Superhydrophobic Membrane Contactor for Acid Gas Removal

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Abstract. Gas–liquid membrane contactor has gained a great attention as an alternative to conventional absorption columns in acid gas removal from natural gas or post-combustion. The membrane contactor offers high mass transfer area and excellent operational flexibility. However, hydrophobic microporous membranes commonly used are still susceptible to wetting by liquid absorbents, leading to the deterioration of absorption performance in long-term operation. Therefore, many studies were recently directed to improve the membrane wetting resistant by endowing superhydrophobicity. This article then presents a review on superhydrophobic membrane development and its application for acid gas removal using membrane contactor. An overview of gas–liquid membrane contactor is firstly presented, followed by the preparation of superhydrophobic membranes. The performances of superhydrophobic membranes in acid gas absorption are then discussed, and the recommendation for future research is finally outlined. This review may provide an insight into the further development of superhydrophobic membrane contactor.

1. Introduction

Acid gases such as carbon dioxide (CO_2), hydrogen sulfide (H_2S), and other sulphuric components are frequently contained in raw natural gases or resulted from combustion of fossil fuels. The acid gases can lead to corrosion and plugging problems. Besides, the global emissions of CO_2 are believed as the main factor causing climate change and global warming, which become a global concern [1]. Whereas sulfur dioxide (SO_2) that can be resulted from H_2S oxidation is one of the major source causing acid rain. Therefore, the acid gases are necessary to be removed from the gas stream either for the operational reason or to meet the environmental emission.

Technologies for acid gases removal include absorption using solvent or solid sorbents, pressure- and temperature-swing adsorption using various solid sorbents, cryogenic distillation, and membranes. Among these methods, absorption using solvent is the most promising current method due to the high selectivity and capacity, and easy regeneration of solvent [2, 3]. Although absorption is well-established process that traditionally uses packed columns, this process has some disadvantages such as difficulty in obtaining an accurate estimate of the gas–liquid mass transfer area and a limited range

of gas and liquid flow rates due to operational problems including flooding, loading, channeling, and foaming [4, 5].

Gas–liquid membrane contactor can be an alternative technology to overcome the drawbacks. This is a hybrid process combining conventional gas absorption in liquid solvent and additional membrane as contacting device between gas and liquid phases, offering some advantages such as high and constant specific interfacial area, independent gas/liquid control with absence of entrainment flooding, downflow flooding or weeping, modularity, and low cost [6, 7]. However, the risk of membrane wettability is a major drawback for its long-term operation. Therefore, wetting phenomena and prevention in gas–liquid membrane contactor is widely investigated recently [8-12]. Different methods have been used to improve the membrane wetting resistant, including optimization of operating condition [13, 14], solvent and membrane selection [14-17], and membrane development by control the membrane pore structure [18, 19] or the membrane surface properties [20-23]. Among these methods, development of membrane with superior hydrophobicity is an attractive way with the expectation of high wetting resistant without sacrifice the high permeability and selectivity with the maintained large pore size and the selected absorption liquid.

Gas–liquid membrane contactor for acid gases removal have been reviewed in some published papers [5, 24, 25]. However, the use of superhydrophobic membrane in such application has not been reviewed in elsewhere. This paper aims to provide a summary and direction of superhydrophobic membrane contactor development for acid gas removal. A brief overview of gas–liquid membrane contactor is firstly given, followed by superhydrophobic membrane preparation method. The performances of superhydrophobic membranes in acid gas absorption are then discussed, and the future outlook is finally outlined. This review may provide an insight into the further development of superhydrophobic membrane contactor.

2. Gas–liquid membrane contactor for acid gas removal

2.1. Process Principle

Gas absorption using gas–liquid membrane contactor is an integrated technology of membrane separation and conventional absorption. The membrane used acts as a support to keep in contact gas and liquid phases and to promote the mass transfer between them [6]. A membrane with hollow fiber configuration is preferentially used [5, 24, 26-28]. The hollow fiber modules give high specific surface area, i.e. in the range of 1500–3000 m^2/m^3, whereas in conventional contactor it is in the range of 100–800 m^2/m^3 [5]. The process of gas–liquid membrane contactor is depicted in figure 1a. Gas flows on one side and the absorbent liquid flows on the other side of the membrane. Mass transfer occurs when the solute gas diffuses through the membrane and absorbed into the liquid solvent. This is nonwetted mode. Besides in nonwetted mode (gas-filled pores), the operation mode can be in wetted mode (absorbent liquid-filled pores) [29]. The former one uses hydrophobic membrane, whereas the latter one uses hydrophilic membrane. However, the nonwetted mode is preferentially used due to the lower resistance to mass transfer [29, 30].

The membrane used is a non-selective barrier, but the selectivity is provided by the absorbent liquid. Thus, the selection of solvent is same with the conventional absorption in term of solvent capability in absorbing the acid gases and easiness of regeneration. The solvents can be chemical solvent, physical solvent, or mixed-solvent [31]. Monoethanolamine (MEA) is a chemical solvent that is commonly used for CO_2 absorption due to its absorption capability in low partial pressure of CO_2 and low cost. Investigation on various absorbents showed that MEA is superior solvent in term of CO_2 removal efficiency, compared with other absorbents such as methyldiethanolamine (MDEA), 2-amino-2-methyl-1-propanol (AMP), and distilled water [16, 32]. However, AMP can be a good choice considering the energy requirement for regeneration [32]. Moreover, MEA can be degraded in the presence of SO_2 and O_2 [3]. Ammonia has been used by Resnik et al. [33] as the solvent to capture CO_2, SO_2, and NO_x simultaneously from flue gas. Besides the benefit of a single process, the ammonia can give CO_2 loading capacity three times higher than MEA. Compared with CO_2, investigation of

removal of other acid gases such as H_2S and SO_2 is rarer. It was reported that high SO_2 removal efficiency above 80% can be achieved using NaOH and Na_2SO_3 aqueous solutions [34]. Regarding the selection of solvent, additional criteria emerged in membrane contactor are surface tension and chemical compatibility with the membrane material [24, 25]. A solvent with lower surface tension gives a higher tendency to membrane wetting [17, 35]. Meanwhile, a solvent with low compatibility may deteriorate the membrane structure and increase the membrane wetting [10].
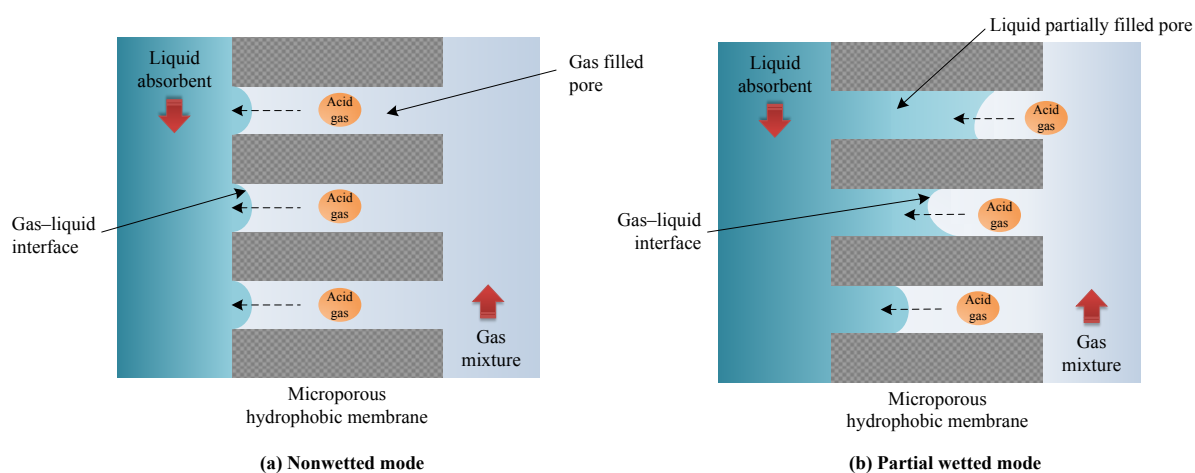

Figure 1. Scheme of hydrophobic membrane-based gas–liquid contactor. Adapted from [29, 30].

2.2. Membrane wetting

The properties of membranes used highly determine the membrane contactor performance. In the case of nonwetted mode, hydrophobic membranes from polypropylene (PP), polytetrafluoroethylene (PTFE), and polyvinylidenefluoride (PVDF) are generally used [6, 16, 34]. These membranes are commercially available. Among these membranes, PTFE membrane is more resistant to wetting [16, 36, 37]. The water contact angles of PP, PVDF, and PTFE membranes are about of 100–127°, 92–130°, and 113–139°, respectively [4, 37, 38]. Even though the membrane used is intensive hydrophobicity, such as PP, PTFE, and PVDF, the absorbents, especially aqueous solution of organic compounds such as alkanolamines, can penetrate into partial pores of the hydrophobic membrane, that is partial wetting mode (figure 1b) [11, 30, 39]. In this case, the gas–liquid interface is pushed to inside the membrane phase. The partial wetting may change the membrane morphology, decrease the membrane hydrophobicity, and in turn deteriorate the membrane performance.

Some studies have reported the morphological changes due to the intrusion of absorbent into membrane pores. Barbe et al. [40] showed that after exposed to water for 72 h, the microporous PP membranes have overall increases in porosity, pore length, and pore equivalent diameter. The increase of pore size becomes more significant with the immersion using alkanolamine solution such as MEA and MDEA [10]. Besides, the interaction between the membrane and absorbent also reduces the surface hydrophobicity which indicated by a reduction of contact angles. The PP hollow fiber membranes have a significant reduction of contact angles, i.e. from 121.6° to 90.8°, when immersed in 30% MDEA for 60 days. The increase of membrane pore size and the decrease of membrane hydrophobicity lead to wetting in a greater extent.

The membrane wetting even the partial wetting can increase the membrane resistance significantly due to the liquid phase occupying the membrane pores [11, 41]. Rangwala [41] found that even below 2% liquid-filled pores, the membrane resistance could be as high as 60% of the total mass transfer resistance. This would result in significant decrease of acid gas transfer rate. It was reported that

partial wetting of PP membrane pores by MEA absorbent caused a significant decrease in mass transfer rate of CO_2 (i.e. decreased to 59% of initial value after 14 days operation time) [42].

The membrane pores may be wetted by absorption liquid when the transmembrane pressure (pressure difference between the liquid and the gas) exceeds the critical transmembrane pressure or breakthrough pressure. Following Laplace–Young equation [43], the breakthrough pressure depends on membrane properties (i.e. pore size), absorption liquid properties (i.e. surface tension), and interaction between the membrane material and absorption liquid (i.e. contact angle). Using membrane with smaller pore size may reduce the membrane wetting. However, it may also result in decreased permeability. Meanwhile, increase the absorption liquid surface tension by reducing the solution concentration may reduce the selectivity. Increasing the contact angle by using superhydrophobic membranes is considered to be an effective way to prevent the penetration of absorption liquid.

3. Superhydrophobic membrane preparation and characterization

The term of a superhydrophobic surface is recently emerged although the investigation on water repellent or highly hydrophobic surfaces has been conducted since a long time ago. In a common agreement, a superhydrophobic surface is characterized by water contact angle (WCA) above 150° and sliding angle (SA) and hysteresis contact angle (HCA) below 10° [44, 45]. To obtain the high WCA, using low surface energy material only without concerning the surface morphology is insufficient, but it is necessary to be combined with the appropriate rough surface. Using this concept, the superhydrophobic membrane preparation is widely developed. In this paper, a review is more focused onto superhydrophobic membrane for membrane contactor that the membrane is usually microporous.

3.1. Inorganic membranes

Among the inorganic materials, ceramics from metal oxides such as alumina, zirconia, and silica are the most used materials for membrane preparation. These materials are hydrophilic in nature due to the presence of hydroxyl (–OH) groups on the surfaces. On the membrane surface, the liquid water can pass through at a fast rate [46]. Therefore, the ceramic membranes need to be surface modified to improve the hydrophobicity, making them are suitable for membrane contactor. For this purpose, surface modification by direct grafting using low surface energy materials such as fluoroalkyl silane (FAS) is commonly used [46-49]. Usually, the ceramic membrane is immersed in FAS solution for a certain time to allowing the grafting process. During the grafting, the OH groups on the ceramic membrane surface react with Si–O–alkyl groups of the silane [50]. Modified ceramic membranes having WCA higher than 150° with enhanced liquid entry pressure of water (LEPw) have been successfully obtained [47, 49]. Studies of the superhydrophobic ceramic membrane are summarized in table 1.

Table 1. Superhydrophobic ceramic membranes prepared by FAS grafting method for membrane contactor

Ceramic Membrane	Grafting parameter	WCA (°)	LEP$_w$ (bar)	Process	Ref.
alumina tube with ZrO_2 layer	FAS: 2 wt.% grafting time: 24 h	153	6.5	CO_2 absorption using MEA	[47]
kaolin-alumina hollow fibre	FAS: 0.01 mol/L grafting time: 4 h	142	2.5	CO_2 absorption using water	[48]
γ-alumina membranes on α-alumina support	FAS: 0.1 mol/L eroding time*: 5 min grafting time: 24 h multiciplity: 4 times	164.5	-	-	[49]

*The membrane was eroded in NaOH

3.2. Polymeric membranes

Differ from inorganic membranes that the superhydrophobization is achieved by surface modification, the polymeric membranes can be superhydrophobized either by surface modification or by direct processing [51, 52]. In direct processing, the superhydrophobic modification can be achieved during the preparation process. Meanwhile, the surface modification can be performed through one-step or more, depending on chosen material and method.

3.2.1. Improvement of membrane preparation process. In this direct processing method, hydrophobicity can be enhanced by improvement of phase separation process, blending method, or electrospinning. In the former way, the enhancement of hydrophobicity is attributed to the surface morphology which is controlled by solution and process parameters. It has been reported that highly hydrophobic membrane with porous structure can be obtained by using coagulant which led to delay demixing phase separation [53], by using long vapor exposure time facilitating crystallization process [54], and by using high air temperature and low PVDF content [55]. Blending method is a simple way to enhance the hydrophobicity of membrane by blending low surface energy material such as surface modifying macromolecule (SMM) [20, 21, 56-60] or by blending nanoparticle [61] into casting solution. However, there isn't a published work reporting superhydrophobic membrane prepared using the methods. Electrospinning method has been widely investigated in superhydrophobic membrane preparation, either from melt or solution of polymer, with or no addition of nanoparticles. This method has been used for superhydrophobic membrane preparation from the solution of polystyrene (PS) [62, 63], PVDF [64], and PP [65]. This method has also been used for superhydrophobic inorganic-polymer composite membrane preparation from PVD/SiO_2 [66-68], polyurethane (PU)/terminal fluorinated polyurethane (FPU)/carbon nanotubes (CNTs) [69], and polyvinylidene fluoride-co-hexafluoropropylene (PcH)/CNTs [70].

3.2.2. Surface modification of existing membrane. Some methods of surface modification have been proposed to obtain a superhydrophobic membrane. Plasma treatment, coating with roughened hydrophobic polymer film, and chemical vapor deposition (CVD) are methods that can render superhydrophobicity on membrane surface through simultaneous surface roughening and hydrophobization using low surface energy materials. These methods are also called one-step surface modification. The rough surface of plasma-treated membrane is resulted from an etching process during the plasma treatment, while the low surface energy is due to fluorine deposition. Plasma treatment using CF_4 [71, 72], PTFE [73, 74], and benzene [75] have been successfully used to prepare superhydrophobic polymer membranes. Besides choosing of plasma reagent, other crucial parameters in this method are plasma treatment time and working power. Excessive treatment could result in no enhancement or even some deterioration in superhydrophobicity [71, 73, 76, 77], thus the optimization of both process parameters is important. Coating with roughened hydrophobic polymer film is also an attractive method for superhydrophobic polymer membrane preparation, which the surface roughening process can be accomplished by using non-solvent [38, 78-81] or nanoparticles [82, 83]. A few works reported superhydrophobic polymer membrane preparation by CVD method [84]. Besides the one-step surface modification, the superhydrophobic membrane can also be produced by two-step surface modification, which the sequence can be surface roughening followed by hydrophobization using low-surface-energy material or vice versa. In this regard, surface coating with nanoparticles followed by coating with fluorosilane compounds is often used. Superhydrophobic membrane preparation with surface roughening by using TiO_2 [85], silver [86], and silica [87] nanoparticles have been reported. Studies on preparation of superhydrophobic membrane from polymeric materials for membrane contactor are summarized in table 2.

Table 2. Superhydrophobic polymeric membranes for membrane contactor

Membrane Base Material	Preparation/Modification Method	WCA (°)	LEP$_w$ (bar)	Process	Ref.
PVDF	SMM blending method	92	3	CO_2 absorption using water	[21]
PVDF	SMM blending method	96.2	8	CO_2 absorption using water	[60]
PDMS/PVDF	Electrospinning and FAS modification	150	-	CO_2 absorption using AMP/PZ	[88]
PP	CF$_4$ plasma treatment	143	-	CO_2 absorption using PZ, MDEA, and AMP	[77]
PP	Plasma treatment using PTFE	151	0.7[*]	CO_2 absorption using MEA	[73]
PVDF	Coating with rough LDPE layer	152	-	CO_2 absorption using MEA	[80]
PP	Coating with rough PP layer	158	-	CO_2 absorption using MEA	[78]

[*]The liquid entry pressure of 2-propanol

4. Performances of superhydrophobic membrane contactor in acid gas removal

4.1. Performances

It has been proved that superhydrophobic modification increases the WCA and LEP, thus it is expected that the superhydrophobic membrane has a higher resistant to wetting than a hydrophobic one. As the superhydrophobic membrane isn't wetted, the gas–liquid interface should be maintained at the pore mouth, which means that there is no additional membrane resistance imposed by the stagnant liquid phase. With this condition, a high mass transfer rate of the acid gas can be achieved.

Investigations of membrane contactor in acid gas removal showed that either superhydrophobic polymeric membranes or superhydrophobic ceramic membranes exhibited a higher and more stable flux than commercial hydrophobic membrane [47, 74, 77, 78, 80]. It was reported by Lin et al. [77] that the CO_2 absorption flux of the superhydrophobic PP membrane contactor only dropped from 7.4 x 10^{-4} mol/(m^2 s) to 7.1 x 10^{-4} mol/(m^2.s), even after 30 days. Whereas hydrophobic PP membrane and hydrophobic PVDF membrane exhibited a significant decrease of CO_2 absorption flux from 6.84 x 10^{-4} mol/(m^2 s) to 7.4 x 10^{-4} mol/(m^2 s) and from 7.05 x 10^{-4} mol/(m^2 s) to 5.52 x 10^{-4} mol/(m^2 s), respectively. The high and stable absorption flux could be attributed to the decrease in wetting degree with the increase of membrane hydrophobicity [73, 77]. An estimation of wetting degree showed that superhydrophobization of PP membrane could decrease the pore wetting degree more than 40% with the increase of membrane mass-transfer coefficient about 207% [73].

However, one matter should be noted is that the benefit of superhydrophobicity rendered in the membrane isn't offset by the negative effect of the superhydrophobic modification to the membrane characteristics such as pore size, porosity, and membrane thickness. As studied by Lv et al. [78], superhydrophobic PP membrane prepared by roughened polymer film coating using solvent/non-solvent method exhibited a slight decrease in CO_2 flux within the first 7 days, approximately 86% of the initial value. Moreover, the CO_2 flux of modified membrane was lower than that of unmodified membrane in the first 6 days, as shown in figure 2. This is due to an increase in the membrane thickness and a decrease in the surface porosity after the superhydrophobic modification. Thus, the superhydrophobic modification that maintains the membrane thickness and porosity is highly desired.
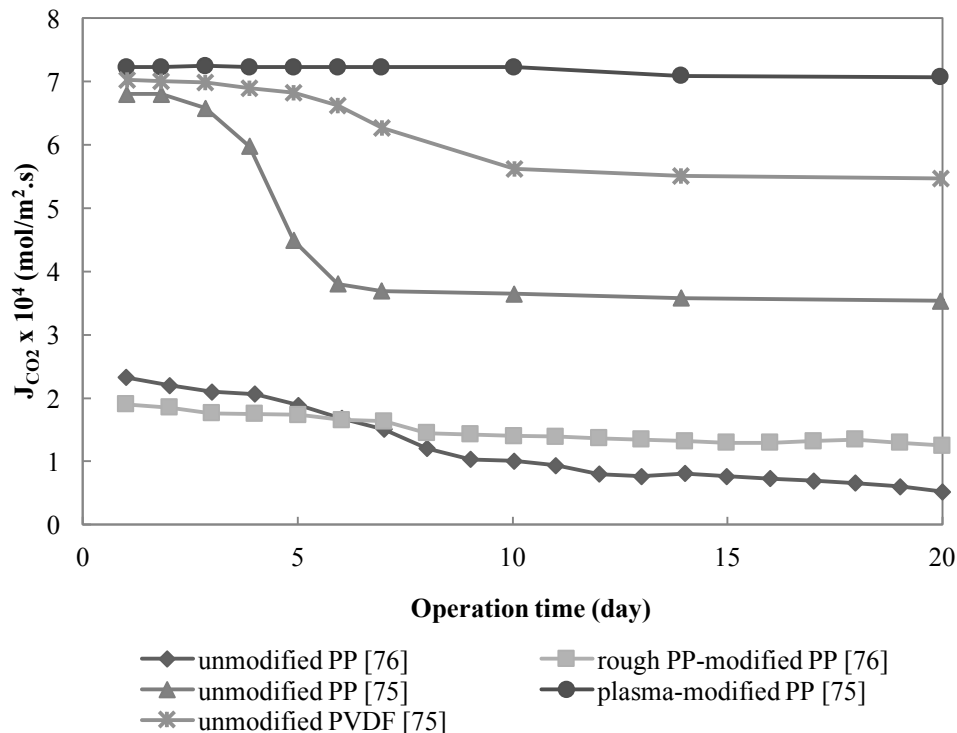
Figure 2. Long-term performances of superhydrophobic membranes compared with hydrophobic membranes [77, 78].

4.2. Self-cleaning function
Fouling is one of the major problems in porous membrane processes such as microfiltration and ultrafiltration [89-92]. In membrane contactor, the fouling isn't significant as much as in membrane filtration since there is no convective flow through the membrane pores. However, in industrial applications such as coal-fired power plant, gas and liquid streams with suspended particles can cause plugging due to the small membrane contactor diameter. Superhydrophobic surface has been acknowledged for its self-cleaning ability [93, 94]. Therefore, the use of superhydrophobic membrane is intended to mitigate the membrane fouling in membrane contactor. Regarding this case, only a few reports that can be found. Yu et al. [47] showed that ceramic membrane with superhydrophobicity exhibited a better anti-fouling ability than PP membrane without superhydrophobicity. The superhydrophobic ceramic membrane could be easily cleaned easily carbon powder, whereas for the hydrophilic ceramic membrane and hydrophobic membrane, the carbon powder could not be effectively removed. Moreover, one-month operation using flue gas from thermal power plant showed that on the superhydrophobic ceramic membrane, there was no obvious dust. Meanwhile, the PP membranes were fouled by dust in the flue gas.

5. Conclusion and recommendation
Membrane contactor offers some advantages over conventional technologies which makes it gained a great attention for acid gas removal. However, membrane wetting by liquid absorbent becomes a drawback which deteriorates the membrane performance. To address it, superhydrophobic membrane contactor was then developed, either from polymer or inorganic materials. Superhydrophobic polymer membrane can be prepared by either direct processing or surface modification. However, surface modification of hydrophobic polymer membrane by plasma treatment or solvent/non-solvent coating method is more likely to be used to prepare superhydrophobic membrane contactor for acid gas

removal. For inorganic membrane, surface modification is obviously required to render superhydrophobicity due to the hydrophilic nature. It has been reported that the increase of hydrophobicity led to a higher and more stable flux compared to the unmodified membrane. However, additional of membrane thickness and pore blocking due to the surface modification need to be concerned because these offset the benefit of superhydrophobicity. Besides offering wetting resistant, superhydrophobic modification also gives self-cleaning ability. It was newly investigated in membrane contactor which is important for industrial flue gas streams. For future improvement of membrane contactor, development of superhydrophobic microporous membrane with main concern on the stability of superhydrophobicity, thickness addition, loss in pore size and porosity is very necessary. As the flue gas from the combustion process is in high temperature, and various absorbents are being developed, the thermal stability and chemical stability of the superhydrophobic membrane also need to be investigated.

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