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REVIEW ARTICLE

Nanoparticles functionalized ceramic membranes: fabrication, surface modification, and performance

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Abstract

Membrane technologies are used intensively for desalination and wastewater treatment. Water filtration using ceramic membranes exhibited high performance compared with polymeric membranes due to various properties such as high resistance to fouling, permeability, rejection rate, and chemical stability. Recently, the performance of nanocomposite ceramic membranes was improved due to the development in nanotechnology. This article focusses on the development of porous ceramic membranes and nanomaterial functionalized ceramic membranes for water filtration applications. At the beginning, various fabrication methods of ceramic membranes were described, and the effect of surface modification techniques on the membrane intrinsic properties was reviewed. Then, the performance of nanoparticles functionalized ceramic membranes was evaluated in terms of physicochemical properties, rejection rate, and water permeability. This work can help new entrants and established researchers to become familiar with the current challenges and developments of nanoparticle-incorporated ceramic membranes for water filtration applications.

Keywords Water filtration · Desalination · Wastewater · Ceramic membranes · Nanomaterials

Introduction

Membrane technology is one of the most effective technologies available for desalination and wastewater treatment. Water filtration using membrane technology is characterized by several features such as small footprint, low sludge production, ease of operation, low operating temperature, and high removal efficiency (Dlamini et al. 2019; Hafiz et al. 2019; Yang et al. 2019).

According to the membrane material, membranes can be classified into polymeric and ceramic membranes. Polymeric membranes have been extensively used for desalination and wastewater treatment (Goh and Ismail 2018). However, the utilization of ceramic membranes is relatively new. Ceramic membranes were utilized in membrane bioreactors (MBRs) (Skouteris et al. 2012), microfiltration, ultrafiltration, nanofiltration, and reverse osmosis (Sheikh et al. 2020). Ceramic membranes have several advantages compared with polymeric membranes including lower fouling propensity, longer lifetime, lower operational cost, stronger mechanical strength, and higher chemical and thermal stabilities (Sheikh et al. 2019). The promising features of ceramic membranes have attracted researchers towards the utilization of ceramic membranes in desalination and wastewater treatment. Currently, there are several studies performed in order to improve the ceramic membranes performance. In addition, many researchers are studying new ways to fabricate ceramic membranes using low-cost membrane materials (Hubadillah et al. 2018a).

The structure of ceramic membrane consists of an active layer, a support layer, and an optional intermediate layer. The support layer provides mechanical strength to the membrane, it is fabricated using metals oxides powders such as alumina and silica in which binders, and peptizing agents are mixed to fabricate the support layer (Cortalezzi et al. 2002; DeFriend et al. 2003). A thin and porous intermediate layer can be used

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between the active layer and the ceramic support layer to increase the permeability of the membrane and to produce a smooth membrane surface (Dong et al. 2020). In addition, the intermediate layer can reduce the pore size of the membrane since most of the low-cost ceramic membranes have a relatively large pore size in the micron range. The intermediate layer can also add different functionalities to the membrane such as hydrophobicity to enhance the membrane performance (Li et al. 2018; Maaskant et al. 2018). The active layer plays a key role in the efficiency of the treatment process because it determines the membrane porosity and contributes to the membrane's mechanical and thermal properties (Gong et al. 2020; Nthunya et al. 2019). Therefore, several attempts have been made to enhance the active layer physicochemical properties by using organic sorbents (Dzyazko et al. 2014), nanoparticles (Park et al. 2012), additives (Wang et al. 2018b), and mixed matrix films (Chong and Wang 2019). A schematic diagram for a nanocomposite ceramic membrane structure is shown in Fig. 1.

There are two main configurations of ceramic membranes, multi-channel flat-sheet membranes, and tubular membranes (Oun et al. 2017; Zhao et al. 2020b). Generally, tubular membranes are preferred over flat-sheet membranes due to their higher surface-to-volume ratio (Kayvani fard et al. 2018). In a multi-channel flat-sheet membrane, the membrane is packed in a plate-and-frame module in which the flat-sheet membrane is sandwiched between the frames (Berk 2018; Martín 2016). In the tubular membrane configuration, the membrane is interposed on the inner wall of rigid porous tubes. The tubes are connected to the plate edges and placed parallel to each other (Berk 2018). The packing density is defined as the membrane area per module. The packing density of the frame configuration is lower than the tubular configuration, which limits its application in the industry (Yasukawa et al. 2018).

Several studies proved that the incorporation of nanoparticles (NPs) to polymeric membranes can enhance the chemical, thermal, and mechanical stabilities of the membranes (Ahn et al. 2008; Dlamini et al. 2019; Li et al. 2010; Maximous et al. 2009; Yu et al. 2009). Also, it can enhance the membrane porosity and reduce the membrane fouling (Dlamini et al. 2019). Therefore, many researchers are currently studying the modification of ceramic membranes with NPs to enhance the ceramic membranes performance. The past 5 years have witnessed an increasing number of published articles related to nanoparticles incorporated ceramic membranes for wastewater treatment and desalination. The fabrication and performance of polymeric membranes for wastewater treatment and desalination have been extensively reviewed (Bassyouni et al. 2019; Karami et al. 2020; Kargari and Rezaeinia 2020; Mansoori et al. 2020; Safarpour et al. 2020; Sheikh et al. 2019; Xu et al. 2019). In addition, the fabrication methods of ceramic components and the application of ceramic membranes have been emphasized (Ayode Otitoju et al. 2020; Issaoui and Limousy 2019). An overview on the most common composite-ceramic membranes in term of improvements and added functionalities for wastewater treatment has been addressed (He et al. 2019). Nanocomposite ceramic membranes fouling issues and performance have been also reviewed (Li et al. 2020a). Therefore, this study aims to review the fabrication and surface modification methods of ceramic membranes with a key focus on nanomaterial functionalized ceramic membranes. The performance of nanomaterial functionalized ceramic membranes was evaluated in terms of mechanical and chemical properties, water flux, and rejection rate. The nanoparticles considered in this study were limited to titania, silver, iron oxide, and zirconium. Finally, general remarks and future perspective for the status of ceramic membranes have been presented.

Fabrication methods

There are several fabrication methods used to produce ceramic membranes. Each method differs depending on the application and the membrane structure. Generally, there are four main stages associated with the membrane fabrication process: formation of particle suspension, formation of membrane structure, membrane sintering, and finishing (Li 2007). In this section, the most commonly used ceramic membrane fabrication methods are described in detail.

Slip casting method

Slip casting is a simple and economical ceramic membrane fabrication method. It is one of the most commonly used fabrication methods at an industrial level, because of the high controllability over the ceramic phase degree of dispersion and the microstructure of the porous material (Studart et al.

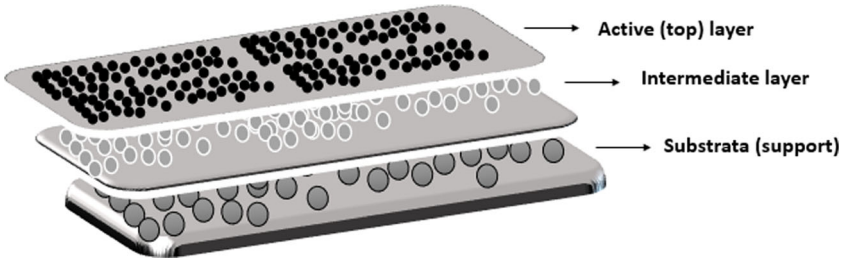

Fig. 1 A schematic diagram for a nanocomposite ceramic membrane structure (Pendergast and Hoek 2011)

2006b). Also, complex shapes of membranes can be produced using this method. Generally, this method involves slurry preparation in which the ceramic powder is mixed with organic binders, chemical deflocculants, and demineralized water. The addition of chemical deflocculants is necessary to maintain the slurry colloidal stability (Wiecinski and Wieclaw-Midor 2020). Then, the slurry is poured into a porous mold where the capillary suction pressure causes the solvent to diffuse through the pores and form a layer on the internal mold surface and later the internal layer consolidates. Choi et al. (2012) prepared tubular oxygen separation membranes using the slip casting method. The ceramic powder (Perovskite BSCF5582) was prepared by mixing $SrCO_3$, $BaCO_3$, F_2CO_3, and CO_3O_4 powders and ground in a ball mill with stabilized zirconia balls. The powder was then exposed to air for 10 h at 950 °C and sieved using a 45-μm sieve. The produced powder was immersed in ethanol and a dispersant mixture that contains Cerasperse 5468 and ammonium salt of polycarboxylic acid. A zirconia ball grinder was used again for 24 h. Deforming agent (SN-deformer 485, polyether), plasticizer (polyvinyl alcohol), and binder (PEG 400, polyethylene glycol) were added. At this stage, the slurry was ready to fabricate the tube using the slip casting method. The slurry was poured into a microporous mold in which the suspension of the solvent is attracted to the pores in the model due to the capillary suction pressure. Consequently, the slip particles consolidated on the surface of the mold in which it can end up by forming particles or a gel layer (Li 2007). The capillary suction pressure is inversely proportional to the pore radius of the mold (Choi et al. 2010). The consolidation process prevents the particles permeation into the mold (Studart et al. 2006a). The consolidation rate is affected by the percentage of solids in the slurry and the casting time. The ceramic tube surface was polished using a 600-grit SiC paper and pasted into an alumina tube with a ceramic sealant at 960 °C. The steps of the slip casting process are summarized in Fig. 2.

A similar procedure was followed by Zhang et al. (2015), where the slip casting method was used to fabricate a dense tubular membrane. The study reveals that slip casting is a simple and a cost effective method that can produce a highly stable membrane. However, it was found that increasing the sintering temperature reduces the membrane relative density and grain size, which results in a lower membrane strength. Moreover, Manni et al. (2020) reported that increasing the sintering temperature decreases the porosity of the membrane due to the partial densification of the ceramic material. This indicates that sintering temperature should be controlled to obtain the desired membrane strength and porosity. Issaoui and Limousy (2019) and Mohammadi and Pak (2003) stated that several low-cost materials such as kaolin and fly ash can be used in the slip casting method to decrease the membrane fabrication cost. The slurry stability, density, and viscosity depend on the particle size, the morphology, solid load, suspension pH, casting time, and thickness (Kim et al. 2015; Kong et al. 2015; Tsetsekou et al. 2001). The main issue encountered in the slip casting method is the weak ceramic membrane strength due to the high porosity and the low toughness of the molds. Also, the sintering temperature should be controlled to control the membrane strength and porosity.

Tape casting

The tape casting method is mostly used to produce thin and smooth ceramic sheets, and it can used to produce highly dielectric and insulated ceramic plates (Howatt Glenn 1952). The method involves the dispersion of the slurry using an adjustable knife that screed the slurry to a desired thickness. Then, the membrane is dried through solvent evaporation from the membrane surface followed by membrane firing, which includes vaporization, decomposition, and oxidation processes. Finally, membrane sintering process is carried out (Schulze and Schiller 1998).

Das and Maiti (1998) fabricated disc-type ceramic aluminum oxide membrane using the tape casting method with a membrane thickness of 200–300 μm. The membrane was prepared by adding alumina powders to MEK ± EtOH (methyl ethyl ketone ± ethyl alcohol), a zeotropic mixture to form a suspension and dispersed into Emphos 2A (phosphate ester). The produced suspension was milled for 4 h. Then, a binder (polyvinyl butyral (PVB)) and plasticizers (benzyl butyl phthalate (BBP) and polyethylene glycol (PEG)) were added to the suspension to be milled for 20 h. The slurry was placed into a reservoir which allows the slurry to pass on the mesh screen surface through the gap between the mesh screen surface and the adjustable knife blade as shown in Fig. 3. The obtained ceramic tape was dried by evaporation. Finally, the membrane was heated up to soaking temperature. The slurry usually contains various types of plasticizers, additives, and binders that are used to produce the dried tape. It was found that a higher binder content causes the agglomeration of particles, which could decrease the pore size of the resulted membrane.

A similar procedure was reported by Das and Maiti (2009), to produce a microfiltration ceramic membrane, followed by using sol-gel to form an ultrafiltration top membrane using the microfiltration layer as a support. The microfiltration layer showed a pore size ranged between 25 and 30 mm. After the deposition of the ultrafiltration layer, the pore size was approximately reduced to range between 30 and 40 nm. However, the membrane stability and strength were not reported. It should be mentioned here that the tape-casting method can be combined with phase inversion (Fang et al. 2013) and freeze casting (Deville 2008) to manipulate the pore morphology.

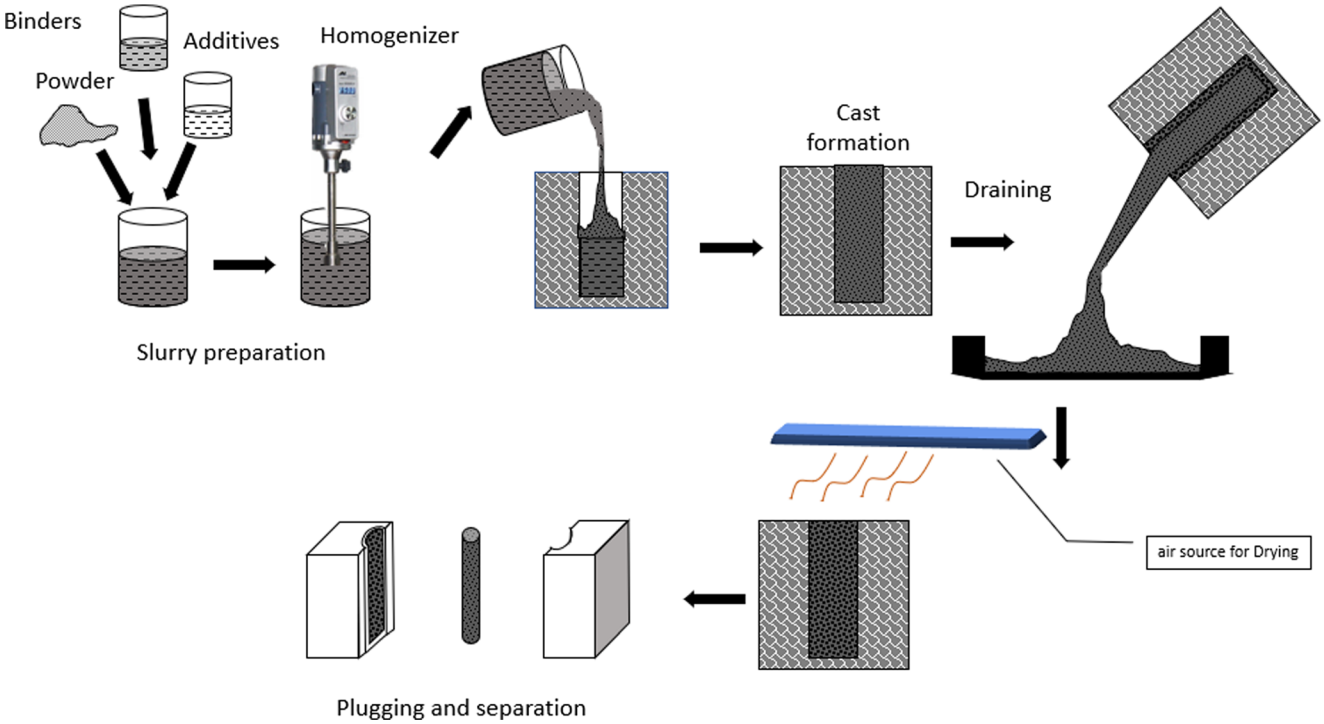

Fig. 2 A schematic sketch summarizing the principle of slip casting method (Choi et al. 2012)

The main disadvantage of the tape casting method is the irregular produced shape of the membrane due to the plaster mold corrosion, especially when using low-cost materials such as kaolin in the slurry mixture (Hao et al. 2021; Lu et al. 2012). Also, crack formation occurs during the organic de-binding and sintering process and the long processing time for the slurry that contains tiny powders. Specific organic compounds should be used as binders, dispersants, and plasticizers in some slurry types such as organic solvents that consist of xylene, benzene, etc., which are toxic, harmful, and expensive (Feng et al. 2020; Nishihora et al. 2018).

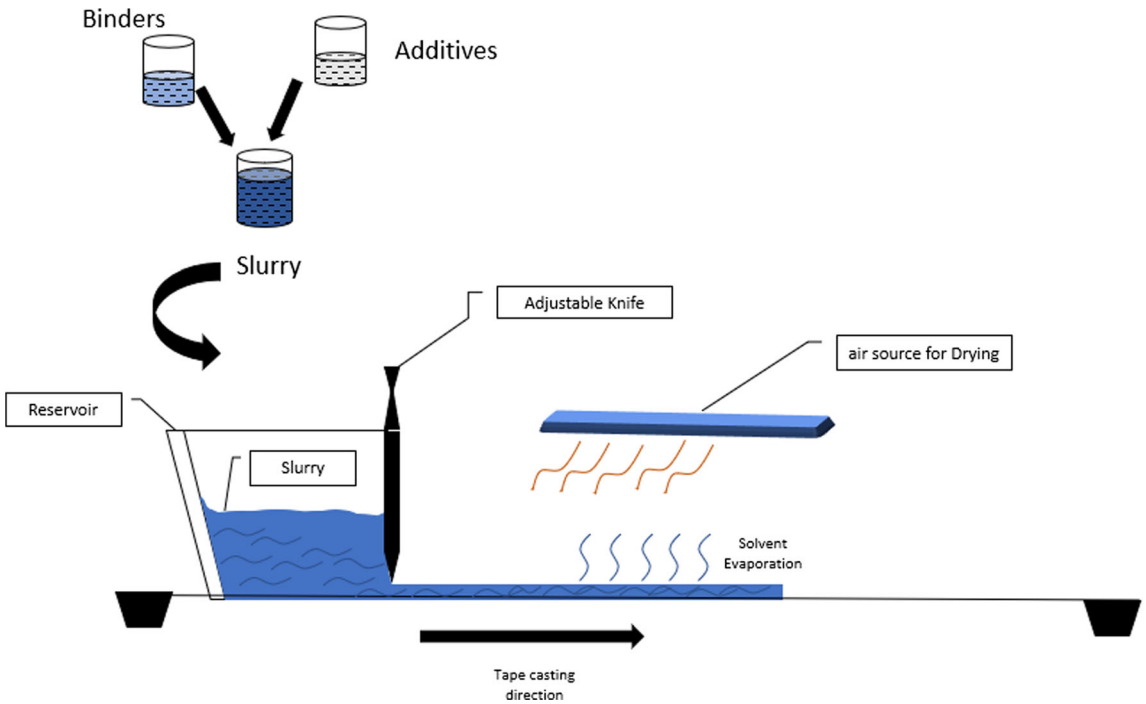

Fig. 3 A schematic sketch summarizing the principle of tape casting (Amin et al. 2016)

Pressing method

The pressing method is considered as the simplest method for the fabrication of ceramic membranes, because the slurry is not required to be discharged to the system (Hubadillah et al. 2018b). This method is based on mixing a dry powder with a pore-forming agent and then pressed uniaxially using a hydraulic pressing machine (Pia et al. 2015). Binders can be added to support the membrane mechanical strength (Manni et al. 2020). Finally, the membrane goes through a heat treatment to obtain a membrane without cracks and deformations followed by sintering process. Del Colle et al. (2011) studied the fabrication of tubular UF and MF ceramic membranes using the pressing method. The ceramic slurry was prepared using structural compounds such as calcined alumina and tetragonal zirconia with an average diameter size of 0.5 and 0.3 μm, respectively. The commercial alumina used in the fabrication process consisted of Al_2O_3 (99.8%) with small fractions of SiO_2, Fe_2O_3, Na_2O, B_2O_3, MgO, and CaO. Sucrose with a purity level of 0.08% was used as a porogenic agent. Moreover, polyvinyl butyral resin (PVB) and isopropyl alcohol were used as binders. For the preparation of porous zirconia and alumina membranes, the isostatic mold was filled with ceramic powder and pressed under a pressure of 100 MPa. The fabrication of the supported membranes (zirconia top layer on zirconia or alumina support) was carried out by dipping the nucleus in the slurry. Then, the membrane was dried in the air by solvent evaporation. This process was repeated twice until obtaining a zirconia thin film on the nucleus. The nucleus is placed in the mold filled with alumina powder and isostatically pressed at 100 MPa. The top layer and membrane support layer were conformed together by co-pressing. An illustration of the pressing step is shown in Fig. 4. Finally, the resulted membrane was placed in a refractory container for thermal treatment at 100 to 160 °C. The membrane produced using this method has a uniform porosity and a homogenous physical characteristics (Li et al. 2016; Mosquim et al. 2020). The top layer of the membrane attained a pore size of 0.01–0.03 μm, whereas the support layer had a pore size of 1.8 μm.

In another study, Ivanets et al. (2016) fabricated a ceramic membrane using the pressing method. Prior to fabrication, sintering of the ceramic material was performed to increase the membrane strength. The results showed that increasing the sintering temperature above 850 °C decreased the membrane strength. The resulted membrane attained a strength of 30 MPa, a pore size of 50–100 μm, and a porosity of 26–28%. In several previous studies, the pressing method has been used as a fabrication process of ceramic membranes with low-cost materials, high porosity, and uniformly distributed pores (Chakraborty et al. 2018; Mestre et al. 2019; Saja et al. 2020; Zou et al. 2019a).

Extrusion

Extrusion is a traditional method used to fabricate porous ceramic membranes with a tubular configuration. Unlike the methods mentioned earlier, a die exhaust is used instead of the mold. The extrusion process involves five steps: blending, pugging, extrusion, cutting and drying, and sintering (Bengisu 2001). In the blending step, the slurry is prepared by mixing the ceramic powder with surfactants, deflocculants, plasticizers, and additives. The produced mixture passes through the pugging step in which the paste is kept at room temperature for hours under high humidity. Then, the paste is injected to a chamber to be extruded. Inside the chamber, there is a die opening with a piston that exerts some force on the paste. The shape, the pore size distribution, and the porosity of the final product are determined by the exerted force. The membrane is cut to the desired length by a sharp blade and dried for several hours. Finally, sintering process is carried out to avoid cracks and bends (Talidi et al. 2011).

Vinoth Kumar et al. (2015) fabricated a tubular ceramic membrane from a clay mixture using the extrusion method

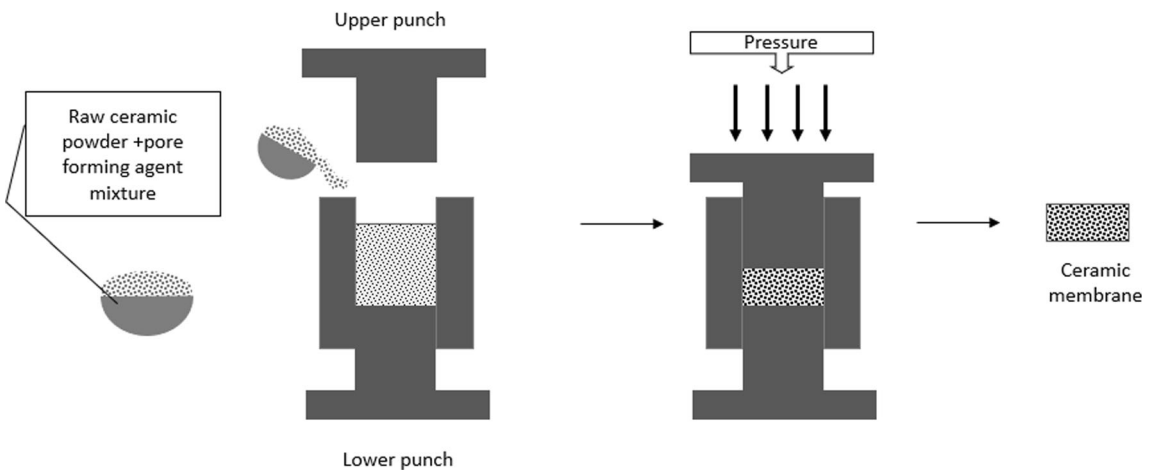

Fig. 4 A schematic sketch summarizing the pressing process of the ceramic powder (Masturi et al. 2019)

for microfiltration of oily wastewater. The study involved a sintering temperature of 950 °C to avoid cracks, deformation, and bends in the membrane. The membrane attained an average pore size of 0.309 μm, mechanical strength of 12 MPa, and a good corrosion resistance in alkaline and acidic environments. Some studies recommend the use of a sintering temperature of 1250 °C to obtain a uniform pore size distribution for membranes made out of clay (Bouzerara et al. 2006; Saffaj et al. 2006).

The geometrical shaping of the production depends on the rheological properties of the slurry. For example, the low viscous slurry is pressed to produce tube geometries. The geometry of the final shaped material depends on the die dimensions and the length of the sample cut (Callister Jr and Rethwisch 2000). Furthermore, this method involves the production of relatively thick walls that decreases the oxygen flux (Athayde et al. 2016). Generally, it is recommended to conduct the extrusion process at high temperatures and relatively low speed to prevent the fracture formation. The extrusion method can also be used to synthesis ceramic membranes from low-cost materials such as apatite powder and kaolin (Abd Aziz et al. 2019; Bouzerara et al. 2006; Mouiya et al. 2019; Saffaj et al. 2006; Vinoth Kumar et al. 2015).

Freeze-casting

The freeze casting method was developed mainly for the fabrication of hierarch ceramic membranes. The method includes physical interaction rather than chemical reactions. It involves the preparation of stable colloidal suspension, pouring the suspension into a mold, freezing the molded suspension, sublimation of the frozen suspension at a low pressure and temperature, and finally sintering the membrane (Athayde et al. 2016). Generally, the colloidal suspension can be a mixture of the ceramic powder, surfactants, and binders. The suspension is frozen for several hours for the solidification process to take place; the slurry particles are trapped between the growing crystals. Pores are formed by sublimation of the slurry solvent. Water is a suitable environmentally friendly solvent to be used in this method. The pore configuration and frequency depend mainly on the solvent crystal growth and the cooling rate (Deville 2008), which are the disadvantages of this method.

Liu et al. (2018) fabricated alumina ultrafiltration membrane using the freeze casting method for the separation of Direct Red 80 dye. This study involved applying airflow treatment followed by heat treatment for the membrane surface during the fabrication process. The results suggest that applying airflow treatment eliminated the formation of cracks and resulted in a smoother surface compared with the fabrication process that did not include an airflow treatment. The membrane that involved airflow treatment attained a pore size of 33 nm, permeability of 457 L m^{-2} h^{-1} bar^{-1}, bending strength of 36.5 MPa, and a rejection rate of 99.6% for Direct Red 80.

Generally, the produced membrane using this method may have low mechanical properties; therefore, a heat treatment is required to enhance the membrane mechanical properties (Moritz and Richter 2007). However, Liu et al. (2013) reported that the pore size, porosity, and mechanical strength can be adjusted by controlling the freezing temperature, solid loading in the suspension, and the powder particle size. Freeze-casting method is distinguished by several features including its simplicity and environmentally friendly nature. The membrane characteristic can be easily controlled by altering the suspension characteristic to obtain the desired membrane pore size and porosity (Gaudillere and Serra 2016; Huh and Kwark 2020). Finally, unlike the extrusion method, the resistance for gas diffusion through the support layer is negligible, which reduces the pressure drop that might occur through the porous support.

Direct foaming

Direct foaming is a low-cost process that can produce ceramic membrane with high porosity and controllable microstructure (Zhang et al. 2014). In this process, the ceramic suspension is foamed by gas incorporation (Barg et al. 2008). Then, the wet foam passes through stabilization, drying, and sintering stages. The stabilization stage is mandatory as the produced wet foam is not thermodynamically stable in which the foam can be degraded as a result of gas bubble coarsening and liquid drainage. Consequently, a stabilization step is needed to avoid the wet foam degradation and to control the microstructure of the ceramic membrane (Barg et al. 2008). Du et al. (2020) fabricated a highly porous silica ceramic membrane by the direct foaming method with an adjustable porosity of 84.1–91.45%. Silica powder with an average particle size of 2 μm and α-Si3N4 with an average particle diameter of 0.42 μm were used as sintering additives. Isobutylene and maleic anhydride solutions were used as gelling agent and a dispersant, respectively. Cationic surfactant cetyltrimethylammonium bromide and the anionic surfactant sodium N-lauroyl sarcosinate as silica powder surface modifiers and foaming agents. First, the slurries were prepared by mixing silica powders with deionized water, silicon nitride, and IBMA then milled for 3 h at 300 rpm. The same amounts of surfactants (0.3 wt%) were added to the slurries and milled for 1 h to complete the foaming process. Then, the resulted wet foams were placed in plastic molds in an ambient environment for 2 days to be dried. After drying, a sintering process took place at 1250 °C in the air. Figure 5 demonstrates the direct foaming main steps and the shape of the ceramic foams in each stage.

This method is attractive due to its operation simplicity, offering a range of pore sizes from micrometers to millimeters and the attainable microstructure of open cell ceramic foams (Kroll et al. 2014). However, the method is considered

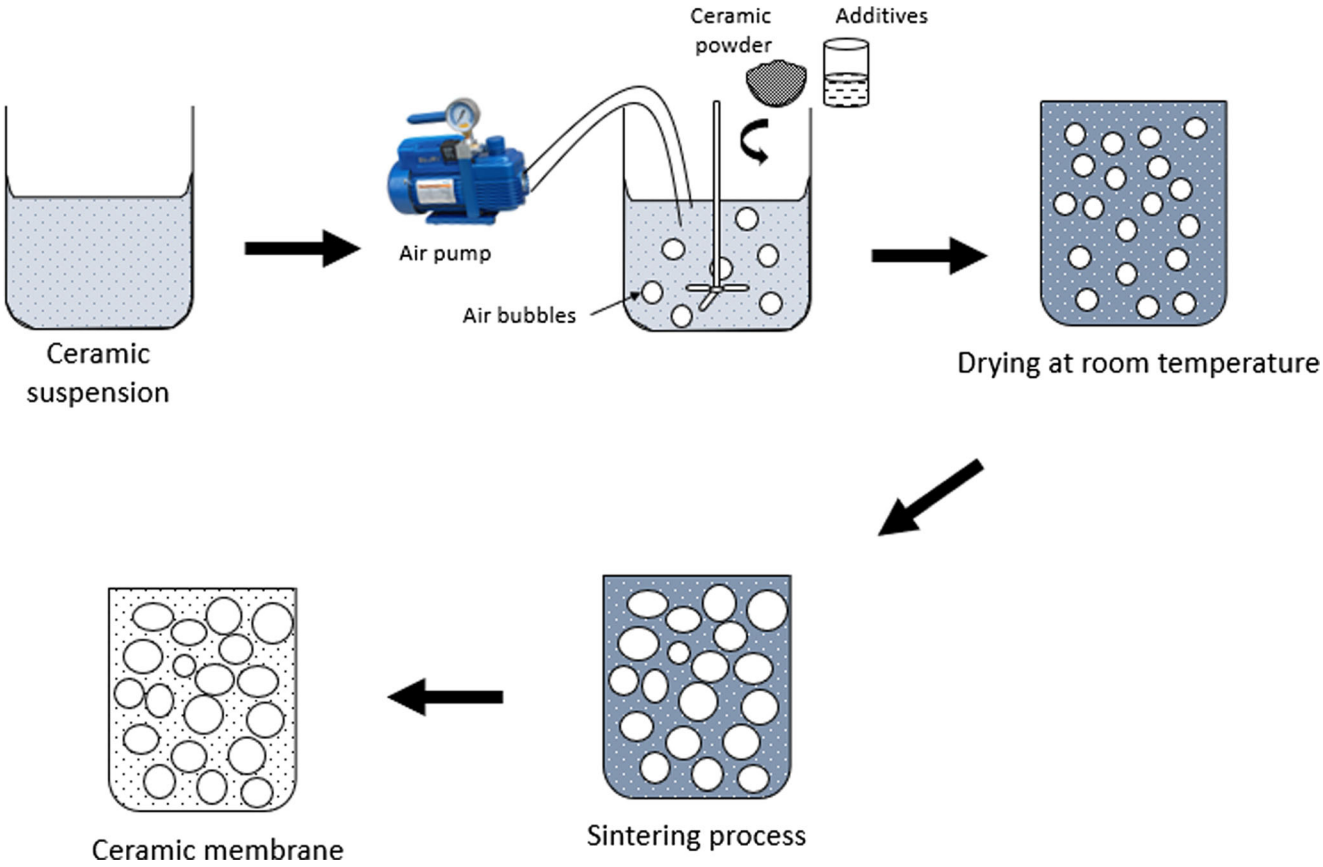

Fig. 5 A schematic sketch summarizing the main steps of direct method (Bhaskar et al. 2016)

expensive and the used binders are not easy to remove. The use of low-cost forming agents can potentially reduce the process cost, but the foam mechanical strength might be affected significantly. Yet, the mechanical strength can be adjusted by controlling the calcination temperature and the foaming volume (Qiao and Wen 2020). Table 1 summarizes the main advantages and disadvantages of each fabrication method used to produce ceramic membranes.

Modification

Ceramic membranes are hydrophilic in nature due to the existence of hydroxyl groups (–OH) on its surface (Krajewski et al. 2006). Most of the commercial ceramic membranes are fabricated using metal oxides such as alumina, zirconia, titania, or natural clay powder (Bouzerara et al. 2006; Larbot et al. 2004; Rigney et al. 1990). The efficiency of membrane based wastewater treatment technologies depends on many factors including hydrophilicity, pore size, and roughness of the membrane, and it also depends on the contaminant size itself. The membrane pore size should be selected depending on the targeted contaminant diameter. Based on the sieving principle of the membrane pore, the contaminant can be trapped, if its diameter is larger than the membrane size (Deng et al. 2021). Furthermore, membrane roughness determines the separation efficiency as it affects the membrane antifouling characteristics (Mao et al. 2020; Woo et al. 2017). Generally, in wastewater treatment using membrane technology, hydrophilic membranes are required to allow the transport of water molecules, while retaining the contaminant in the waste stream. However, in oil-water separation, the membrane can be either hydrophobic-oleophilic or hydrophilic-oleophilic. Hydrophobic-oleophilic membrane restricts the water permeation and allows the oil permeation, while hydrophilic-oleophilic allows water permeation and restricts the oil permeation (Baig et al. 2020). In addition, in membrane distillation, hydrophobic membrane is preferred over hydrophilic membranes to allow the passage of water vapor molecules for better performance (Wae AbdulKadir et al. 2020). Therefore, alternating the membrane hydrophilicity depends on the application, but most of wastewater treatment application targets the use of hydrophilic membranes. Generally, the hydrophilic nature of membranes prevents the passage of the aqueous solution through the membrane pores, unlike other particles that gradually accumulate on the membrane surface (Hafiz et al. 2020; Lawson and Lloyd 1997). The accumulation of particles on the membrane surface is defined as membrane fouling, which is the main challenge in any membrane process and it must be minimized (Tang et al. 2019; Thabit

Table 1 The main advantages and disadvantages of each fabrication method used to produce ceramic membranes

Method	Inexpensive	Simple operation	Environmentally friendly	Multiple configuration can be produced	Produce relatively high membrane strength	Other
Slip casting	√	√	X	√	X	- Difficult to control the membrane thickness - Low production rate
Tape casting	√	√	X	X	X	- Easy to control the membrane thickness - Can produce only flat disc shape
Pressing method	X	√	√	X	√	- Can produce only flat disc shape
Extrusion	√	X	√	X	X	- Usually produce tubular configuration
Freeze casting	√	√	√	√	X	-
Direct foaming	X	√	X	√	√	- Even pore size distribution - High porosity

et al. 2019). Membrane fouling depends on the membrane hydrophilicity that is affected by the surface charge and roughness of the membrane (Ayyavoo et al. 2016). In this context, several attempts have been made to alter the membrane hydrophilicity, oleophicity, pore size, and surface roughness according to the application through surface modification. The surface modification could be achieved by several modification techniques which involves the use of either chemicals or the addition of new material (e.g., nanoparticles) to the ceramic support layer (Hubadillah et al. 2018b). In this section, physical and chemical modification methods applicable to ceramic membrane have been reviewed. Modification processes have been included along with their effect on the membrane properties and performance. Figure 6 illustrates the different types of chemical and physical modifications methods.

Physical modification

Surface coating

Surface coating has been widely used in the modification of ceramic membrane to minimize fouling while retaining the rejection rate and permeate flux. Surface coating may enhance the performance of a ceramic membrane by adding various functionalities such as resistance to biofouling and enhancement of membrane hydrophobicity (Li et al. 2009). There are several widely available coating materials including polymers (Changmai and Purkait 2018), nanoparticles (Gao and Xu 2019), surfactants (Xomeritakis et al. 2003), and polyelectrolytes (Dotzauer et al. 2009). The interaction bonds between the added thin layer and the ceramic membrane are Van der Waals, electrostatic, and hydrogen bonds. These bonds are weak; therefore, the produced membrane might be unstable in long-term operations. In addition, the coated layer may get corroded during the physical or chemical membrane cleaning processes (Thombre et al. 2020). Chemical cleaning is the most commonly used method for membrane cleaning which involves the use of chemical reagents such as surfactants that might damage the membrane material and cause the separation of the coating layer from the membrane surface (Garmsiri et al. 2017; Zhao and Yu 2015; Zhou et al. 2017). Physical cleaning such as backwashing has a long processing time, and it interrupts the continuous filtration process (Muthukumaran et al. 2004).

There are several coating methods including dip-coating, spin-coating, spray-coating, and filtration coating. In the dip-coating method, the ceramic support layer is immersed in a coating solution; then, evaporation of the residual solvent occurs. The viscosity of the particle suspension, coating time, and coating speed are crucial factors in the dip-coating method (Cui 2015). Changmai and Purkait (2018) fabricated a poly(2-ethyl-2-oxazoline)-coated ceramic membrane using the dip-coating method. The process involved the immersion of a ceramic porous membrane in poly(2-ethyl-2-oxazoline) solution for various times from 20 to 60 s. Then, the coated membrane was kept on a flat glass surface for 24 h at 24 °C. During the drying process, acetone evaporated from the membrane top layer, this contributed to the membrane morphology and formation of pores. The field-emission scanning electron microscope (FESEM) images reveal that increasing the polymer concentration reduces the composite membrane pore size, which could be attributed to the change in viscosity. The increase in polymer concentration increased the viscosity of the polymer solution, which caused a low droplet growth rate of the membrane pores. The increase in dipping time caused a significant reduction in pore size from 6.61 to 0.24 μm due to the deposition of poly(2-ethyl-2-oxazoline) on the ceramic support surface. The water contact angle of the coated ceramic membrane varied from 37.4 to 76.98°. Zhang et al. (2016)

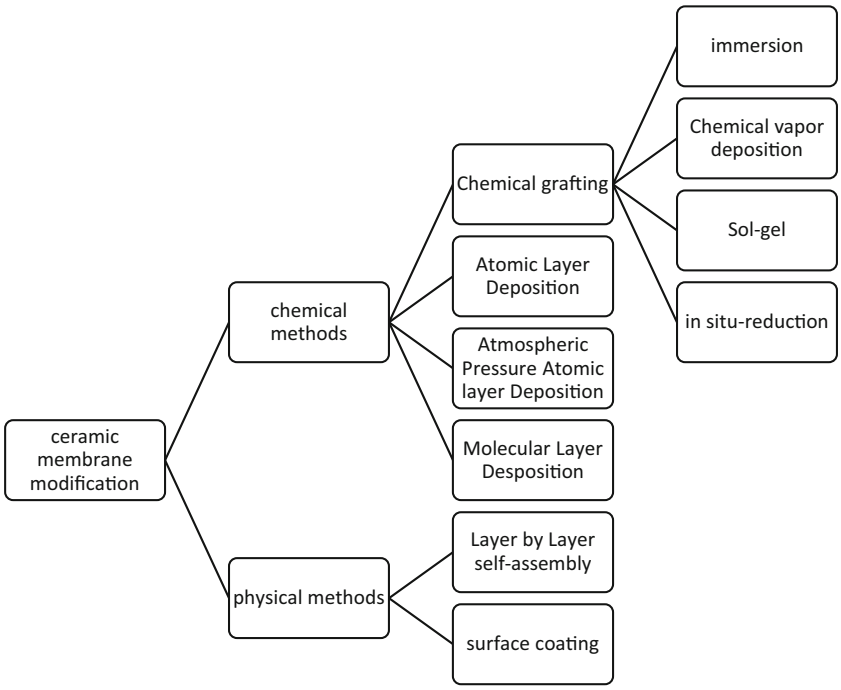

Fig. 6 Chemical and physical modification methods applied for ceramic membranes

used the dip-coating method in coating ceramic hollow fiber membranes with TiO_2 nanofibers. The thickness of the TiO_2 nanofiber layer was determined by the coating time. The thickness increased from 2.14 to 7.14 μm as the coating time increased from 15 to 45 s. The porosity of the coated membrane ranged from 75 to 85%, whereas the uncoated ceramic membrane attained a porosity lower than 36%. In addition, the coated membrane surface became smooth and stable and cracks were not observed.

The spin-coating method is another coating method used for ceramic membranes coating. This process involves the deposition of few drops of the coating solution on the sample surface followed by rotating the support layer at a constant rotational speed; this causes the liquid to spread in the outward direction. Finally, the volatile liquid evaporates from the membrane surface, which causes the film thickness to decrease (Lue et al. 2015). Bouazizi et al. (2017b) used the spin-coating method for coating a ceramic membrane with titania nanoparticles (TiO_2/UF). The ceramic membrane was successfully coated as confirmed by the XRD pattern and the SEM images. The SEM images revel that TiO_2 was uniformly distributed on the ceramic membrane surface. In addition, the coated membrane surface was rough compared with the ceramic support layer. The pore size of the coated membrane ranged between 50 and 100 nm. The membrane showed a maximum DR 80 rejection of around 98%, yet the stability of the membrane was not investigated.

Saja et al. (2020) used bentonite layer to coat a ceramic membrane support layer using the spin-coating method followed by a sintering process at 500 °C. A uniform distribution of bentonite over the ceramic support layer and strong adhesion force was observed. The pore size decreased from 17 to 4 nm as the bentonite content increased from 0.25 to 1.50 wt%. In addition, the water contact angle of the coated membrane increased from 0.75 to 68.20 as the bentonite content increased, which indicate that the hydrophobicity of the membrane increased reflects.

In the spray-coating method, the coating solution is sprayed on the ceramic support layer. The spraying gun is connected to an air compressor to provide constant pressure. The thickness of the coated layer can be controlled by adjusting the volume of the coating solution (Liu et al. 2017). Similar procedures were followed by Zhang et al. (2019b), where the slurry (mixture of polyvinyl alcohol, powder silica, wetting agent, antifoam, and graphite) was used to coat a silica membrane support layer using the spray-coating method. It was observed that cracks were eliminated for a spray cycle greater than three cycles and the pore size attained a value of 23.49 μm. The resulted membrane showed high thermal shock resistance and good permeability. The major disadvantage of this method is the need for multiple spray cycles to eliminate surface cracks. However, thermal-spray coating method can be used to eliminate the surface cracks without multiple spraying cycles to produce ceramic membranes with a small pore-size. Zou et al. (2019b) proposed a novel thermal spray-coating process to fabricate fly ash/alumina composite membrane for oily water treatment. The process involves heating the support layer to a specific temperature prior to the spraying step. After spraying the support layer with the fly ash dispersant, the dispersant volatilizes, which causes the dispersant's particles to form clusters. The clusters form a giant network, which causes the formation of small pores of around 100 nm. Unlike the

conventional spray-coating, this method eliminates the use of an intermediate layer, which reduces the membrane fabrication cost. However, the temperature should be adjusted because high temperatures could accelerate the volatilization of water from the dispersant. This causes the formation of bubbles that negatively affect the membrane integrity and might cause surface cracks. The fly ash/alumina composite membrane showed a total organic carbon and stannic acid rejection above 99% and a permeability of 445 L m^{-2} h^{-1} bar^{-1}.

Filtration coating is a recent novel process which was proposed by Chung et al. (2018). In this method, Al–Zr nanoparticles were prepared prior to the coating step in the membrane operation module. The piling up of nanoparticles on the membrane surface was prevented by using a cross flow velocity of 15 cm s^{-1}. The coating process took 6 h, and the flux stabilized at 10 bar. The drying step was implemented using a microwave to prevent the formation of cracks followed by 3 h calcination at 700 °C. After the coating process, the membrane pore size was reduced from 300 to 4.3 nm with no cracks. The coated membrane showed a rejection rate of 91% for uranium ion. Figure 7 shows the SEM images for the UF ceramic support before and after filtration coating process.

Layer-by-layer self-assembly

Layer-by-layer self-assembly is a process that involves the formation of particles into a well-defined network on the membrane surface (Gao et al. 2020). It is characterized by several features including ease of operation, versatility, minimal energy requirement, and minimal material waste (Costa and Mano 2013). It is widely used for various types of materials such as nanoparticles and polyelectrolytes (Alsyouri et al. 2006; Chen et al. 2007). There are four major steps in this method which are as follows: (1) adsorption of positive components, (2) washing, (3) adsorption of negative components, (4) washing. These steps may be repeated to obtain a satisfactory thickness of the film (Kotov 1999). The studies on the self-assembly method for composite ceramic membranes are limited, yet it was used extensively for locating nanoparticles in polymeric matrixes (Li et al. 2020c; Radjabian and Abetz 2020). In the layer-by-layer self-assembly, the interaction type between the first deposited layer and the ceramic membrane is an electrostatic interaction (Dhar and Patil 2012). The other layers can have various interactive mechanisms such as electrostatic interaction, chemical interaction or mediating agents (Chen et al. 2020; Dhar and Patil 2012; Huie 2003). The addition of coupling agent such as silane before the membrane assembly can improve the electrostatic interaction and increase the membrane stability (Wang et al. 2015). Chemical self-assembly is preferred over electrostatic interaction self-assembly as the produced membrane is more stable due to the presence of a chemical bond between the deposited particles and the ceramic support layer (Huie 2003). Multiple layers can be added to the ceramic support surface using the layer-by-layer assembly method compared with other modification methods with high adhesion force that can be obtained using coupling agents. Zhou et al. (2019) fabricated polydiallyldimethylammonium chloride (PDDA)–modified MoS_2/polyelectrolyte composite membrane on a ceramic tube. The results reveal that there was a strong interaction between the nanosheet (PDDA@MoS_2) and the polyelectrolyte that resulted in a dense layer and decreased the flux. The membrane showed an excellent operation stability and a rejection up to 97.8%.

Chemical modification

Chemical grafting

Grafting is the most frequently used technique for the hydrophilization of ceramic membranes which is mainly used

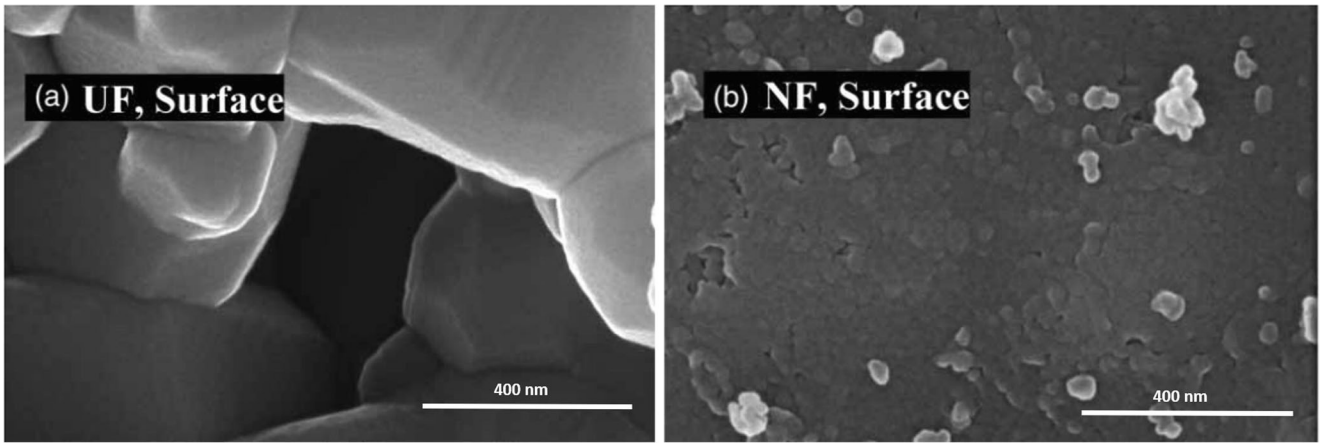

Fig. 7 SEM images for **a** UF ceramic support before coating and **b** the membrane after coating with Al–Zr nanoparticles (Chung et al. 2018)

for membrane distillation application (Wae AbdulKadir et al. 2020). This technique is based on low surface agents that can be added to the membrane surface and linked with the hydroxyl group. Silane agents (especially fluoroalkyl silanes (FAS)), graphene oxide, and graphene oxide quantum dots are commonly used hydrophobic agents in the grafting method that can be applied for different surface types (Fang et al. 2012; Krajewski et al. 2006). By applying this technique using FAS, a single layer of organosilane will be formed on the membrane surface. Organosilane compounds react in a hydrolysis reaction to form a new compound with multiple hydroxyl groups. Then, the newly formed compound reacts using dehydration with the hydroxyl groups that are present on the membrane surface to end up with a hydrophobic grafted product (Chen et al. 2018). Graphene oxide has attracted the researchers' attention during the last years especially in the membrane surface modification applications (Guan et al. 2020; Ray et al. 2015; Wang et al. 2019). Graphene oxide (GO) is a hydrophilic compound which has a negative charge and contains high concentration of oxygen functional groups such as –COOH and –OH. This could be used to mitigate the membrane fouling problem by removing some of the charged particles. Grafting is one of the most common and promising mechanisms to modify the membrane surface using GO. Graphene oxide quantum dots (GOQDs) are extended derivatives of the GO with a size in nanometers. Compared with GO, GOQDs are hydrophilic due to the presence of epoxy, hydroxyl, and carbonyl functional groups, which contain high oxygen amounts. In addition, GOQD fine particles can be applied on inorganic materials such as ceramic under an external driving force without the need of steric blocking effect. Chemical grafting is achieved through mainly three techniques: immersion, chemical vapor deposition, and sol-gel.

Immersion method This method is considered to be the most well-known, simple, and fast technique among the ceramic membrane hydrophobization techniques that uses grafting of silane agents (Lu et al. 2009). In this technique, silane agents must be activated to silanol species before the immersion process. This could be achieved by mixing the silane agent with a solvent such as alcohol, hexane, or water. The reactive species are converted into hydroxyl groups. Then, the ceramic membrane is immersed into the activated silane agents where a chemisorption of silane molecules takes place on the ceramic membrane surface. Hence, the remaining Si–OH forms a bond with the adjacent silane molecules to form Si–O–Si bond. The bonds formed by the chemisorption process on the membrane surface are strong and immobilize due to the intermolecular forces between the forming molecules (Dimitriadi et al. 2019). As a result, the formed thin film improves the chemical and mechanical stability of the ceramic membrane surface which in return enhances the hydrophobic nature. It is worth noting that there are some factors which affect the immersion process such as quantity of the OH ions on the membrane surface, concentration of the silane agent, time and number of grafting, and surface roughness (Johnson and Hilal 2015). The main disadvantage of this method is the high cost of the silane agents. Hence, optimization studies should be carried out to achieve the optimum silane agent's concentration with the least cost and grafting time. In order to examine the quality of the hydrophobic layer formed on the surface of the ceramic membrane, an analysis for the pore size, permeability, and water contact angle should be carried out. Moreover, the relationship between the grafting time and pore size must be investigated. Hence, the improvement of these properties will help to enhance the anti-fouling resistance of the ceramic membrane.

Zhang et al. (2019a) studied the modification of β-SiAlON membranes used in oil-in-water emulsion separation processes. They evaluated the production of modified β-SiAlON membranes using the immersion method with high strength by applying SiO_2 nanoparticles to decrease the pore size and enhance the anti-fouling characteristics and surface roughness. β-SiAlON membranes were fabricated using the immersion-induced phase inversion technique at 1650 °C for 4 h. Then, the SiO_2 particles were fabricated by preparing two solutions; the first solution is 100 ml of tetraethyl-orthosilicate (TEOS) and ethanol in equal amounts. The second solution was 283 ml of ethanol, 147 ml water, and 70 ml ammonia aqueous solution. The two solutions were placed in a beaker to be mixed for 2 days at room temperature. Then, the produced gel was washed with water and dried for 30 min. The β-SiAlON membranes were immersed into a stable solution of SiO_2 particles at 90 °C for 30 min. The membranes were placed in a furnace at 600 °C for 4 h. The pore size was reduced significantly from 1.05 to 0.65 μm after the modification of the membrane. The oil rejection rate increased from 39.8 to 90%. The modified membranes showed an outstanding anti-fouling ability. Kujawski et al. (2016) carried out a grafting procedure by immersing TiO_2 membranes in two different FAS agents namely C6 and C12 with different grafting periods to analyze the effect of grafting time on the hydrophobicity and pore size for vacuum membrane distillation application. It was found that as the grafting time increased, the liquid entry pressure (LEP) value increased until reaching a constant value of 14 bar after 67 to 84 h of grafting. This is due to the fact that the ceramic membrane surface has a limited number of OH groups to be linked with silane agent. Hence, this can be used as an indicator of membrane hydrophobicity improvement. Yang et al. (2017) performed an experiment using 1H,1H,2H,2H-perfluorooctyltrichlorosilane (PFAS) and 1H,1H,2H,2H-perfluorodecyltriethoxysilane (PFDS) as FAS surface modifiers by immersion method followed by ultraviolet radiation to create a superhydrophobic membrane for vacuum membrane distillation. An alumina membrane with a mean pore size of 200 nm, a porosity of 35%, and a

water contact angle of 46° was used. The results showed that the water contact angle increased from 46 to 159° that improved the membrane hydrophobicity and decreased the pore size. Hence, the membrane fouling decreased.

Chemical vapor deposition Chemical vapor deposition (CVD) is a commonly used technique in membrane surface modification. The technique is based on depositing a solid layer of the same or different compounds on the membrane surface. This can be achieved by carrying out chemical reactions in a gaseous medium at high temperature. In general, CVD process requires a system to deliver the mixture of reactive and carrier gases to a heated reaction chamber where film formation occurs (Khatib et al. 2011). CVD method is commonly used in the production of thin film coating on membrane's surface (Vlassiouk et al. 2013). Unlike the immersion method, thermal energy is required for the activation of silane agent to react with the hydroxyl groups on the ceramic membrane's surface using the same mechanism of the immersion method. Silane agents and ceramic membranes are placed in an oven to be heated until the silane agent reaches the boiling point.

Amanipour et al. (2012) studied the fabrication of tubular ceramic membranes by the deposition of dense silica-alumina layer on the surface of a multi-layer substrate using CVD to enhance the hydrogen permeation for an effective separation system. The dense deposited layer was prepared using macroporous (<50 nm) or mesoporous (pore size of 2–50 nm) alumina support, and the coating has been done with graded γ-alumina multi-layer. The preparation process has two major steps, the fabrication of intermediate γ-alumina and the decomposition of silica and alumina precursors on the substrate layer at high temperature using a co-current chemical vapor deposition method. It was observed that the addition of the dense layer on the membrane decreased the permeance values, but it increased the hydrogen selectivity. Furthermore, the usage of silica-alumina modified membranes enhanced the membrane performance by improving the structural stability in the presence of steam and by increasing the gas permeability. Excessive amounts of alumina in the dense layer caused a reduction in the H_2 selectivity. Khatib and Oyama (2013) highlighted that the CVD method is not a good choice to be applied with MD application due to the difficulty of controlling the pore sizes which leads to an increase in the permeability up to 50%. In another study, Gu et al. (2020) used the CVD method to adhere GOQDs on a ceramic membrane using triethoxysilane (APTES) as a linking agent in order to enhance the membrane anti-fouling characteristics, pore size, hydrophilicity, surface roughness, and permeability. GOQDs were produced by facile pyrolysis of citric acid and were applied on a MF ceramic membrane. In this process, APTES reacted with the hydroxyl groups of the ceramic membrane using hydrolysis reaction; then, the GOQDs were grafted on the ceramic membrane through a dehydration process between amino groups and carboxyl groups. By carrying out GOQD surface modification to the ceramic membrane, an enhancement was observed on most of the membrane characteristics. The membrane average pore size decreased slightly from 200.8 to 191.3 nm while obtaining a uniform pore size distribution. The water permeability improved by 30%, and the surface of the modified ceramic membrane was smooth and less hydrophilic compared with the virgin membrane.

Sol-gel method Sol-gel is another method of producing a hydrophobic layer on the membrane surface. In this method, the produced ceramic membranes have high purity, long lifetime and uniform distribution of pores (Chen et al. 2001). Sol-gel method is commonly used for oil-water separation processes. In this method, the preparation of an unsupported membrane is done by mixing the synthesized ceramic with the alumina sol and drying it for 24 h to obtain the gel. The gel is oxidized at a temperature range between 400 and 900 °C. The alumina is coated by the sol and kept for drying. The only disadvantage of this method is the crack formation in the gel drying stage. To overcome this issue, organic binders must be added to prevent the formation of cracks in the drying step (Ahmad et al. 2005). Sol-gel is a slow process, because the preparation of the sol-gel takes long time. The sol-gel solution is prepared by the modified Stöber method. Tetraethoxysilane (TEOS) and methyltriethoxysilane (MTES) are the most common FAS agents used in this method (Hubadillah et al. 2019). Tetraethoxysilane is mixed with an ethanol-water solution which reacts at 25 °C to form colloidal silica. Then, methyltriethoxysilane and ethanol are added to the mixture and mixed at 60 °C; the solution is left for a period of 3 days. Moreover, membranes with thiourea derivatives involved in their fabrication became promising and attractive to many researchers in the last years (Abraham et al. 2015; Jumal et al. 2012; Wilson et al. 2010) due to the ability of this group to enhance the hydrophobic nature of the membrane. Tomina et al. (2019b) studied the selective layers of amino and thiourea groups on planar and tubular ceramic alumina membranes surface using the sol-gel method by the hydrolytic polycondensation reaction of alkoxysilanes. The membrane support layer was activated, boiled in HCl for 5 h, and finally rinsed with water until obtaining a pH of approximately 7. Then, the membrane was dried in a furnace for 6 h at 320 °C. The excess sol can be removed from the membrane surface by air purging. Then, the membrane was left at room temperature for 2 h, then 2 h at 30 °C and 50 °C, later 20 h at 80 °C. As a result of the ceramic membrane modification using silica nanoparticles with thiourea functions from MTES or ETUS as a source of functional thiourea groups, the hydrophobicity of the membrane increased. In this method, there is high controllability on the porous structure of the surface layer when it forms and ripening of silica sol and its

transition to gel, by changing the ratio of reactants, the nature of the solvent, and the drying mode. Su et al. (2012) motivated the superhydrophobic behavior using sol-gel method. This could be achieved by preparing sol-gel solution from TEOS, ammonia, distilled water, and ethanol mixture at 30 °C and then apply it to the ceramic membrane after 7 days. This modification method showed a reduction in the ceramic membrane pore sizes.

In situ reduction In a recent work (Peng et al. 2020), half encapsulated silver nanoparticles by a loosely polyimide (PI) were grafted onto ceramic membrane via in situ reduction during thermal imidization to fabricate polyimide-silver nanoparticle- ceramic membrane (PI-Ag/CM). The preparation included the addition of 10 ml of 3-aminopropyltrimethoxysilane (APTMS), 10 pieces of ceramic membrane, 100 ml ethanol, and 1 ml deionized water into 250-ml three-necked flask. The reaction took place for 12 h at a constant temperature of 70 °C to obtained APTMS/CM. Finally, 100 ml of anhydrous DMF, 0.11 g pyromellitic dianhydride DMF, and 0.405 g $AgNO_3$ were added to 10 pieces of APTMS/CM. The mixture was stirred for 24 h at 25 °C followed by drying at 90 °C for 5 h and heating at 120 °C, 160 °C, 240 °C, and 300 °C for 1 h intervals. The results indicate that silver nanoparticles with a size of 50–80 nm were uniformly embedded onto PI. PI-Ag/CM showed a rejection rate of 99.4% for *Escherichia coli*. In addition, the study confirms the high stability of silver nanoparticles in PI-Ag/CM and significantly lasting anti-biofouling performance. In another work, Ngoc Dung et al. (2019) prepared silver-nanoparticle ceramic filter (SNP-CF) by in situ reduction. The results show that SNPs were uniformly distributed over the ceramic structure. Furthermore, SNP-CF attained a 100% rejection of *Escherichia coli* and coliform. In a long-term filtration test, the results suggested that SNP-CF performance can be extended up to 10 weeks, which is a result of the uniform distribution of Ag NPS and their slowly release into the filtration media in a controllable manner.

Atomic layer deposition and atmospheric pressure atomic layer deposition

There are few methods which can tailor the pore size of a ceramic membrane. This is due to the difficulty of observing the relationship between the production parameters and the membrane microstructure. Therefore, applying simple techniques for the purpose of tailored production of ceramic membranes is an appealing research topic. Atomic layer deposition (ALD) is a self-limiting gas-phase deposition derived from CVD method used to modify the ceramic membrane structure and tailor the pore size. This can be done by the deposition of uniform layers with a controlled thickness of the ceramic support layer (George 2010). A monolayer is formed by each cycle and the layer thickness can be tuned by increasing the number of cycles. This process is based on pulsation of precursor gases on the targeted surface followed by a chemisorption process (Leskelä and Ritala 2002). In ALD, the inert gas is purged to the reactor between the precursor gases pulses. Under some experimental conditions, the growth is taking place steadily and the layer thickness increases in each deposition cycle. The self-limiting reactions which have been carried out on the gas-solid interfaces help the growth of uniform thin film on large areas. ALD became a new promising technique for producing high-quality modified membranes in the last years (Berger et al. 2020; Feng et al. 2019; Weber et al. 2020). Li et al. (2012) modified a tubular ceramic membrane by deposing an AL_2O_3 layer on the ZrO_2 selective layer using a commercial ALD reactor. The membrane pore size decreased as the ALD cycles increased until the pores were completely sealed which mean that the pore size can be tuned to the desired pore size. The pore size of an UF ceramic membrane approximately decreased from 50 to 6.8 nm, and the water permeability decreased from 1698 to 118 L $(m^2\ h\ bar)^{-1}$ by alumina deposition.

As a modified version of ALD, atmospheric pressure atomic layer deposition (APALD) is a new economical, simpler, and efficient process, which does not required the costly vacuum. Furthermore, APALD provides an easy scale-up that is applicable for large volume manufacturing. Shang et al. (2017) fabricated tight ceramic NF membranes using APALD and examined the process effect on the water permeability, rejection of polyethylene glycols (PEGs), pore size, and the molecular weight cutoff (MWCO). TiO_2 was coated on the outer and inner channel surface of the membrane using a flow type APALD reactor. Moreover, nitrogen gas was used to dilute $TiCl_4$ and demineralized water was used as a precursor, then a chemisorption process was used to apply $TiCl_4$ on the membrane surface. Later, a dry nitrogen gas was used to purge the excess $TiCl_4$ and HCl vapors. Nitrogen gas purging was used again to clean up the excess water and HCl vapors before repeating the process. An infrared lamp connected to a digital temperature probe was used to heat the APALD reactor to 180 °C. The pore size decreased from 0.7 to 0.5 nm with a more uniform size distribution. In addition, molecular weight cutoff (MWCO) was reduced from 400 to 260 Da with maintaining the same water permeability.

Molecular layer deposition

The mechanism of molecular layer deposition (MLD) is similar to the conventional ALD. MLD is based on self-limiting surface reactions where a precursor molecule reacts with the membrane surface group and forms a chemical bond. The newly formed layer is attached chemically to the functional group developed on the precursor molecules (Zhou et al. 2013). The main difference between ALD and MLD is the compatibility of ALD with in-organic materials only, unlike MLD which is compatible with organic and in-organic

materials (Dameron et al. 2008; Song et al. 2015). A hybrid ALD-MLD process for in-organic and organic hybrid thin film was used for various applications, which is expected to be a vital research area for ceramic membrane modification (Sood et al. 2013). It is difficult to control the pore size at a nanometer level in ALD. The transmembrane resistance might increase due to the dense layer deposited using the ALD method (Li et al. 2012; Song et al. 2016). However, an ultra-thin porous layer can be formed on the support surface using MLD along with a controllable pore-size (Song et al. 2016). In order to obtain a uniform porous structure, the organic contents should be removed from the membrane surface by mild water etching at room temperature or by calcination at elevated temperatures (Liang et al. 2009; Van de Kerckhove et al. 2018). There are several studies reported the performance of MLD modified ceramic membrane. Song et al. (2016) used MLD to synthesize TiO_2 nanofiltration ceramic membrane (AAO-60TiO_2), where $TiCl_4$ and ethylene glycol were used as a precursor. The study involved the elimination of organic matter by calcination at 250 °C for 4 h followed by cooling at a rate of 1 °C min^{-1}. The ceramic support layer exhibited a pore size ranged from 20 to 30 nm with a porosity of 60%. After modification, a smooth membrane was obtained using 60 cycles of MLD. The modified membrane had a pore size of 1 nm and improved fouling resistance. Na_2SO_4, $MgSO_4$, Methylene Blue, and natural organic matter attained a rejection of 43%, 35%, 96%, and 99%, respectively. Similarly, Wu et al. (2019) used $TiCl_4$ and ethylene glycol to fabricate a nanofiltration TiO_2 ceramic membrane. The membrane was calcinated at temperatures of 250, 300, 350, 400, and 500 °C for 4 h. The results indicated that the calcination temperature had a significant impact on the membrane morphology and performance. The thickness of the titancone film (TiEG) on the membrane support surface decreased as the calcination temperature increased due to the removal of organic constituents. Also, the surface of the modified membrane became smoother at a calcination temperature of 250 °C. This can be attributed to the conversion of TiEG film to amorphous TiO_2 at a calcination temperature of 250 °C and then to crystalline anatase TiO_2 at 400 °C. This caused the membrane pore size to increase, which affected the water permeability and the rejection rate. As a result of calcination at 250 °C, the water permeability dropped to 7 L m^{-2} h^{-1} bar^{-1} due to the thick TiO_2 coating layer. Table 2 shows an overview of different modification methods.

Nanoparticle-incorporated ceramic membrane performance

The incorporation of nanoparticles into ceramic membranes can potentially add several characteristics that may enhance the membrane performance. The addition of nanoparticles to polymeric matrix has been extensively applied for wastewater treatment and desalination. Many of these studies reveal that using nanoparticles have enhanced the membrane performance by adding several functions to the membrane such as antifouling and antimicrobial functionality (Karami et al. 2020; Lam et al. 2018; Ng et al. 2013; Saleem and Zaidi 2020). The functionalization of ceramic membranes with nanoparticles has not been studied to a large extent, but there are several studies indicate that nanoparticles can potentially enhance the ceramic membrane performance.

The microstructure of nanoparticle incorporated ceramic membrane consists of two main layers and an optional layer of different pore sizes as described earlier. The active layer consists of nanoparticles with smaller diameter compared with the substrate layer. The addition of nanoparticles to a ceramic membrane can be achieved by several methods such as in situ reduction and dip-coating method as discussed earlier. Some of these methods may cause the aggregation of nanoparticles and poor adhesion, which negatively affects the membrane mechanical stability. Consequently, other methods are preferred to localize nanoparticles such as chemical grafting, self-assembly, and physical and chemical depositions.

Despite the fact that surface modification can reduce membrane fouling, further development of the technology is still required (Kim and Van der Bruggen 2010). The integration of nanomaterials into a membrane can potentially enhance the water purification process by improving the membrane properties such as porosity, membrane stability, and hydrophilicity (Jiang et al. 2020; Zhao et al. 2020a). Furthermore, the addition of nanomaterials into a membrane may enhance the photoactivity functionality, which can improve the membrane performance (An et al. 2014; Desa et al. 2019). These functionalities depend on the properties of the used nanomaterial such as surface area, porosity, and surface functional groups (Lu and Astruc 2020). The amount of nanomaterial must be selected carefully, since shortage in the amount of nanomaterial shall not be sufficient to cause any remarkable changes in the membrane performance (Desa et al. 2019). The most common used nanoparticles in ceramic membranes are iron oxide (Fe_2O_3), aluminum oxide (Al_2O_3), titanium dioxide (TiO_2), and silver nanoparticles (Ag). The additional functionalities provided by nanoparticles can potentially degrade foulants under oxidizing conditions. Table 3 summarizes the performance of nanoparticles incorporated ceramic membrane for water filtration applications. The integration of nanoparticles with ceramic membranes is a challenging process. The main challenge is finding the proper binding medium to create a link between the ceramic membrane surface and the nanoparticles (Ding et al. 2019). The improper binding may cause a reduction in the permeate water quality. Thus, binding ceramic membranes with nanoparticles does not necessary enhance the performance of the membrane and increase the permeate water quality.

Table 2 An overview of different modification methods

Modification name			Methodology	Advantages	Disadvantage	Ref.
Physical methods	Surface coating	Dip-coating	Immersion of membrane into a solution followed by drying	- Controllable thickness	- Coating parameters should be controlled accordingly to avoid surface cracks and defects - Difficult to control the membrane thickness	Changmai and Purkait (2018), Chung et al. (2018), Zhang et al. (2019b)
		Spin-coating	Deposition of few drops of coating solution on the ceramic support followed by its rotation at constant rotational speed	- Uniform distribution of the coating materials and controllable thickness	- Large amount of the coating material is wasted	
		Spray-coating	Spraying the ceramic support with the coating solution using a spray gun	- No need for intermediate layer - Nanometer pore size can be achieved	- Multiple coating layers are need to avoid surface cracks	
		Filtration coating	Filtering the coating solution through porous membrane	- Nanometer pore size can be achieved	- Long coating time	
	Layer-by-layer self-assembly		Deposition of oppositely charged layers into the membrane surface	- Ease of operation, inexpensive and controllable thickness	- Huge amount of solutions is needed	Li et al. (2020b), Toma et al. (2015)
Chemical methods	Chemical grafting	Immersion	Immersion of the membrane in into silane agents	- Simple and fast operation	- High cost of silane agents - Solution concentration and immersion time should be controlled accordingly	Devi (2013), Kuznetsova et al. (2019), Livage and Ganguli (2001), Wang et al. (2018a)
		Chemical vapor deposition	Deposition of precursor on the ceramic membrane through chemical interaction	- Nanometer pore size can be achieved - Simple operation	- Difficult to control the membrane thickness	
		Sol-gel	Coating the membrane with a gel that is formed from the condensation of a liquid precursor	- Uniform distribution of pores	- Long modification time and high cost	
		In situ-reduction	Growth of a material over ceramic membrane hydrothermally	- Uniform pore distribution can be obtained	- Long modification time - Uncontrollable membrane thickness	
	Atomic layer deposition		A chemical deposition of a monolayer on the ceramic support in a vacuum reactor.	- Controllable thickness and pore size	- Low deposition rate - Compatible for in-organic materials only - Operation under vacuum conditions, which increases the process cost	Chen et al. (2017), Feng et al. (2018), Kemell et al. (2008)
	Atmospheric pressure atomic layer deposition		A chemical deposition of a layer on the ceramic support under atmospheric pressure	- Does not require vacuum - Easy to scale-up	- Low deposition rate	Guo et al. (2020), Shang et al. (2017)
	Molecular layer depositions		A chemical deposition of molecules that forms a chemical bond with the membrane surface group	- Compatible for in-organic and organic materials - Controllable pore size	- Operates under vacuum - Low growth rate	Liang and Weimer (2015), Wu et al. (2019)

Titania nanoparticle–incorporated ceramic membranes

Titania nanoparticles exhibit unique characteristics including high chemical resistance, good stability, antimicrobial activity, and good photochemical and catalytic properties, which qualify these nanoparticles to be utilized in membrane technology (Cheng et al. 2020; Hong et al. 2020; Keshmiri et al. 2004; Wu and Lee 2004). However, titania nanoparticles have relatively low surface area and low stability of the anatase structure at high temperatures. The photochemical and catalytic properties of titania nanoparticles can separate the organic pollutants from wastewater. Several studies reported the performance of TiO_2 ultrafiltration membrane in dye removal using bentonite as a low-cost support layer (Bouazizi et al. 2017a; Bouazizi et al. 2017c). Bouazizi et al. (2017a) studied the performance of nano-TiO_2 ceramic membrane made from natural Moroccan bentonite using the dip-coating method for dye removal. Nano-TiO_2 showed good adhesion and a uniform distribution over the support layer. The membrane was characterized to be slightly hydrophilic which might increase the membrane fouling, yet the author did not study the fouling propensity of the synthesized membrane. The rejection of Direct Red 80 was 98%, Acid Orange 74 was 85%, and Methylene Blue was 94%. Bouazizi et al. (2017c) studied the performance of nano-TiO_2 ceramic membrane made from natural bentonite and micronized phosphate using the spin-coating method for the removal of Direct Red 80. The results showed that the membrane had good mechanical strength, and the maximum rejection rate was 98% at 100 ppm feed concentration. Although it was proved that nano-TiO_2 ceramic membrane has a good rejection rate, membrane fouling is expected to be an issue due to the hydrophobicity of the membrane. The membrane fouling issue does not affect oil/water emulsion filtration applications (Suresh and Pugazhenthi 2017; Zhu et al. 2016). Zhu et al. (2016) proved that despite the fact that nano-TiO_2 thin layer is hydrophilic, the membrane fouling is weak due to the high repulsive force between oil droplets and nano-TiO_2 thin layer, which minimizes the influence of surface adsorption. The maximum total COD rejection was 97%. Thus, nano-TiO_2 ceramic membranes are effective in oil/water emulsion filtration in term of rejection and antifouling propensity property. In dye removal application, the membrane is good in terms of rejection but the membrane resistance to fouling must be enhanced further.

Silver nanoparticle–incorporated ceramic membranes

Silver nanoparticles can enhance the membrane rejection rate and increase the membrane resistance to biofouling due to their antimicrobial activity (Balkenov et al. 2020; Little Flower et al. 2019). Also, it can improve the hydrophilicity, water permeability, and selectivity of the membrane. The main challenge for using silver nanoparticles in membrane modification is that it can be released as particles or as dissolved ions into the permeate solution. This may cause a serious environmental issue because silver is a non-biodegradable element and can result in serious diseases to humans (Maharubin et al. 2019). Liu et al. (2020) evaluated the use of silver membrane technology in wastewater treatment and noticed that the production of black silver and silver sulfide particles during the regeneration process. Consequently, it is important to ensure the proper binding of silver on the membrane support.

Gao and Xu (2019) developed a super-hydrophobic and oleophilic silver–coated membrane HDT-Ag-PDA-Al_2O_3 and modified HDT-PDA-Al_2O_3 membrane for water-in-oil emulsion separation. The results revealed that polydopamine (PDA) caused the membrane to become hydrophobic and have non-adhesive behavior to the water droplet under oil. Furthermore, the deposition of silver nanoparticles facilitated thin PDA homogenous coating over the ceramic support. Among these membranes, HDT-Ag-PDA-Al_2O_3 membrane had the lowest membrane fouling, whereas the unmodified ceramic membrane had the highest membrane fouling. The rejection rate of unmodified ceramic membrane was 82.7%, HDT-PDA-Al_2O_3 membrane was 95.4%, and HDT-Ag-PDA-Al_2O_3 membrane was 98.6%. The low rejection rate of the unmodified ceramic membrane is due to its hydrophilic nature. In contrast, both modified membranes had high rejection value, but HDT-Ag-PDA-Al_2O_3 had the highest rejection rate owing to it is surface properties and the hydrophobicity of the membrane. The membrane non-adhesion to water property exhibited by HDT-Ag -PDA-Al_2O_3 membrane inhibited the formation of fouling layer. Therefore, it is recommend to use Ag-HDT-PDA-Al_2O_3 instead of HDT-PDA-Al_2O_3 membrane.

Silica nanoparticle–incorporated ceramic membranes

Silica nanoparticles are characterized by several unique features such as high specific area, high chemical purity, controllable particle size, high stability, and low production cost (Karnati et al. 2020; Sábio et al. 2019). In ceramic membrane technology, silica nanoparticles can potentially enhance the membrane performance in terms of rejection and antifouling. There are few studies on the use of silica nanoparticles in the modification of ceramic membranes (Abadikhah et al. 2019; Huang et al. 2018; Tolba et al. 2016; Tomina et al. 2019a; Tomina et al. 2017; Zhang et al. 2019a). Tomina et al. (2017) studied the performance of silica nanoparticles with amino-functionalized membrane in Ni(II) and Pb(II) microfiltration. The aim of using silica nanoparticles was specifically to act as a sorption material to adsorb heavy metals. The membrane showed a retention of 95% for Ni(II) and 20–30% for Pb(II) during the 2 h experiment. Abadikhah et al. (2019) synthesized SiO_2 nanoparticle–incorporated ceramic membrane for

Table 3 Performance of nanoparticle-incorporated ceramic membrane nanocomposite for water filtration

Nanomaterial	Application	Membrane pore size (nm)	Nanoparticle diameter (nm)	Max. Rejection %	Permeability (L m^{-2} h^{-1} bar^{-1})	Remarks	Ref.
MnO_2–CO_3O_4	Catalytic ozonation for benzophenone-3 degradation	20–50	7.05	81.2	-	- The removal of benzophenone-3 was enhanced by MnO_2-CO_3O_4 coated ceramic membrane compared with uncoated ceramic membrane	Guo et al. (2016)
Fe_2O_3	Ultrafiltration of oil/water emulsion	-	784.4	97.8	-	- Fe_2O_3-coated ceramic membrane had a rejection of 97.8% and the rejection rate of uncoated ceramic membrane was 95% - Using Fe_2O_3 for coating ceramic membrane reduced the membrane fouling propensity	Lu et al. (2016)
MgO/diatomite	Tetracycline removal	1900	50	99.7	-	-	Meng et al. (2016)
Amorphous nanosilica	Dye removal (MB)	-	10–30	99	-	- The water flux and rejection rate of dye increased as the nanosilica content on the ceramic membrane surface increased	Tolba et al. (2016)
Titania-zirconia	-	3.5	-	92	60	- The stiffness of the mixed matrix gel on the surface was improved by the addition of TiO_2 nanoparticles, which can potentially reduce the film cracking caused by the coarse surface and large pores of the support	Yin et al. (2016)
Multi-titania	Microfiltration separation of oil/water emulsion	110	-	97	-	- The hydrophilicity of the multi Titania ceramic membrane increased and decreased the membrane fouling propensity - The water flux of multi-titania membrane was lower than multi-hollow fiber membrane.	Zhu et al. (2016)
TiO_2	Ultrafiltration of dye removal	10	21	98	16.08	- The membrane showed a good stability in acidic and alkaline conditions. - The rejection rate of Red 80, Acid Orange 74, and Methylene Blue were 98%, 85%, and 94%, respectively.	Bouazizi et al. (2017a)
TiO_2	Ultrafiltration for dye removal	72	21	99	33	-	Bouazizi et al. (2017c)
Stainless steel nanoparticles	Microfiltration of BSA solution	288–324	30–60	-	213–2460	- The nanocomposite membrane mechanical strength exceeded 400 MPa. - The addition of nanoparticles reduced the pore size of the ceramic membrane, which resulting on a high-water permeation flux	Chong et al. (2017)

Table 3 (continued)

Nanomaterial	Application	Membrane pore size (nm)	Nanoparticle diameter (nm)	Max. Rejection %	Permeability (L m^{-2} h^{-1} bar^{-1})	Remarks	Ref.
Titania	Ultrafiltration for dye removal	50	10	99	117	- The clay alumina support exhibited a permeability of 850 L m^{-2} h^{-1} bar^{-1}, and Titania nanocomposite ceramic membrane had permeability of 117 L m^{-2} h^{-1} bar^{-1}	Oun et al. (2017)
TiO_2	Microfiltration for Oil water emulsion	980	338.2–477.7	99	-	- The composite ceramic membrane porosity was 43.32% - The rejection rate for oil was 99% using TiO_2-coated ceramic membrane.	Suresh and Pugazhenthi (2017)
Titania-smectite	Ultrafiltration for industrial wastewater	18	8–12	>65	80	- The turbidity removal exceeded 95% and the COD removal was 65%	Aloulou et al. (2018)
CuO	Ultrafiltration for chromium(VI)	3.2	1.15	88.8	85.51	- Green synthesis of CuO nanoparticles was successfully obtained.	Choudhury et al. (2018)
Silica/alumina	Vacuum membrane distillation	400	10–100	99.9	-	- The rejection exceeded 99.9% for 3.5 wt% NaCl solution at 70 °C	Huang et al. (2018)
SiO_2	Microfiltration of oily wastewater	525	-	95	-	-	Abadikhah et al. (2019)
CuO/TiO_2	Ultrafiltration for ciprofloxacin	-	3.5–20	99.5	218	-	Bhattacharya et al. (2019)
Ag	Oil in water emulsion filtration	-	30	98.6	-	- The rejection rate of the coated membrane was 98.6% and 82.7% using the uncoated ceramic membrane	Gao and Xu (2019)
Alumina-zirconia	Ultrafiltration for radioactive uranium removal	4.3	44	91	-	-	Kang et al. (2019)
Hydroxyapatite	Ultrafiltration for Pb(II) removal	2.8	100	99.6	79.8	-	RoyChoudhury et al. (2019)
Silica	Removal of copper, cadmium and silver	-	60–70	99	-	-	Tomina et al. (2019a)
ZrO_2	Ultrafiltration membrane	8	1.5–120	90	150	-	Yan et al. (2019)
SiO_2	Oil water emulsion separation	650	-	60	-	-	Zhang et al. (2019a)

oily water microfiltration. The results revealed that silica particles were uniformly distributed over the membrane surface. The membrane surface became smoother and more hydrophilic. A rough surface increases the friction force between the contaminants and the membrane. This will cause more accumulation of contaminants on the membrane surface. The membrane showed an excellent rejection of 95% and permeates flux of 690 L m^{-2} h^{-1} compared with 55% oil rejection and 234 L m^{-2} h^{-1} permeate flux for the unmodified ceramic membrane. Similar results were obtained by Zhang et al. (2019a), where the SiO_2 nanoparticle ceramic membrane exhibited antifouling properties and high oil rejection rate of 60% compared with 39.8% for the unmodified ceramic membrane. Silica nanoparticles are also used in membrane distillation application. Huang et al. (2018) synthesized a stable superhydrophobic ceramic membrane with silica/alumina nanoparticles using a simple preparation process without the sintering process. The rejection rate was found to be 99.9% for NaCl with a flux of 29.3 L m^{-2} h^{-1}. The silica/alumina nanoparticles did not affect the hydrophobicity of the membrane, but it reduced the porosity of the membrane to 30.7%. Thus, silica nanoparticle–incorporated ceramic membrane can be used in water remediation and distillation application. It can effectively decrease the surface roughness that will reduce membrane fouling.

Iron oxide nanoparticle–incorporated ceramic membranes

The incorporation of iron oxide nanoparticles into ceramic membranes has great potential in water filtration applications due to its high adsorption capability to organic material and heavy metals (Hocaoglu et al. 2019). Iron oxide nanoparticles can be used in acidic environments due to their excellent resistance to acids and corrosive materials (Cortalezzi et al. 2003). Iron oxide nanoparticles are nontoxic, chemically stable, and biocompatible (Bhateria and Singh 2019). Consequently, the incorporation of iron oxide nanoparticles with membranes can potentially enhance the water remediation performance. As an example, iron oxide nanoparticles can be incorporated into ceramic membranes for the purification of drinking water in a catalytic ozonation process (Karnik et al. 2005). Lu et al. (2016) studied the performance of precoated and self-forming Fe_2O_3 dynamic membrane in ultrafiltration of oil/water emulsion. The diameter of Fe_2O_3 was found to be 784.4 nm, which is higher than the pore size of the ceramic support membrane. This eliminates the possibility of the penetration of Fe_2O_3 particles through the membrane that could possibly contaminate the permeate water. Also, it was found that the dynamic membrane is more stable in pH < 8 since Fe_2O_3 particles have a positive charge, which increases the electrostatic interaction between the two layers. The highest flux recovery was obtained using a pre-coated membrane, and the lowest flux recovery was obtained using a pre-forming composite membrane. Despite the fact that self-forming Fe_2O_3 membrane exhibits a high rejection rate compared with other membranes, it had a higher fouling propensity that could be attributed to either the penetration of some oil droplets and the absorption of oil droplets to Fe_2O_3. The incorporation of iron oxide nanoparticles could enhance the water flux recovery, but it may negatively affect membrane fouling.

Zirconium dioxide nanoparticle–incorporated ceramic membranes

Zirconium dioxide (ZrO_2) nanoparticles have high chemical and mechanical stability and a hydrophilic nature (Pang et al. 2014). ZrO_2 nanoparticles have been extensively applied in polymeric membranes to reduce the membrane fouling propensity and the membrane pore size (Ng et al. 2013). In ceramic membranes, ZrO_2 nanoparticles have been used to a limited extent. Yan et al. (2019) fabricated high-performance ZrO_2 nanoparticles ceramic membrane. The water permeability of the fabricated membrane was 150 L m^{-2} h^{-1} bar^{-1} with molecular weight cut off 32,000 Da and a rejection rate exceeding 95%. Chung et al. (2018) reported that the incorporation of alumina and ZrO_2 nanoparticles into a ceramic membrane can effectively reduce the membrane pore size from 303.2 to 4.3 nm with a crack free membrane surface. The modified membrane exhibited higher membrane resistance and higher rejection rate of $CaCl_2$ from 4.1 to 58.2%. The effect of incorporating ZrO_2 nanoparticles on membrane fouling was not studied yet. Even though the addition of ZrO_2 nanoparticles to ceramic membranes can potentially enhance salts rejection, the membrane fouling must be evaluated in future studies.

Summary and future perspective

Ceramic membranes have attracted attention due to high mechanical and chemical stabilities and long lifetime. There are several fabrication methods for ceramic membranes production that have been used in most of the studies including slip-casting, tape casting, pressing, extrusion, freeze-casting, and direct foaming methods. The current research trend related to ceramic membrane fabrication is directed towards employing low-cost materials in the production process to reduce the membrane production cost. Furthermore, more investigation should be carried out related to the fabrication of ceramic membrane using freeze cast method and efforts are still needed to implement it on pilot and industrial scale. Other methods involve the use of toxic chemicals. Future research should be directed towards methods that are more environmentally friendly fabrication processes such as the freeze casting method.

The incorporation of nanoparticles into ceramic membranes can potentially add unique functionalities and enhance the membrane overall performance. Several chemical and physical surface modification methods were developed to enhance the membrane properties. Physical modification such as surface coating in which the ceramic support is coated with a material to enhance the membrane surface characteristics along with its intrinsic properties such as pore size. Filtration coating, which is one type of surface coating methods that was developed recently can reduce the pore size significantly, e.g., from 300 to 4.3 nm with a free-crack surface. However, the process might take more than 6 h; therefore, more research needs to be carried out to reduce the modification time. Physical modification is not widely used because of the weak interaction between the coating material and ceramic support, e.g., Van der Waals interaction. Consequently, the coated layer might be eliminated in long-term operation or during the cleaning process, which might involve the use of strong chemicals. Chemical modification is commonly used for ceramic surface modification due to the formation of strong covalent bonds.

Nanoparticles can be incorporated into ceramic membrane through chemical and physical modification such as MLD and surface coating methods. The incorporation of nanoparticles to the ceramic membrane may add several functionalities, which enhance the membrane performance. It was confirmed by several studies that the incorporation of nanoparticles into ceramic membrane reduces the membrane fouling and enhances the rejection rate. Each nanoparticle can add a special functionality to the ceramic membrane. Silica can decrease the surface roughness, silver add the biofouling mitigation functionality, and enhance the membrane hydrophilicity significantly, and titania showed an outstanding performance in oil/water emulsion filtration. Future research must be directed to the incorporation of low-cost nanoparticles (e.g., bentonite nanoparticles) and biosynthetically nanoparticles to investigate their performance.

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Compliance with ethical standards

Disclaimer The contents herein are solely the responsibility of the author(s).

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