assisted chemical vapor deposition (AACVD) [227] and sol-gel syntheses of inorganic nanoparticles inside the pores of the membranes [228]. In spite the low cost, the relatively simple preparation, and mild operating

condition, this technique, experience a major disadvantage. This disadvantage is the aggregation and leaching of the incorporated nanoparticles [229]. The nanoparticles leaching reduces the antifouling resistance gradually with time. Table 8 shows several polymeric membranes with different ceramic fillers [230].

Table 8: Polymeric membranes with different ceramic fillers [230]

Filler	Polymer	Type	Syntheses	Improvements
Alumina	PPY	UF	Phase inversion	Adsorption capacity
	PES	MBR	Phase inversion	Hydrophilicity
Silica	PES	MF	Vapor induced phase separation	Hydrophilicity
	CA	UF	Phase inversion	Hydrophilicity
	PVDF	UF	Thermal induced phase separation	Hydrophilicity
Titania	PVDF	-	Directional melt crystallization	Hydrophilicity
	PVDF	UF	Solution casting/ Phase inversion	Photo-catalytic activity
	PVC	UF	Non-solvent induced phase separation	Hydrophilicity
	PES	UF	Phase inversion	Hydrophilicity
Zirconia	PES	MBR	Phase inversion	Hydrophilicity
Clay	PVDF	UF	Phase inversion	Hydrophilicity Mechanical properties

4.2.3 Surface grafting

As mentioned earlier, surface grafting is usually initiated by UV irradiation, electron beam irradiation, plasma treatment, layer-by layer modification, or Grignard and phosphoric acid grafting. UV irradiation is one of the most attractive techniques for surface grafting to prepare polymeric membrane with high ability for antifouling [157,228]. This is primarily because it is simple to operate, requires low cost, involves fast reactions, easy to commercialize and the graftation is restricted to small region adjacent to the surface [231]. UV grafting may take place

with or without photo initiators. Commonly, photo initiators are used to initiate the grafting reaction. The photo initiators react with polymer membrane under UV irradiation to produce the free radicals. These photo initiators are either dissolved in the monomer casting solution [232] or adsorbed on the membrane surface [233]. The most commonly used photo initiator is benzophenone (BP) [234] and BP-based compounds [235]. Moreover, there are numerous compounds often used as photoinitiators. Examples are xanthone (XAN), benzoyl peroxide (BPO), vinyl acetate (VAC), (4-benzoyl benzyl) trimethylammonium chloride (BTC), isopropylthioxanthone (ITX), 2,2 -azo-bisisobutyronitrile (AIBN), anthraquinone (AQ), anthraquinone-2-sulfonate sodium (AQS) and 4,4 - bis(diethylamino)-benzophenone [233,236-238],

Dissolving the photo initiator in the monomer solution usually produce low local concentration of the photo initiator on the membrane surface and a high photo initiator concentration in the bulk. The former leads to low grafting efficiency while the latter promotes homopolymerization [228]. This can be avoided by increasing the photo initiator concentration at the surface through adsorption [239]. Hence, the bulk concentration of the photo initiator decreases, and consequently, the homopolymerization is declined.

The UV grafting can be also initiated without a photo initiator. In this case, the free radicals are directly produced from the polymers under the UV irradiation [240]. Two techniques are typically used for membrane modification, dip and immersion techniques [241]. During the immersion technique the UV-irradiation takes place while the membrane is immersed in the monomer solution. On the other hand, in the dip technique, the UV irradiation started after dipping the membrane the monomer solution. In this case, the UV irradiation occur under inert atmosphere. Usually, dipping technique resulted in 2 to 3 times more grafting than the immersion technique.

Plasma grafting is ecofriendly and suitable for grafting thin membrane surfaces when operated at pressures less than 100 Pa with a very high frequency of around 13.66 MHz [242,243]. This technique employs gas (eg. He, O₂ and N₂) or vapor (eg. H₂O and NH₃) reactants [244–246], hence, it is able to treat the membrane surface in a short treatment time [242]. He and H₂O are economically viable and produce adequate functional bonding [247]. In this technique the free radicals' generation is initiated by plasma induction [248,249]. These radicals are stable in

vacuum, however, they are very active and can react very fast when they are in contact with gaseous or solution monomers. The main parameters that determine the grafting quality are the plasma operating condition such as power, pressure and reactant gas, and polymerization conditions such as grafting time and monomers concentration [157,250]. Plasma grafting can be accomplished in several approaches. It can be done in solution, in vapor phase or in plasma reactor. Experiments performed on PSO showed that grafting in vapor phase had the highest flux [251].

Plasma grafting has many advantages such as improving the regeneration properties for hydrophobic membranes [252], it requires a very short time and it can modify the membrane surface without affecting its bulk. However, it is hard to scale up from laboratory scale to industrial scale and the treatment is highly influenced by the system parameters experience a lack of reproducibility [157]. Figure 5 shows a schematic of plasma reactor used to treat asymmetric PSf membranes [246].

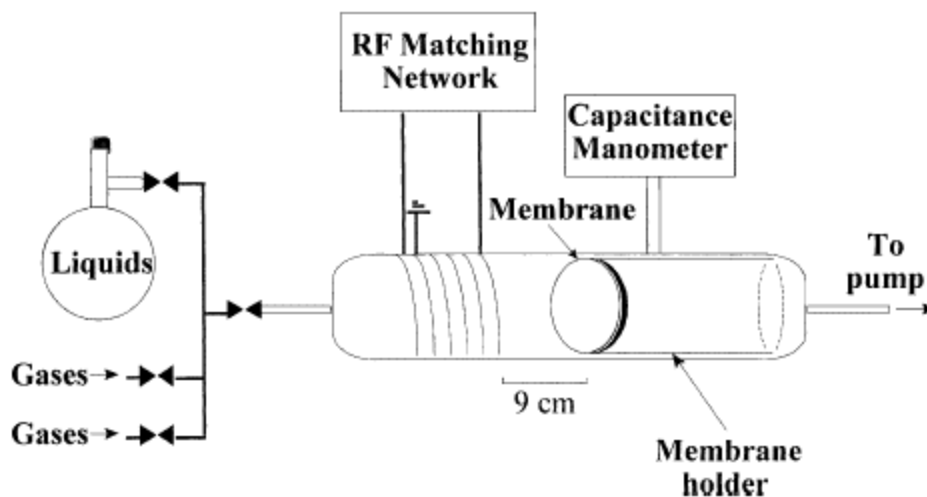


Figure 5: Schematic of plasma reactor used to treat asymmetric PSf membranes [246].

Electron beam (EB) irradiation is also used to initiate surface grafting and modify the membrane antifouling resistance [253,254]. This technique utilizes high energy beta radiation to generate free radicals. These free radicals react with the solution monomers, hence, they boost the changes in polymer-matrix and modify the polymers cross linking [255]. It is a very effective and ecofriendly technique, since a high energy electron beams is used and no strong additive chemicals or catalysts are required to achieve the membrane modification [256]. The monomers are not exposed directly

to the radiation beam in this technique, hence, less homopolymerization is anticipated. Several grafting monomers have been investigated in literature such as, 4-styrenesulfonate [257], styrene [258] and Nisopropylacrylamide [259]. Moreover, EB irradiation is also used to graft zwitterionic molecules on polymers surfaces and on hollow fiber membranes. Grafting these zwitterionic molecules lowers the membrane fouling effectively [260–262]. The nature of the grafted monomer has a great influence on the uniformity of the formed chains, hence the efficiency of the grafting. Nevertheless, a possible drawback may result from a high degree of grafting [253]. This usually leads to long straight chains which in turn may plug the membrane pores, hence, lowering the membrane permeability.

4.3 Inorganic membranes

As mentioned earlier in this context, inorganic membranes are mainly metallic, ceramic, zeolite and carbon based. They are generally prepared by solid state [263], sol-gel [264], chemical vapor deposition (CVD) [265], slip casting [266,267], freeze casting [268], tape casting [269], dip coating [270], pressing [271], extrusion [271,272], and atomic layer deposition [273]. Several types of inorganic membranes are utilized for water treatment and desalination, however, ceramic membranes, in particular, have received a great attention. The ceramic membranes that are mostly used in water treatment and desalination are alumina, silica, titania, zirconia, zeolite and silicon carbide [274–279]. Nevertheless, the production cost of these membranes is very expensive, hence, cheaper raw materials were utilized to lower the production cost. These raw materials include clay [280], quartz sand [281], fly ash [282] and kaolin [283].

4.3.1 Ceramic membranes

Ceramic membranes are prepared in two different structures: asymmetric and symmetric. In symmetric structure, the size of the pores is equal and the membranes' properties are uniform throughout the cross section of the membrane [284]. On the other hand, asymmetric membranes are composed of a layered structure. In this structure, a thin selective layer is prepared on top of strong support layers. The top thin layer is the selective layer with a small pore size. The thin layer is supported by a strong support layer to provide the required mechanical strength to restrain the collapse of the membrane during operation [285]. In some cases, intermediate layers are required

to bridge the gap between the small pores of the selective layer and the large pores of the support layer [286]. The pore size is gradually increasing through the intermediate layers up to the bottom support layer [123]. Nevertheless, the pores of the support layer and intermediate layers are orders of magnitude larger than the pores of the selective layer [287]. Hence, these layers afford no significant separation with a least flow resistance. Asymmetric membranes are superior compared to symmetric membranes because the flux is determined by the thickness of the top thin selective film and not by the whole membrane thickness [288]. Most ceramic membranes have an asymmetric structure with either a dense or a porous active layer [289]. Table 9 summaries various types, materials, fabrication technique and Configuration of inorganic membranes [123].

Table 9 Types of ceramic membranes [123].

Type	Structure	Materials	Technique	Configuration
Single wall	Asymmetric	Alumina	Anodic oxidation	Sheet, tube, monolith
Single wall	Symmetric	Alumina Silica	Anodic oxidation Track etch	Sheet Tube
Multilayered	Asymmetric	Alumina, titania, zirconia, ceramic-metal, ceramic-ceramic	Dip coating, slip casting, sol-gel	Disk, tube, monolith
Modified structures	Asymmetric	Ceramic-ceramic ceramic-organic	Pore plugging, intra pore deposits, intra pore coating	Disk, tube, monolith

Ceramic membranes are typically classified according to their pore size to macroporous, mesoporous and microporous. Macroporous membranes have a pore size greater than 50 nm and are utilized usually as supports in composite membranes. They have high permeability but with no

notable functional selectivity. Hence, they are used mostly in MF and UF applications [290]. Mesoporous membranes have a pore size between 2 and 50 nm, hence, they provide high permeability with very low selectivity. Consequently, they are used in the preparation of composite membranes and they are used mainly in NF [291]. Microporous membranes have a pore size less than 2 nm, hence, they can separate mixtures using the molecular sieving mechanism. They can provide a very high selectivity, however, their permeability is very low. Microporous membranes are used usually for NF and gas separation (GS) [292,293].

Nonporous membranes or dense membranes consist of a dense film through which the permeants are transported by diffusion. The separation in nonporous membranes occur through the solution-diffusion mechanism. Solution-diffusion mechanism, consists of three steps: firstly, the molecules are dissolved into the membrane or absorbed at the membrane surface on the feed side. Secondly, the dissolved molecules diffuse through the membrane material. The third step involves desorption of these molecules on the permeate side [294]. In this mechanism, the separation is achieved between the different permeants due to the differences in their solubility coefficients and diffusivity coefficients in the membrane material. Consequently, nonporous membranes can separate components of similar size if their solubility in the membrane material is significantly different [295]. Nonporous membranes are employed mainly in GS applications and they provide high selectivity but their permeabilities are usually low [296]. Table 10 shows the pore size, permeation mechanism, applications, and advantages and disadvantages for each of these membranes [297].

Table 10 Classification of ceramic membranes based on their pore size [297].

Porous Membrane	Pore size (nm)	Permeation Mechanism	Applications	Advantages/ disadvantages
Macroporous	> 50	Poiseuille flow	MF and UF	High permeability/ no functional selectivity
Mesoporous	2 – 50	Knudsen flow	UF and NF	High permeability/ low selectivity

Microporous	< 2	Molecular sieving	NF and GS	Low permeability/ high selectivity
Nonporous	–	Solution-diffusion	GS	Low permeability/ high selectivity

4.3.2 Preparation techniques of ceramic membranes

Most of the ceramic membranes have asymmetric structure, in other words, they consist of several layers. Ceramic membranes possess several geometric configurations such as flat, tubular, monolith, capillary and hollow fiber. Despite the geometric configuration, ceramic membranes prepared by three main steps. The first step is the preparation of the particles paste or suspension. In this step the ceramic material mixed with proper additives to stabilize the suspension and enhance the membrane microstructure characteristics. Examples of these additives are sintering additives, plasticizers, lubricants, binders, dispersant and antifoaming agents [298,299]. Sintering additives are used to lower the sintering temperature, in particular, for alumina membranes. Some of these additives are magnesium oxide, manganese oxide, copper oxide and titanium oxide [300-302]. Plasticizers are used to enhance the rheological characteristics, and improve demixing resistance of the suspension, facilitate the handling and sustain the membrane configuration and shape [228,303,304]. Methocel [305], sodium silicate [306], triethanolamine and triethyleneglycol [303] are examples of common plasticizers.

The second step is the forming the suspension or the paste into the desired shape, flat, tubular, monolith, etc. The final step is the heat treatment step through sintering and calcination. This step is the most important where the membranes are heated to their sintering temperature, then cooled to the room temperature [279, 307]. Low heating and cooling rates are especially used throughout the critical temperature region, where the organic additives decompose and burn [287]. The low heating and cooling rate is necessary to prevent defect formation during the heat treatment [308,309]. Removal of the organic additives is associated usually with shrinkage of the membrane

crystallites. This leads to creation and/or enlargement of the intercrystalline pores [310]. In the case of zeolite films, the heat treatment removes the templates from the zeolite pores and strengthens the bonding between the zeolite film and the support. However, it leads to compressive stresses especially during the cooling period [311]. Additional heat treatment is required for the membrane modifications such as depositing extra layer, controlling the pore size distribution or tuning other membrane properties [312]. Ceramic membranes are prepared by several methods and they are summarized in the following sections:

- Sol-gel

Sol-gel is an attractive process for the preparation of ceramic membranes. It is simple and easy to adopt and it allows a firm control of the compositions, pore structure, pore size, pore size distribution and catalytic activity of the membrane [313]. It also allows the deposition inside the pores of porous supports, preparation of multilayer thin film coatings and it is also used to prepare inert and catalytic mesoporous and microporous membranes [314]. Most of the membranes prepared by sol-gel technique were either thin films on top or inside the pores of a porous support [315-317]. Sol-Gel involves two main routes: The colloidal route and the polymeric route. For both routes the precursor is typically a metal alkoxide. The major difference between the two routes is that in the colloidal route the precursor is dissolved in aqueous solvent to form a colloidal sol while in the polymeric route the precursor is dissolved in organic (alcohols, aldehydes or ketones) to form a sol [314,318]. After coating, the sol is coated on the porous support, then dried and sintered at the proper temperature. Due to the difference in the solvent type, the colloidal route produces mesoporous membranes while the polymeric route produces microporous membranes [319]. Furthermore, the sol-gel method was employed to prepare alumina [320], silica [321], titania [322], and zirconia [323] ceramic membranes.

- CVD

This technique is attractive for deposition of very thin solid films with high purity on the surface of solid porous substrates. In CVD, the precursor stream consists of a single gas or a mixture of gases and the substrate is placed inside a heated chamber. The precursor constituents react or decompose

on the surface of the substrate leading to the aimed deposition. Basically, CVD can be performed by two distinct approaches. The main difference between these two approaches is the way of supplying the precursor constituents to the substrate [324]. In one approach, the precursor constituents are supplied to the side of the substrate where the thin film is to be deposited and a vacuum is applied on the other side. In the other approach, instead of the vacuum, another precursor stream is supplied to the opposite side of the substrate. The reactions products taken out of the chamber by a flowing gas. The main factors affecting the deposited film composition, thickness and the pore size distribution inside the film are the type of the gases in the precursor stream, their concentration, temperature and pressure inside the reaction chamber [325].

There are several advantages associated with the CVD technique such as the flexibility and huge variety of possible precursors and consequently enormous choices of film materials and compositions, the easy control of the prepared film composition, morphology and orientation, the easily controllable deposition rates, the reasonable reproducibility, the ability to produce uniform coatings with good coating coverage and to coat substrates with complex shapes, the operation at high temperatures which eliminates the heat treatment step, hence, prevents defect formation and shrinkage of the membrane crystallites [326,327]. However, CVD suffer from several disadvantages such as the raw materials high cost, the difficult control of the multicomponent materials deposition, the possibility of film hydrocarbon contamination, and the hazard effects caused by the use of explosive, flammable, toxic and corrosive gases [327]. Nevertheless, CVD techniques were used to prepare silica [328], silica-alumina [329]. Alumina-zirconia [330], and Silicon carbide–titanium carbide [331] composite membrane. Figure 6 shows a schematic illustration of the key CVD steps during deposition [327].

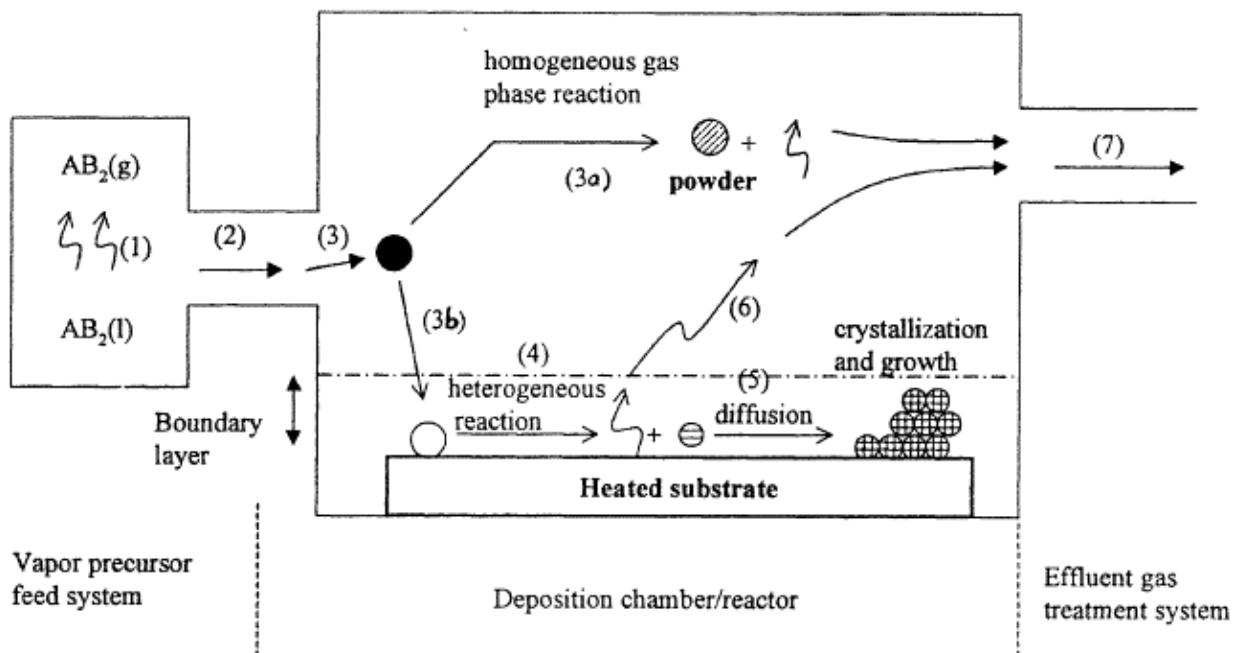


Figure 6: A schematic illustration of the key CVD steps during deposition [327].

- Slip Casting

Slip casting is the most common preparation technique for membrane synthesis [332]. It is simple and can be employed economically for the preparation of inorganic tubular and ceramic hollow fiber ceramic membranes [333]. In this technique, the powder suspension is mixed and poured on the surface of porous substrate (or mold), the solvent penetrates through the pores of the substrates via capillary forces [334]. Hence, the suspended particles form a gel layer which is deposited on the surface of the substrate resulting in a dense film. This should be followed by quick consolidation to prevent the particles penetration inside the substrate pores, hence, clogging them [335]. The art of slip casting process depends mainly on the permeability of the porous substrate and the extent of the capillary pressure [336].

Slip casting major advantages are the homogenous structure of the deposited film and its remarkable adherence with the porous substrate. The latter allows the membrane to operate under high back pressure [337]. However, the main drawbacks of this technique are the prolonged casting time, the difficulty in controlling the thickness and uniformity of the deposited film, and the mold

breakage after few casts which, hence, new molds are required which raise the production cost [338]. Numerous materials such as alumina [285], titania [275], zirconia [339], and perovskite [340] have been utilized in slip casting technique to fabricate MF and UF ceramic membranes.

- Freeze casting

Freeze casting or ice-templating is a new technique that received a great attention in the last decade for the preparation of porous ceramic membranes. This is mainly because this technique is able to produce hierarchical ceramic membranes with a very high porosity [341]. This technique consists mainly of two steps: freezing step and drying or sublimation step. The freezing takes place at the bottom of the suspension or slurry and followed by the sublimation step [342]. Both freezing and sublimation take place at low pressures and temperatures. Freezing and sublimation steps repeated sequentially to control the vertical growth of the crystals toward the course of the freezing [343]. The suspension consists mainly of ceramic powder, solvent, and additives. The pore shape usually depends on the type of the used solvent and the most common solvents are water [344], tert-butyl alcohol [345] and camphene [346]. The additives include dispersants to enhance the stability and the viscosity of the suspension, binders, to improve the mechanical strength, antifoaming to eliminate any air bubbles and increase the homogeneity of the suspension [347]. A possible approach to eliminate the air bubbles is by using vacuum desiccator before starting the freezing step [348]. The freezing and sublimation steps produce membranes with poor mechanical properties. However, these properties can be prominently improved after the heat treatment step [349].

The main factors affecting the microstructure of the prepared membrane are the constituents of the suspension and the freezing operating conditions such as temperature, pressure, direction and velocity [350,351]. Freezing techniques can be used to prepare membranes not only from most of the ceramic materials but also from some metals. Examples of these materials are alumina [352], titania [353], yttria-stabilized zirconia [354], nickel oxide–yttria stabilized zirconia [355], perovskite [356] and metals [357].

- Tape Casting

This technique is used to prepare thin flat ceramic sheets. It consists of casting tape, casting knife, moving carrier and casting reservoir [338]. The suspension is added to the reservoir and the thickness of the produced film is controlled by the distance between the casting knife and moving carrier. The casting suspension consists of ceramic powder, organic solvent, dispersant, binder and plasticizer. After the casting, prepared film kept on the moving carrier and passed through a drying region where the evaporation of the organic solvent occurs [358]. The drying should occur at temperatures below the boiling point of the solvent and consequently, higher drying rate [269,359]. Moreover, cautious control of the drying process is essential, otherwise, cracks generate in the casting tape [269,360]. The main factors determining the film characteristics are the distance between the knife and moving carrier, the viscosity of the suspension, the depth of the slip reservoir, the contact angle and the quality of casting tape [361]. A schematic diagram of tape casting process using a doctor blade is shown in Figure 7 [358].

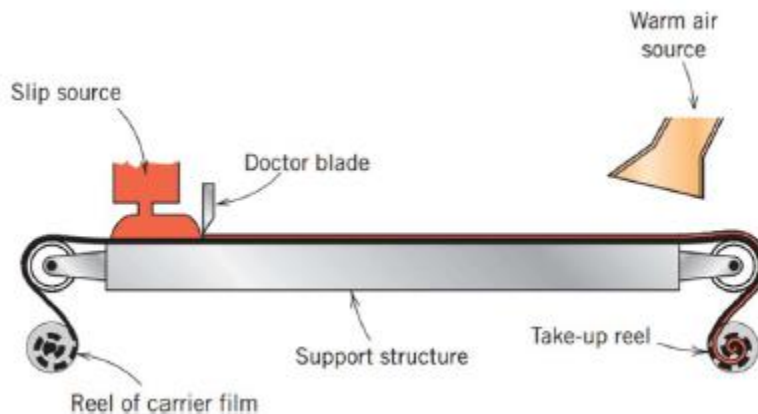


Figure 7 Schematic diagram of tape casting process using a doctor blade [358].

The membranes prepared by tape casting are characterized by their homogenous microstructure throughout the whole thickness, and their thickness is few millimeters with smooth surface finish [362]. Nevertheless, the main drawbacks of this technique are the corrosion of the mold which leads to poor precision of the produced shape, the health and environmental impacts due to solvent evaporation, and the very long casting time especially in fine powder casting. Several researchers

have addressed these drawbacks and these efforts resulted in a significant increase of the production cost [363-365].

The tape casting was used to prepare MF alumina [366,367], UF zirconia [368], silicon nitride [369,370], and Kaolin [371] ceramic membranes. It was also used to prepare MF metal membranes [372]. Despite that the cost of the hydrophobic kaolin membranes were very low, they showed excellent performance in treating oily waste water [371].

- Dip Coating

This technique is used to prepare very thin selective films with high quality for asymmetric membranes. It consists of five paces, substrate immersion into the sol solution, then the startup of the sol attachment to the substrate due to the capillary suction. This is followed by the deposition of the sol on the surface of the substrate during the substrate withdrawal from the solution. The deposition leads to the drainage of excess liquid from the surface of substrate and simultaneous evaporation of the solvent, and subsequently, a formation of a gel layer on top of the support surface [373].

The main factors that control the thickness and morphology of the prepared film are the speed of the substrate withdrawal from the solution, the solid volume fraction, and with viscosity, density, surface tension, pH and temperature of the dip coating solution [374-376]. The major ones are the withdrawal speed and the sol viscosity. As the withdrawal speed and the viscosity increases, the thickness of the film increases [377,378]. For instance, for silica membranes preparation, the typical withdrawal speed is in the range of 1 to 20 cm/min and the dilution with ethanol is up to 20 times the original solution volume [230,379]. Thicker films are inclined to form cracks during the solvent evaporation, hence, it is favorable to operate at low withdrawal speeds along with low viscosity sols [230,378]. Typical thickness of dip coated films is between 0.1 and 100 μm . Dip coating technique was used to prepare several types of ceramic membranes such as alumina [380], silica [381], titania [382], zirconia [383] and zeolite [384] membranes.

- Pressing

This technique is used to prepare MF and UF supported and non-supported ceramic membranes for oil/water separation. A high pressure in the range of 10 to 100 MPa is applied to the surface of the raw material powder to prepare symmetric membranes [230]. These membranes are in flat disk shape of few centimeters in diameters and their thickness is in the range of 0.5 to 1 mm [271]. The microstructure of the prepared membranes is controlled by mixing raw materials with inorganic and/or organic pore formers such as calcium carbonate and starch [271,385]. Typically, this method yields quite uniform films with high quality membranes, it utilizes a variety of raw materials and especially inexpensive materials and allows the formation of multilayers membranes [386]. However, this process is batch in nature, which limits the production rate and produced membranes size [387]. Hence, it is restricted to laboratory scale production. It is very difficult to control the thickness to diameter ratio in this technique. Examples of materials employed to prepare ceramic membranes are alumina and kaolin [388], clay [269] YZX [389], and natural zeolite [390].

- Extrusion

Extrusion is similar to pressing since a high pressure is applied to force the raw material paste through a nozzle. Hence the resulted membranes will be in tubular shape. Beside the ceramic powder, the paste consists of an organic solvent, a plasticizer, a dispersant and a binder. After the extrusion these constituents should be removed by drying and then heating the membranes to high temperatures up to 600°C before sintering [391]. This technique allows a continuous production and an adequate control of the shape, pore size distribution and porosity of the prepared membrane [391,392]. The former, endorse the mass production and consequently, lower the production cost. Extrusion method was used to prepare MF membranes using alumina [393], clay [271,272], cordierite [394], and Moroccan Perlite [395].

- Atomic Layer Deposition

Atomic layer deposition (ALD) technique is based on alternating pulses of two vapor precursors that are separated from each other. Since both precursors are in vapor phase, hence, they are easily adsorbed on the pore walls of the substrate and the latter adsorbed precursor reacts with the formerly adsorbed one [396]. Furthermore, ALD is a gas phase self-limiting deposition technique that operate on cycles [397]. Each cycle consists of four steps, firstly, exposing the substrate surface to the first precursor, followed by evacuating the reaction chamber by purging an inert gas, then, exposing the substrate surface to the second precursor, and finally, evacuating the reaction chamber again by purging an inert gas [398]. Thus, each cycle produces a monoatomic thick film and these cycles can be reiterated until the desired film thickness is achieved. Moreover, the thickness of the deposited film can be precisely controlled by solely repeating the deposition cycle [399]. ALD is utilized primarily for adding functional groups to porous materials and for the purpose of pore size reduction [397]. It is employed mainly to reduce the pore size for gas separation membranes. It is also used in liquid separation membranes for pore size reduction to sub angstrom scale, and for improving their hydrophilicity and enhancing solvent resistance [400]. A schematic diagram of the ALD chamber with a tubular membrane inside is shown in Figure 8 [397].

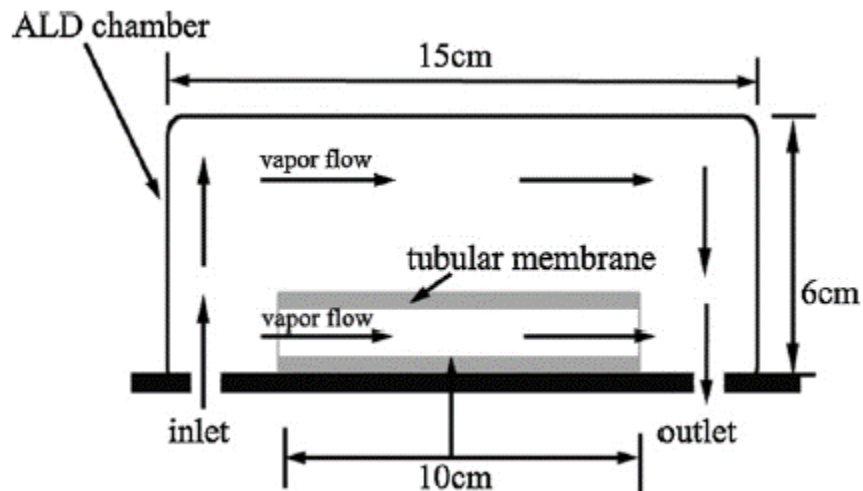


Figure 8: A schematic diagram of the ALD chamber with a tubular membrane inside [397].

ALD technique is used to prepare films that are highly uniform and conformal [401]. It possesses several superior advantages. ALD controls the film thickness precisely and it can operate with large areas and at low temperatures. Moreover, the process can be scaled up easily with outstanding reproducibility. The gas phase reactions are completely restricted in ALD and it is capable of depositing different raw materials for the preparation of multilayer films in a continuous manner [402]. ALD technique was used to reduce MF zirconia membrane by depositing alumina layer with Trimethylaluminum as a metal precursor and deionized water as an oxidant [397]. It was also employed to reduce the pore size of alumina tubular membranes by depositing silica and titania layers [403]. The alumina pore diameter reduction was in the rate 1.3 Angstrom per cycle when silica was deposited and 3.1 Angstrom per cycle when titania was deposited. ALD was successfully utilized in subsequent deposition of alumina and zirconia layers [404].

4.3.3 Ceramic membranes for oily water separation

Recently, numerous studies have been conducted to develop new ceramic membranes for oily water separation. Ceramic membranes possess several advantages when compared to polymer membranes. These advantages mainly the homogenous and narrow pore size distribution, the high mechanical, chemical and thermal stabilities, and weak bonding between the fouling substances and their surface [405]. Consequently, ceramic membranes can withstand harsh operating conditions, they operate under high temperatures and pressures, and endure high acidic and alkaline environments [406]. Moreover, ceramic membranes are durable and can resist corrosion, abrasion, bacteria and fouling [407]. Hence, they are easy to regenerate and can achieve very high flux and backwashing efficiency [408]. Due to the high chemical stability, they can withstand wastewaters with excessive oil content and the cleaning with strong chemicals, thus incessant performance can be guaranteed [10,409]. Nevertheless, ceramic membranes main drawbacks are the high initial cost, the heavy weight and the low area to volume ratio. This is usually compensated by the long service life. Table 11 summarizes the key advantages and disadvantages of ceramic membranes as compared to polymeric membranes [410].

Table 11 Advantages and disadvantages of ceramic membranes as compared to polymeric membranes [410].

Advantages	Disadvantages
<ul style="list-style-type: none"> - Very high flux - High mechanical, chemical and thermal stabilities - Long term stability under high temperatures - Lower tendency for fouling and easy cleaning after fouling - Operate under high pressures and resist high pressure drops - Withstand harsh chemical environment, resist corrosion and abrasion and Inert for microbiological degradation - Easy catalytic activation 	<ul style="list-style-type: none"> - High production cost - Low area to volume ratio - Brittleness - Low selectivity in large scale microporous membranes - Low permeability of highly selective dense membranes - Sealing is a challenge

Oily water is typically treated using MF [411], UF [412], and NF [413] ceramic membranes. MF membranes were also used to separate oil, grease and suspended solids from produced water [269]. The most utilized materials for ceramic membranes preparation are alumina, titania, silica and zirconia or zirconia based membranes [274–277]. Clay membranes were investigated for oily produced water purification [414–416]. It was evident that clay membranes are not suitable to treat produced water with high concentration of total dissolved solids [417]. Zeolite membranes which possess three-dimensional well defined microporous structure were also considered for oil/water separation [418,419]. Moreover, zeolite membranes represent a favorable alternative to separate distinct ions from oil field waste water through RO [420,421]. However, they suffer from serious

drawbacks when utilized for produced water separation. These drawbacks include the low permeability, the performance decay, the sensitivity to the flow variation, the modest predictability of the membrane life and the relatively high cost [422,423]. Hence, their industrial scale use for desalination is limited. Different ceramic membranes techniques for produce water treatment is presented in Table 12. And Table 13 presents typical operating conditions for oily wastewater ceramic membranes [424]

Table 12: Produced water treatment by different ceramic membranes [230]

Material	Type	Flux (L/hr/m ²)	Removal efficiency
Alumina	MF/UF	118 - 125	Oil and turbidity 99%, TSS 100%
α-Alumina	MF	250	Oil 95%
Alumina/ titania	MF/UF	3.4 -3300	Oil 99.5%, TOC 49%
Alumina/ Zirconia	NF	190- 250	TDS 95%
Zirconia	UF	600	Oil and turbidity 90%, TSS 100%

Table 13: Typical operating conditions for oily wastewater ceramic membranes [424]

Membrane (pore size)	Treated water	Operating conditions
Alumina (0.1, 0.2, 0.5 μm)	Oil/water emulsion	TMP = 0.5–2.0 bar CFV = 4.5 m/s
Alumina (0.2 μm)	Refinery wastewater	TMP = 0.75–1.75 bar CFV = 0.75-2.25 m/s
Alumina (0.05 μm)	Synthetic oily wastewater	TMP = 0.5–3.0 bar CFV = 0.2-1.7 m/s
Alumina (0.2, 0.8 μm)	Synthetic OPW	TMP = 0.7–1.4 bar CFV = 0.24-0.91 m/s
Alumina/titania (0.05, 0.1, 0.2 μm)	Synthetic OPW	TMP = 0.5–2.0 bar CFV = 0.6-1.3 m/s
Alumina, zirconia (0.05, 0.1, 0.5 μm)	Oil/water emulsion	TMP = 1.0–4.0 bar

		CFV = 0.47-2.16 m/s
Zirconia (0.2 μm)	Refinery oily water	TMP = 0.45–1.55 bar CFV = 0.58-2.56 m/s

OPW: Oilfield produced water

Ullah et al. investigated the use of slotted pore membrane with a 4 μm slot width and 400 μm slot length [425]. The use of slotted pore membrane lowers the fouling and results in higher separation efficiency than circular pore membrane under lower trans-membrane pressure. The permeation through circular pore membranes is controlled by the trans-membrane pressure, hence, it is possible for spherical droplets to plug the pores of circular pores. However, in the case of slotted pore membrane, the permeation is controlled by the drag force around the oil droplets and it is impossible for a spherical drop to completely plug a slotted pore [426].

5. Conclusion

Industrial activities generate large amounts of oily wastewater which imposes severe effects on the environment. Hence, several oily wastewater treatment techniques were investigated in the literature. Membrane separation technology was proposed as a pretreatment method for saline oily water. Studies in the literature showed very promising results.

The process of separation using ceramic membranes, in particular, was addressed in several studies. These studies showed that ceramic membranes are efficient and economically viable alternative when compared to other treatment methods. The studies in the literature also investigated: 1) different membranes materials and their performance in treating oily wastewater and 2) the possible techniques to enhance the membranes characteristics, hence their separation performance. The results revealed that several modifications have been employed to further enhance the membranes performance. Particularly, surface modifications to improve the fouling resistance received great attention. Operating conditions have a great effect on the performance of the effectiveness of the membrane separation. Thus, many studies have been directed to investigate their effect on the separation performance along with possible ways to optimize them. Membrane separation processes have been evaluated for their commercialization potential. It appeared that

ceramic membranes, in particular, are very promising candidates for treating oily water, thus, they received a special attention. However, their production cost is still high. Hence, more efforts are required to promote cheaper inorganic membranes and further improve their separation performance. These efforts will hopefully result in a more viable industrial scale membrane separation process for oily wastewater.

Abbreviations

A: Membrane Effective Area

AACVD: Aerosol Assisted Chemical Vapor Deposition

AC: Alternating Current

AIBN: 2,2 -azo-bisisobutyronitrile

ALD: Atomic layer deposition

AQ: Anthraquinone

AQS: Anthraquinone-2-sulfonate Sodium

BOD: Biological Oxygen Demand

BPO: Benzoyl Peroxide

BTC: (4-benzoyl benzyl) Trimethylammonium Chloride

CA: Cellulose Acetate

CFV: Cross Flow Velocity

cGO: Carboxylated Graphene Oxide

CNT: Carbon Nanotubes

COD: Chemical Oxygen Demand

CVD: Chemical vapor deposition

DC: Direct Current

EB: Electron Beam

EOR: Enhanced Oil Recovery

GO: Graphene Oxide

GS: Gas Separation

HMWC: High Molecular Weight Compounds

IP: Interfacial Polymerization

ITX: Isopropylthioxanthone

J: Permeate Flux

LMWC: Low Molecular Weight Compounds

MBR: Membrane bioreactor

MF: Microfiltration

NI: Nanoparticles Incorporation

NF: Nanofiltration

OPW: Oilfield produced water

PAN: Polyacrylonitrile

PEG-POSS: Polyethylene Glycol-Functionalized Polyhedral Oligomeric Silsesquioxane

PES: Polyethersulfone

PHCs: Petroleum Hydrocarbons

PP: Polypropylene

PPY: Polypyrrole

PSO: Polysulfone

PVC: Polyvinyl Chloride

PVDF: *Polyvinylidene* Difluoride

RO: Reverse Osmosis

R_o: Oil Rejection Coefficient

t: Permeation Time

TDS: Total Dissolved Solids

TMP: Trans-Membrane Pressure

TOC: Total Organic Carbon

TSC: Total Surface Charge

TSS: Total Suspended Solids

UF: Ultrafiltration

UV: Ultra-Violate

VAC: Vinyl Acetate

VOCs: Volatile Organic Compounds

V_P : Permeate Volume

XAN: Xanthone

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