

CO₂ and H₂S Separation Technologies

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The emerging economic activities, modernization of technology and progressive transition from coal utilization to low-carbon fuel drive the rising energy demand. Natural gas is well-known as clean energy resources for electricity generation and fuel for transportation. Currently, CO₂ and H₂S content in gas fields accounts for up to 90% and 15%, respectively. Apart from fulfilling the market demand, the removal of these contaminants from natural gas is critical due to their corrosive natures, low heating value of natural gas and greenhouse gas effect. To date, numerous gas fields remain unexplored due to high presence of contaminants. Several technologies such as absorption, adsorption, membrane separation and cryogenic distillation have been developed for natural gas sweetening. This entry comprehensively discussed the mechanisms and state-of-the-art of these conventional technologies.

absorption

adsorption

cryogenic distillation

high CO₂ separation

H₂S removal

membrane separation

1. Absorption

At the beginning of the 21st century, the first solvent that was used for the absorption process was a carbonate solution that was applied in dry ice plants to separate CO₂ from flue gas ^[1]. Sodium carbonate solutions were rapidly phased out after the introduction of alkanolamine, as this solvent absorbs CO₂ faster and can attain extremely high CO₂ removal efficiency ^[1]. Starting in 1930, primary generation alkanolamine solvents such as diethanolamine (DEA), (MEA) and diglycolamine (DGA) were developed for the CO₂ removal process ^{[1][2][3]}. Then, secondary alkanolamine solvents such as diethanolamine (DEA) and diisopropanolamine (DIPA) were invented as alternatives to MEA ^[4]. The difference between primary and secondary is that the primary alkanolamines contain hydrogen atoms directly bonded to nitrogen ^[4]. A tertiary alkanolamine solvent, which is methyldiethanolamine (MDEA), was proposed by Frazier and Kohl to promote the selectivity of H₂S ^[4]. In 1983, 2-amino-2-methyl-1-propanol (AMP), which hindered amines, was developed at Exxon Research and Engineering Company ^[5]. From 1995 to now, there has been a lot of research into sterically hindered amines as potential alkanol amine absorbents. There are also lots of studies that report the promoters, such as piperazine, potassium carbonate, mono ethanol amine (MEA) and diethanolamine (DEA), that can be blended with amine solvent, such as MDEA, to enhance the reaction rates of the absorption process ^[4]. Starting in 2005, the interest in the usage of amino acid for acid gas removal started to develop in using the sodium or potassium salt glycine (NaGly), which

is the simplest primary amino acid, for CO₂ capture [6]. Recently, in 2003, ionic liquids, ILs, were branded as “solvents of the future”, as they have the potential as alternative solvents for acid gas removal [7][8].

The absorption process is the process where a gas mixture comes into contact with a liquid (solvent). The gas phase is transported into the liquid phase in the absorption process. This process has been used in the natural gas industry for 100 years for the gas sweetening process to separate sour gas from natural gas. It is used to remove any impurities or contaminants in gas mixtures to recover valuable products. This process can be classified into two types of solvents, which are chemical solvents and physical solvents. In chemical solvent processes, alkanolamines or alkaline salts with weak acids, such as sodium or carbonates, are used as solvents for the absorption process. The sweetening of sour gas via absorption is referred to in **Figure 1** by using alkanolamines. The sour gas is injected through a tower and makes contact with alkanolamine solutions for the absorption process to happen. The solvent absorbs sulfur compounds from H₂S and release the effluent gas. Then, the desorption process, or regeneration process, is performed to strip the acid gases from the solvent at low pressure or high temperature.

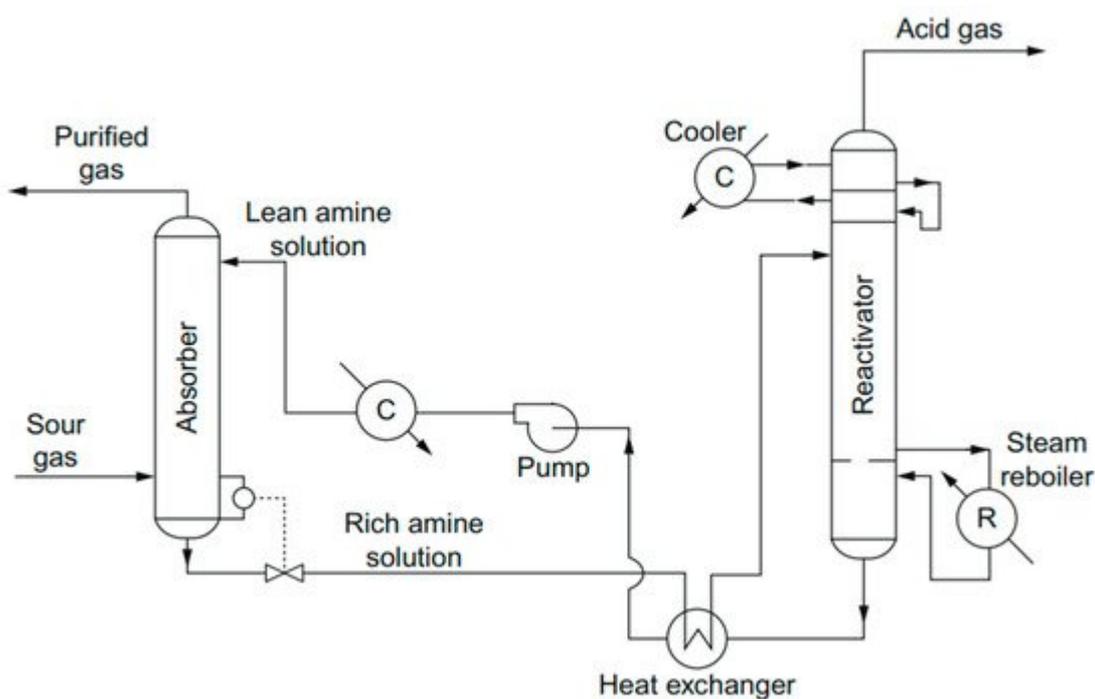


Figure 1. Schematic diagram of the amine sweetening process [9]. Reprinted with permission from Speight, J.G. (2015). Copyright 2021 Elsevier.

In physical solvent processes, organic solvents such as methanol, N-Methyl-2-Pyrrolidone, Poly(Ethylene Glycol) Dimethyl Ether, Sulfolane and Diisopropanolamine are used to absorb H₂S and CO₂ depending on the partial pressure. The performance of the absorption is increased at high partial pressure and low temperature. This process does not have any corrosive effects and also does not require the addition of heat in the stripping process. In addition, physical solvents can strip off impurities without any additional heat. However, this process is not

suitable for bulk absorption of CO₂ gas, as it requires high pressure and low temperature [10]. Figure 2 shows the flow diagram for the physical absorption process.

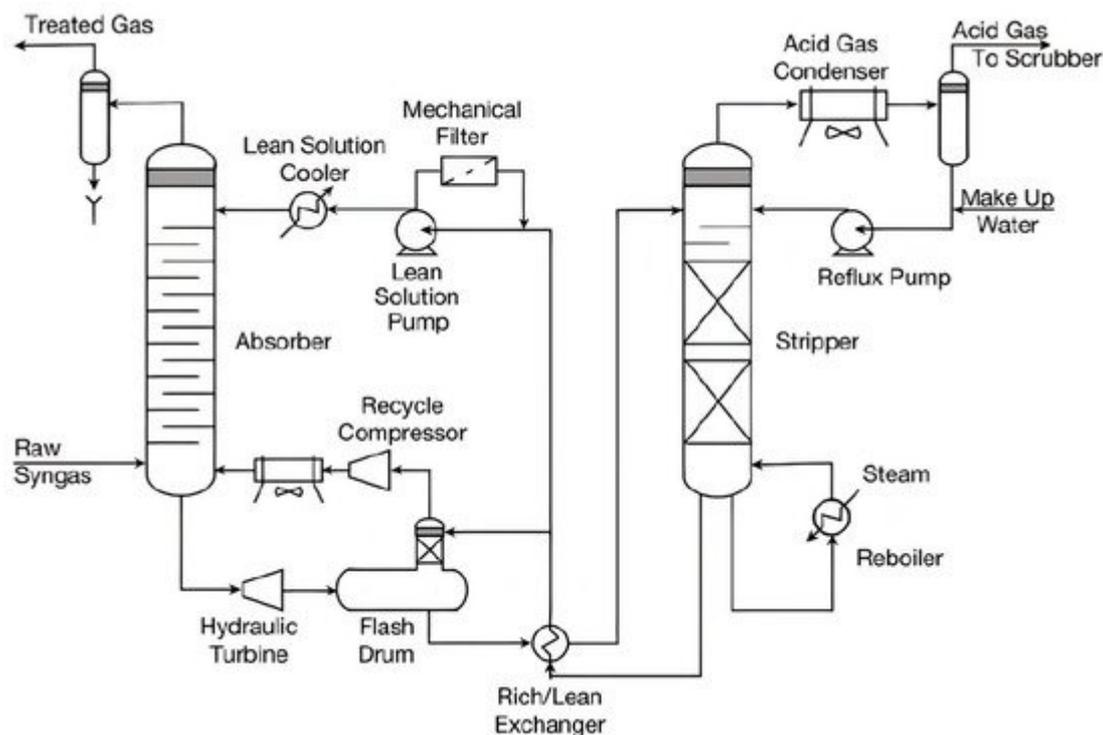


Figure 2. Schematic diagram of common physical absorption process (Selexol process) [11][12]. Reprinted with permission from Miller, B.G. (2011). Copyright 2021 Elsevier.

In chemical absorption, an aqueous solution of alkanolamines is one of the most effective absorbents that is still widely used for the absorption process for natural gas purification. Nevertheless, this solvent has many drawbacks, including foaming, high cost, high energy consumption for regeneration, high toxicity and high corrosivity. The drawbacks faced by amine-based solvents can be catered back by using aqueous amino acids, as they have the same functional group. Amino acids are one of the alternative solvents that have many advantages, such as high stability towards oxidative degradation, high chemical reactivity with carbon dioxide and low vapor pressure [13]. Due to their physical properties, amino acid solvents have high reactivity towards CO₂ compared to conventional solvents such as MEA and MDEA [14]. Hu G et al. stated that amino acids such as lysine, proline and sarcosine have a larger reaction than monoethanolamine (MEA) for the absorption of CO₂ [13]. The absorption of CO₂ is influenced by the ionic strength, pH and cations of weak bases such as sodium and potassium for the amino acid to react with the CO₂ gas [13]. It was also reported that the use of amino acids blended with amine shows better absorption of CO₂ than the neutralization of amino acids with potassium hydroxide [15]. Nevertheless, the combination of these solvents for large-scale absorption application is not economical because it requires higher regeneration temperature, as stated in Erga et al. [16]. Knuttila et al. [1] found that the use of sarcosine requires a higher energy cost compared to MEA.

Ionic liquids (ILs), particularly functional ILs, are known as unique absorbents for the removal of H₂S from gas mixtures because they can absorb a large amount of H₂S at high pressure. Ionic liquids (ILs) have been widely studied for CO₂ and H₂S absorption, as they have several advantages, such as environmentally friendliness, extremely low vapor pressure, tuneable structure and high thermal and chemical stability [17]. Most of the research found that functionalized ILs can act as catalysts or substitutes for amino-based, metal-based, substituted benzoate-based and pyridinium-based solvents for H₂S removal. However, the major drawbacks of IL-based solvents are high cost, high viscosity, complicated synthesis process and that they cannot be applied to a large-scale industry. In addition, most industries still prefer gas–liquid systems because this process requires low cost and low energy consumption during solvent regeneration. Overall, the previous literature reviews show that CO₂ and H₂S removal using green solvents such as amino acid and ionic liquid are not economical for commercial application due to high cost and energy consumption during operation and regeneration. Moreover, most of the studies for green solvents such as amino acid only focus on CO₂ removal, whereas there are only a few literature studies about H₂S removal [17].

2. Adsorption

The first systematic research on adsorption application began in 1773, conducted by Scheele to observe the adsorption of air by using charcoal via a volumetric apparatus [18]. The first commercial application of the adsorption process was purifying white sugar. Back in 1783, charcoal had been used as the adsorbent to remove impurities and contaminants from sugar [19]. However, during that era, people were not concentrating on the improvement of adsorbent properties and only relied on the natural adsorbent properties such as charcoal, clay and peanut hull [20]. In the 19th century, the study to improve the properties of adsorbent started along with the industrial revolution. The researchers concentrated on the surface area and the porosity of the adsorbent [21][22]. The idea to improve the adsorbent based on the porosity and the surface area began after Chappius measured the isotherm from the adsorbent layers [23].

Adsorption has had numerous applications in industry in the past 30 years, such as in the purification of gas mixtures, mainly in petrochemical, environmental, electronic and medical industries, due to its unique characteristics [24]. There are many types of adsorbents available with different pore sizes and selectivity that create flexible designs to separate and purify gas mixtures for particular desired goals. The impurities removal application includes organic and inorganic impurities removal, electronic gas purification, air pollution control, gas drying, solvent vapour recovery and nuclear waste management. Moreover, the gas separation application includes methane and carbon dioxide separation, hydrogen recovery, air separation, alcohol dehydration, production of ammonia synthesis gas and isoparaffin separation [25]. The well-known technologies commercially used are pressure swing adsorption (PSA), temperature swing adsorption (TSA), vacuum swing adsorption (VSA) and electric swing adsorption (ESA) [26][27]. For NG application, TSA is used for gas purification, whereas bulk gas separation often uses the PSA process [24].

PSA technology was first patented in 1932 by Charles Skarstrom for oxygen enrichment, in which the cyclic adsorption process was employed using four main steps that included feed, blowdown, purge and pressurization

[28]. In PSA, the acid gas stream that contains high CO₂ makes contact with spherical adsorbents packed in the column which are typically arranged in parallel to maximize the energy efficiency [29]. The feed gas is initially fed into the column and pressurized to a pressure higher than atmospheric pressure. CO₂ is selectively bound and adsorbed on the surface of the adsorbent at high pressure and low temperature until equilibrium is achieved. The adsorbent is saturated with adsorbed CO₂. Hence, the regeneration of the adsorbent takes place by restricting the gas flow and depressurizing the column to liberate the CO₂ from the surface of the adsorbent [30]. In contrast, the regeneration of adsorbent in VSA is performed at vacuum condition by reducing the pressure below atmospheric pressure, whereas in TSA, CO₂ is liberated by increasing the temperature at a constant pressure [31][32][33]. Meanwhile, ESA is named due to the low-voltage electric current being introduced to heat the adsorbent by the direct Joule effect [34]. The difference between TSA and ESA is that ESA heats the adsorbent by using electric power while TSA heats the adsorbent by using waste heat from noncondensable gas [35].

There are a wide range of adsorbents that have been utilized according to their level of selectivity attraction towards contaminants to be removed. Furthermore, adsorbent enhancement could be achieved by synthesizing and impregnating other compounds onto the surface of the adsorbent. There are two types of adsorption approaches, either physisorption or chemisorption. The physisorption process is a physical process directed by weak Van Der Waals bonds between the adsorbent and adsorbate [36]. On the other hand, chemisorption is governed by the bond formation between the functional groups that attach at the surface of the adsorbent and the adsorbate [36]. Due to the attached functional groups, chemisorption is generally more efficient than physisorption, specifically when using basic and oxygen-containing groups, as CO₂ is acidic in nature; hence, these functional groups play a major role in the removal of CO₂ from natural gas [37]. The specific surface area of the adsorbent also has a major effect on the adsorption process. It is well-known that the higher the specific surface area, the higher the adsorption capacity. The major disadvantage of the adsorption system is waste generation. One of the solutions to overcome this issue is introducing the regeneration process to the adsorbent. However, regeneration may not be effective in the long run. The highest number of regeneration cycles obtained in the literature is 5 cycles, after which removal efficiency starts to decrease [38].

The interest in H₂S removal in natural gas processing units enhanced several studies to develop regenerable solid sorbents. However, the low adsorption capacity of commercial solid adsorbents needs a large amount of adsorbent bed while frequent disposal of saturated adsorbents is one of the major environmental concerns. The adsorbent-filled fixed bed reactors for H₂S capture may operate according to two different mechanisms, either physisorption or chemisorption [39]. Zinc and copper oxides supported on porous adsorbent silica have attracted recent attention for H₂S capture due to the potential for the combination of sulfidation thermodynamics and active metal oxides [40][41]. The porous adsorbent is generally synthesized from various composite materials based on metal foams [42], zeolites [43], laterites [44], kaolin [45], silica [46], carbon of miscellaneous sources [47] and other solid phases impregnated with metal salts and their mixtures in order to enhance the adsorption capability of the fixed bed. Bahman Elyasis et al. stated that Cu-ZnO nanoparticles impregnated with mesoporous silica show a significant impact on H₂S removal. The amount of H₂S captured is 75 mg/g_{sorbent}, while the commercial ZnO only captures about 34 mg/g_{sorbent} [48].

NG often contains significant amounts of CO₂ and H₂S that have to be reduced to less than 1% for CO₂ and 4 ppm for H₂S to meet the fuel gas specifications for pipeline transportation [49]. Adsorption also has been recognized to be an energy-efficient technology for CO₂ and H₂S removal, provided that the material exhibits high and stable adsorption capacity along with excellent selectivity toward acid gases. The early work for simultaneous adsorption separation of CO₂ and H₂S from methane was performed by Huang et al. by using amine impregnated on silica xerogel. They found that the excellent performance in terms of CO₂ and H₂S adsorption by amine-modified silica is due to large amounts of amine groups on the surface resulting from their high surface areas [50]. However, the CO₂ and H₂S selectivity was not mentioned. Recently, Ma et al. reported that polyethylenimine (PEI) impregnated with silica is capable of selective adsorption of both CO₂ and H₂S. However, it was stated that not only is the adsorption of CO₂ diffusion limited, but also the optimum temperature for CO₂ adsorption is 75 °C, whereas the H₂S is 25 °C. Moreover, CO₂ seems to strongly inhibit the H₂S adsorption at room temperature [51].

The usage of amines as activators for the adsorbent demonstrates certain limitations to CO₂ and H₂S separation because by nature the CO₂ has higher acidity than H₂S. Due to both CO₂ and H₂S being electron acceptors, they have a strong effect on the amines group which leads to reactive and nonreactive adsorption, hence restricting the efficiency of CO₂ and H₂S separation. This phenomenon occurs due to the adsorbent being more selective toward H₂S than CO₂ [52]. Most of the commercial gases used in industries consist of both acid gases, such as CO₂ and H₂S, and polar species, such as H₂O or CO. A study performed by Billow et al. investigated the reaction of CO₂ and H₂S adsorption on LTA and FAU zeolite. They found that H₂O was the highest preferred by the adsorbent, followed by H₂S and CO₂ [53]. Hence, a proper study needs to be conducted to investigate the selectivity ratio of H₂O, CO₂ and H₂S for better separation efficiency.

3. Membrane

Over the past few decades, membrane technology emerged as a sustainable process in various applications such as wastewater treatment, food technology, medicine, pharmaceuticals and petrochemicals [54]. In the 1980s, the first commercial membrane technology was introduced by Air Product for air and hydrogen production using polysulfone-based hollow fiber membrane [55]. Presently, membrane technology continuously evolves as a mature and competitive technology that contributes to 10% of the market share in natural gas purification [56]. According to Mordor Intelligence [57], the gas separation membranes market will progress at a compound annual growth rate (CAGR) of 5% between 2020 and 2025. The expansion of the membrane separation market is fueled by the growing demand for CO₂ removal attributed to the rising environmental concerns and low monetization of sour gas fields. Membrane-based acid gas removal technology is ideal for the purification of highly sour natural gas with CO₂ concentration of more than 50 mol% and H₂S up to 10 mol% [58]. Moreover, the simplicity of the membrane process enhances its potential for installation at offshore platforms for gas processing with a low flow rate of <6000 Nm³/h [59]. The emerging development of membrane technology is rendered by its flexibility for scale-up, small footprint and high energy efficiency. Compared to absorption, membrane separation is more economical and environmentally friendly, as it does not require the utilization of any chemical solvent, which eliminates the need for compression to regenerate solvent.

Membranes are generally classified based on material (e.g., glassy or rubbery polymers) and configuration (e.g., flat sheet, hollow fiber or spiral wound). Gas separation is achieved through selective gas transport across the semi-permeable barrier. **Figure 3** shows the selective separation of CO₂ and H₂S from natural gas using a hollow fibre membrane system. Gas transport in membrane separation is driven by the pressure and concentration gradient. Theoretically, gas transport behavior in membrane separation is governed by the solution–diffusion mechanism where solubility (S) and diffusivity (D) play important roles to determine gas permeability. The types of membrane material and properties of the permeants, such as condensability, influence gas transport across the membrane. In addition, the molecular size of the gas molecules significantly affects the gas diffusion rate in the order of C₃H₈ > C₂H₆ > CH₄ > N₂ > O₂ > CO₂ > H₂S > H₂ [60]. Membrane separation also requires sufficiently high pressure to drive gas separation. In addition, the pretreatment process is required prior to gas separation to remove any water or heavy hydrocarbons from the feed gas to avoid membrane fouling [61].

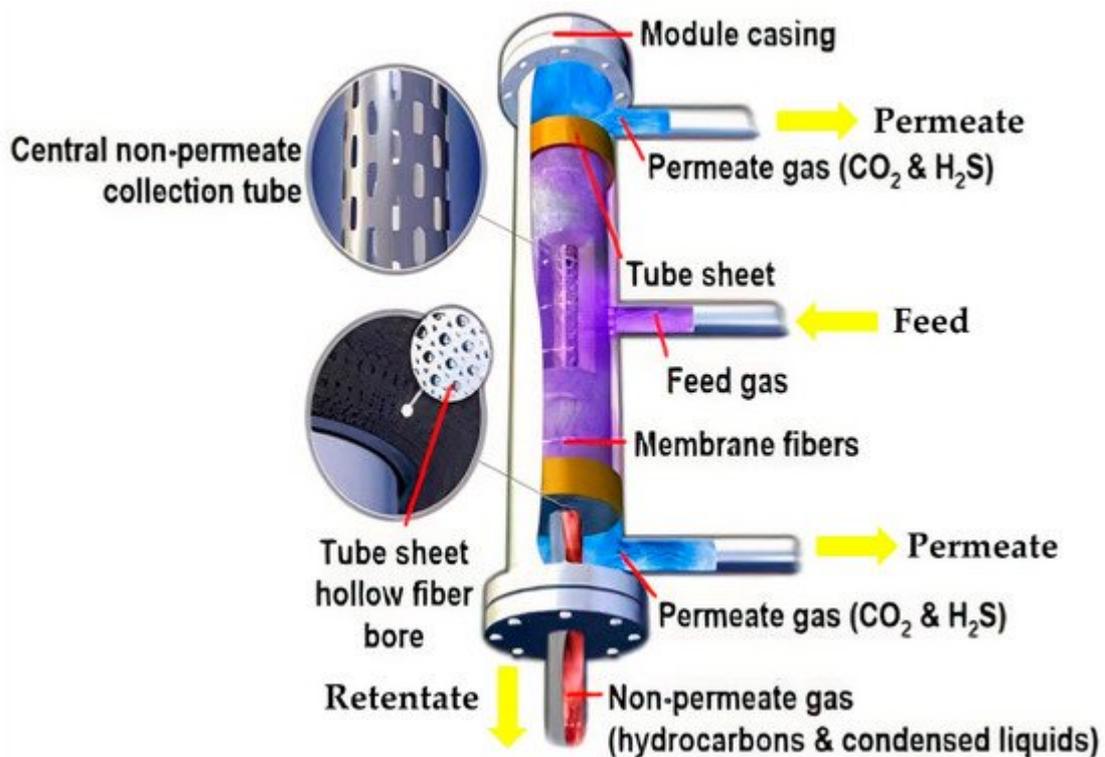


Figure 3. Illustration of selective separation of CO₂ and H₂S from natural gas using hollow-fibre membrane module [62]. Reprinted with permission from Sanghani, P. et al. (2020). Copyright 2021 Society of Petroleum Engineers (SPE).

Commercial membranes such as cellulose acetate [63], polyimide [64][65] and polyamide are the leading materials used in membrane gas separation due to their robustness, superior permeability and selectivity, as well as excellent durability. Glassy membranes such as cellulose acetate are typically effective for CO₂ separation, while rubbery membranes are typically effective for H₂S separation. Cyanara-NATCO [63] developed a cellulose triacetate (CTA)-based hollow fiber membrane system for natural gas sweetening in the offshore processing facility at the Cakerawala platform located in the Gulf of Thailand. The membrane system is capable of handling 228.89 m³/s process gas and successfully reduces the CO₂ content from 37% to 15%. Recently, Nafisi et al. discovered the

potential of fluorinated polyimides (6FDA) for CO₂ separation, where CO₂ permeability attained as high as 1468 Barrer with CO₂/CH₄ selectivity of 22.6 [66]. Besides polymeric membranes, zeolite membranes such as silicalite-1 [67], SAPO-34 [68][69] and DDR [70][71] also demonstrated a promising performance for separation of CO₂ from CH₄. Zeolite membranes are known for their high selectivity, which is attributed to the uniform pore structures. Poshuta et al. reported a separation factor of 36 with CO₂ permeance of 2×10^8 /mol m⁻² s⁻¹ Pa⁻¹ by using SAPO-34 membrane [72]. Furthermore, Cui et al. demonstrated higher performance of zeolite T membrane, where CO₂ permeance and CO₂/CH₄ selectivity was found to be 4.6×10^8 /mol m⁻² s⁻¹ Pa⁻¹ and 400, respectively [73]. The application of zeolite membrane is presently confined to lab-scale study due to its brittle nature and complex fabrication procedure which hamper its commercial opportunity.

On the other hand, some membrane materials also demonstrate promising capability to handle contaminants other than CO₂, such as H₂S. Currently, the studies on H₂S removal using membrane separation are usually confined to low H₂S concentration due to high toxicity and stringent safety requirement during handling. Moreover, the simultaneous existence of these impurities also leads to competitive sorption that consequently minimizes membrane separation performance [74]. Recently, polymers with an etheric oxide (EO) unit, such as commercial PEBAX[®], appear as promising membranes that offer high H₂S/CH₄ selectivity ascribed to their high specific interaction with polar molecules such as H₂S [75]. Previous studies demonstrated that PEBAX[®] exhibit H₂S/CH₄ selectivity up to 80 [76][77]. Moreover, some membrane materials also have inherent copermeation properties of CO₂ and H₂S with relatively high selectivity over CH₄. Cellulose acetate (CA) membrane was the first commercialized polymeric membrane since the 1980s and is presently used in the industry for acid gas removal [74]. Achoundoung et al. [78] reported the equivalent CO₂/CH₄ and H₂S/CH₄ selectivity of CA membranes up to 30 under feed pressure of 3447 kPa. However, CA membranes suffer loss of selectivity under aggressive feed conditions.

In 2017, Schlumberger in collaboration with PETRONAS successfully installed and commissioned the CYANARA PN-1 acid gas removal membrane system at onshore gas processing facilities in Terengganu Gas Terminal (TGAST), Malaysia [79][80]. The dual-zoned cellulose acetate-based hollow fibre membrane system efficiently processed 228.89 m³/s and produced a gas stream containing 12 mol% to 25 mol% of CO₂ and H₂S to meet product specifications of <8 mol% CO₂. Compared to the conventional absorption process, the CYNARA PN-1 membrane offers economic advantages through 60% reduction in footprint and 50% installation cost savings. In addition, the installation of the membrane system also is anticipated to reduce operational expenditure (OPEX) by USD 180 million for 20 years of estimated operating cost [79]. Evonik recently launched a high-performance polyimide-based hollow fibre membrane (SEPURAN NG[®]) in 2018 for selective separation of CO₂ and H₂S from natural gas. However, the application of SEPURAN NG[®] is yet to be reported elsewhere.

The application of membrane separation is usually limited to moderate gas flow rate, as performance may deteriorate at high volumes of gas processing. Multistage membrane separation may be necessary to achieve high product purity, which creates additional expenditure. Plasticization and compaction of membranes over a long operation period are the major drawbacks which may impede the excellent progress of membrane gas separation technology. The presence of impurities such as CO₂, H₂S and other heavy hydrocarbons may degrade the integrity

of the membrane and thereby result in poor separation performance. Research methods are underway to cater these limitations through the crosslinking of polymer chains, modification of polymer properties and thermal treatment. Mixed matrix membrane (MMM) paved a new avenue in membrane development to improve their separation performance by synergistically combining the processability of polymeric membrane and high selectivity of inorganic filler.

On the other hand, the growing interest in the development of hybrid processes, which combine membrane and other conventional separation technologies, creates opportunity in gas separation. Bhide et al. [81] developed a process design study and economic assessment of hybrid processes by combining membrane separation and amine absorption, which aimed to purify natural gas that contains up to 40 mol% CO₂ and 1 mol% H₂S. The simulation showed that the hybrid process yielded lower operating costs, costing 1.516 MM USD/year, compared to the independent amine process, costing 2.853 MM USD/year. Furthermore, the total capital investment (TCI) of the hybrid process was estimated to be USD 4.196 MM, which was significantly cheaper than the amine process, which had a TCI of USD 6.226 MM. The cost benefits are due to the fact that the membrane removed approximately 78% of CO₂ from the feed stream and thereby minimized the solvent circulation rate as well as the design capacity required for absorption and the solvent regeneration process. Rezakazemi et al. [82] suggested that the hybrid process is feasible for industrial gas separation with feed flow rate more than 8.17 m³/s and CO₂ content higher than 12%. Moving forward, the progress of membrane technology in natural gas sweetening will focus on the development of high-performance membranes with an active layer in the order of 0.1 μm to enhance their competitiveness as a sustainable separation process.

4. Cryogenic Distillation

Numerous distillation applications were applied in the 19th and 20th centuries, particularly for the alcohol separation process [83]. In 1945, F. Taylor invented the first concept of distillation using laboratory apparatus consisting of three main pieces of equipment: a vessel, a condensing apparatus and a receiver [84]. Then, John M. Chambers (1953) established a more structured distillation column concept to purify the fermented alcohol and remove impurities from the feed stream to obtain high-purity ethanol considering the optimum reflux ratio and heat input in a more stable operation [85]. The demand for distillation technology is increasing rapidly, and the concept is growing and evolving very fast with increased opening of oil refineries and petrochemical plants around the world. Nowadays, most refineries or petrochemical plants have distillation columns in the process, and the application is not just at atmospheric pressure. Still, distillation has been expanded and upgraded for more challenging environmental conditions such as high pressure, low-temperature and high-temperature process, cryogenic temperature, vacuum condition, etc.

The distillation column is designed to separate two or more components from the feed gas stream. The concept of separation is based on differences in boiling point or relative volatility of the component. The low boiling point component is left as a top product, while the higher boiling point component is at the bottom of the column as a liquid product. The operating temperature and pressure are critical parameters in achieving the separation performance [86][87]. Typically, the distillation column operates at a two-phase region where the vapor and liquid

contact plays an important role in mass transfer. The liquid will flow from top to bottom of the column, while the vapor will flow counter-currently to the top, and the vapor–liquid interaction is where the mass transfer for the separation occurs [88].

The internal design of the distillation column, including the selection of trays or packing, is key for mass transfers to meet the performance. The selection of the internals will depend on the purpose of the separation, physical properties, service or system, operating conditions, etc. Typically, the designer will conduct the process simulation to evaluate the feasibility of the design before proceeding with the detailed engineering design of the column [87][88][89][90][91][92][93][94]. The design and operation of the distillation column are challenging and require good process control to have stable operation, since it involves changes in phase inside the distillation column due to different temperature profiles in each theoretical stage. Furthermore, maintaining the operating pressure and controlling the cooling and heating rate at the condenser and reboiler are important to achieve the separation target [93][95]. Cryogenic separation is one of the most efficient methods to separate high CO₂ concentration together with H₂S. Cryogenic separation is widely used commercially to separate CO₂ content to meet the pipeline specification [96]. Despite the bright potential of the technology, the operation of the distillation column is highly energy-intensive, and it consumes almost 40% of energy in the chemical industry to meet the separation performance of the process [97]. Therefore, a lot of optimization and improvement of the distillation column is currently ongoing by researchers to minimize the project's overall cost.

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