Carbon dioxide removal from biogas through pressure swing adsorption – A review

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ARTICLE INFO

ABSTRACT

Biogas is considered a future energy, alternative to natural gas due to its high methane content. Different substrate feedstock determines the composition of the produced raw biogases. Upgrading or purifying the raw biogas by removing carbon dioxide and thereby increasing its methane content to produce high purity biomethane is mandatory before delivering for natural gas substitute. At present, the pressure swing adsorption (PSA) plays a vital role in biogas upgrading along with water scrubbing, chemical absorption, and membrane permeation. In PSA, the core system lies in the adsorbent choice, which determine the effectiveness of separation by measuring the adsorbent's adsorptive capacity and selectivity. In fact, the PSA system itself is bound for many configurations that require optimization for better tuning the system. As such, it is imperative to comprehend the parameters affecting the PSA performance, thus optimizing them to achieve the objective functions. The optimization through the design of experiment techniques provides realistic statistical information and is among the simplest PSA optimization strategies. Owing to the attractiveness of PSA in many gas separation applications from historical and practical evidences, it is expected that this technology will become more competitive for biogas upgrading

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1. Introduction

The present global energy crisis has shifted the focus towards the production of renewable energy (RE) from various possible indigenous resources in order to address the worldwide energy demand. Because fossil fuels are non-renewable and are used at a tremendous rate, an alternative energy source that is renewable, abundant, and cost-effective become necessary. Renewables are anticipated to account for a higher percentage of global energy supply by 2050, despite the fact that the current pace of advancement is substantially delay because of the economic implications of the technology (Ahmed et al., 2021).

Various renewable and sustainable energy sources have been introduced to mitigate the environmental issues and minimize the dependence on fossil fuels, such as hydropower, solar, biomass, biogas etc. Among several sources of renewable energy mentioned, researchers have been given a penalty of attention to biogas production as a source of fuel. As such, significant efforts have been deployed to investigate the biogas due to significant achievements in the renewable energy sector as an effective source of energy. Due to its abundant and diverse input sources and applications make biogas as a valuable and sustainable energy vector (Rafiee et al., 2021).

Biogas is a main product of bacterial conversion of organic material present in wastes like municipal, agricultural, and industrial sources at moderate-to-high temperatures without the presence of free oxygen. Given the availability and diversity of biogas sources, it has the potential to serve as a substitute for fossil-based energy sources and help reduce the dependency on fossil-based sources. As of the year 2022, it is anticipated that the biogas energy output would increase more than double, from 14.5 GW in 2012 (Abd et al., 2021; Sun et al., 2015). Though the latest market in 2019

seems to be lesser than the estimated, with the reported biogas energy output of only 19.5 GW (Abanades et al., 2022), with the introduction of new technologies and innovation, the world production of biogas is expected to keep rising, demonstrating the worth of biogas in the future; despite some delay worldwide particularly in the next few years from 2022 to 2027 (Research and Markets, 2022).

Upon the formation of biogas, some impurities are formed as by-products of the reactions. Some of the impurities in biogas have the potential to cause significant adverse effects on biogas utilization as fuel sources. In order to realize biogas as a natural gas alternative, it is important to condition and upgrade raw biogas to a higher specification. Recently, upgrading biogas to biomethane is one of the vigorous topics in the scientific literature that attracts great interest in the bioenergy industry due to national and international renewable energy objectives, environmental concerns, and the need to transition away from fossil fuels and thus, toward sustainable energy alternatives.

Table 1 summarizes the recent review articles related to biogas upgrading. This review intends to improve and add to previous reviews, by centering the focus on pressure swing adsorption as one of the chosen technology for removing CO_2 from the biogas stream. Despite the existing reviews covering much information on the different technologies for biogas upgrading, there is a limited review that focuses on the pressure swing adsorption (PSA) technology, except several reviews, particularly focusing on the PSA for biogas upgrading and its parametric investigations that are acknowledged (Abd et al., 2021; Shah, Ahmad et al., 2021).

The PSA technology is an important gas separation technology that requires further investigations on various applications, particularly for biogas upgrading. Since the biogas is still a growing energy resource, and its plant capacity is usually small, the PSA technology is the most suit technology

Table 1 – Summary of review articles concerning biogas upgrading technologies.

| References	Key findings related to biogas upgrading technologies
Sahota et al. (2018)	• Review of matured and new biogas upgrading technologies and provides a comparative analysis of the available technologies in terms of economic, technology, and environmental sustainability
Abd et al. (2021)	• A brief review of several biogas upgrading technologies • Focusing on pressure swing adsorption for biogas upgrading with a brief review of the PSA design parameters (adsorption pressure, PSA sizing, P/F ratio, and adsorption time)
Shah, Ahmad et al. (2021)	• Review of biogas upgrading technologies (scrubbing, membrane, cryogenic, in situ), and later focusing on swing adsorption technologies • Detail review of the effect of various factors (adsorbent types, bed configuration, composition of biogas, time cycle, and operating conditions) on the efficiency of the biogas purification process
Golmakani et al. (2022)	• A comprehensive review of 4 main biogas upgrading technologies (absorption, adsorption, membrane, and cryogenic) and provides technological comparison
Struk et al. (2020)	• Detail review of physical and chemical technologies (adsorption, absorption, membrane separation, cryogenic separation, and advanced oxidation) for biogas upgrading, as well as biological methods
Sun et al. (2015)	• Comprehensive review on techno-economic performance of various biogas upgrading technologies • Provide guidelines for the upgrading technology selection
Aghel et al. (2022)	• Provides a detail review of both conventional and recent biogas upgrading technologies, including the hybrid between some of them

for upgrading biogas into biomethane, as it is suitable for handling small- to mid-range of product capacity (Ahmed et al., 2021), thus balancing between the cost and profit.

With regard to the attractiveness of the PSA technology for biogas upgrading, in this review, we review the biogas upgrading by removing carbon dioxide, emphasizing the technology of cyclic separation through pressure swing adsorption (PSA). Section 2 describes the biogas as a sustainable energy source by means of its diverse and abundant feedstock of biogas and its future perspective in the global energy share. Section 3 addresses the biogas upgrading technologies, including water scrubbing, chemical absorption, membrane separation, and PSA. Then, in Section 4, the review focuses on adsorption by discussing the adsorbent as the core element in the PSA. Next, further elaboration focusing on PSA for biogas upgrading is discussed in Section 5, including the parameters affecting its performance, the design of the experiment and the optimization strategy for PSA process. In Section 6, mathematical modeling of PSA is presented with some samples of simulation results for model validation purposes, and followed by some challenges and future research in Section 7, and concluding remarks in Section 8.

2. Biogas - A sustainable source of energy

Biogas is expected to be one of the future renewable energy sources that is produced through methane fermentation in an anaerobic digestion, involves complex microbial interactions in four main biological and chemical stages, which are hydrolysis, acidogenesis, acetogenesis, and methanogenesis

(Koniuszewska et al., 2020). The acetate accounts for 70% CH_4 in biogas production, with CO_2 and H_2 accounting for 30%, while CO_2 is produced in large amounts from the acetogenesis and methanogenesis reactions (Andriani et al., 2014). The stoichiometric production of methane in anaerobic digestion can be calculated using the Buswell equation, as shown in Eq. (1) (Deublein and Steinhauser, 2011).

$$C_\alpha H_\beta O_\delta N_\gamma S_\varepsilon + yH_2O \rightarrow xCH_4 + \gamma NH_3 + \varepsilon H_2S + (\alpha - x)CO_2 \qquad (1)$$
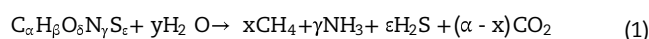
$$x = (4\alpha + \beta - 2\delta - 3\gamma - 2\varepsilon)/8; y = (4\alpha - \beta - 2\delta + 3\gamma + 2\varepsilon)$$

The methane production rate can be estimated using these stoichiometric equations from the decomposition of some type of substrates, particularly the organic materials of chemical composition $C_c H_h O_o N_n S_s$. The average level of CH_4 in biogas is often reported to be around 50–75%, followed by CO_2 around 25–50%, along with trace amounts of impurities including hydrogen sulphide, nitrogen, siloxanes, etc (Atelge et al., 2018). The main difference between biogas and natural gas falls on the CO_2 composition. Biogas composition varies with the nature and type of substrate and the operating conditions at which the anaerobic digestion occurs. Table 2 presents the different type of substrates on the biogas composition and their lower heating value (LHV). The highest methane content, 80% can be obtained in the biogas from agricultural waste and the lowest, 35% from landfill gas. It shows that methane yield can vary between 35% and 80%, depending on the type of substrate.

Biogas upgrading accounts for around 90% of total biomethane production worldwide (Rafiee et al., 2021). Upgrading biogas can improve its heating values to 15 – 30 MJ/

Table 2 – Variability of biogas composition and calorific value from different sources (Baena-Moreno et al., 2020; Chen et al., 2015; Hosseini et al., 2015; Sun et al., 2015).

Parameter	Sources			
	Agricultural waste	Landfills	POME	Natural gas
CH_4 (%)	50 – 80	35 – 65	65 – 70	90 – 95
CO_2 (%)	30 – 50	15 – 50	25 – 30	0.2 – 2
H_2S (%)	0.7	0 – 100 ppm	200 – 1500 ppm	0 – 10 ppm
N_2 (%)	0 – 1	5 – 40	–	0 – 0.5
Siloxanes (%)	Traces	–	–	–
LHV (MJ/Nm^3)	24	16	22	36
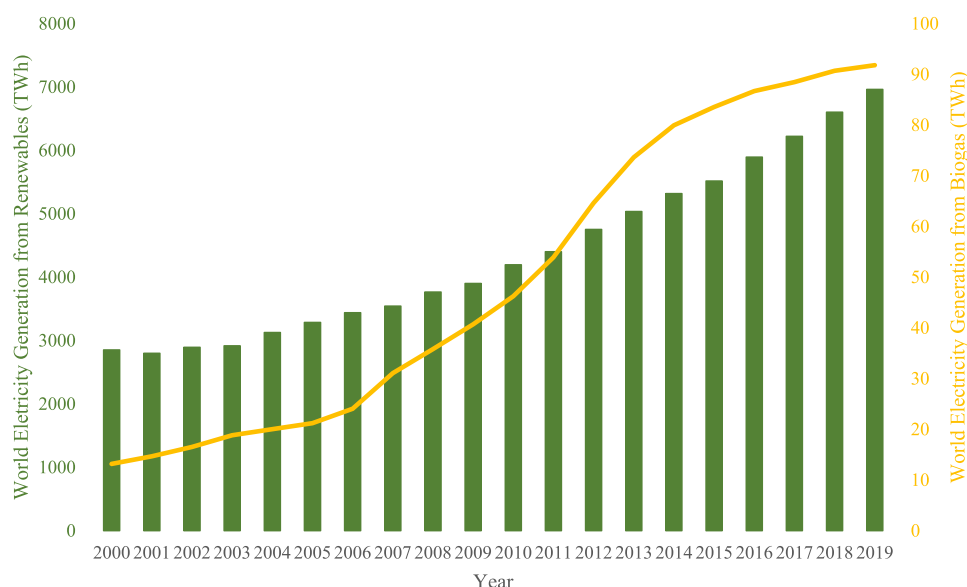
Fig. 1 – (a) World electricity generation from renewable sources (left ordinate) and biogas (right ordinate) between years 2000 – 2019.
Data source taken from IRENA (2021).

Nm^3 (Baena-Moreno et al., 2020). A major benefit of bio-methane over other renewable energy sources (such as hydrogen) is that it can be directly used in the existing natural gas infrastructure and appliances, because of the similarity of both gases (Rafiee et al., 2021). The biogas consumption in 2030 and 2040 is expected to be around 95 and 150 Mtoe. Fig. 1 depicts the world electricity generation from renewable energy sources from 2000 to 2019. The world electricity generation from renewables shows an exponential increase with more than double increment from the year 2000–2019, with 2853 TWh in 2000, to 6963 TWh in 2019 (IRENA, 2021). So, the biogas showed the increment in the world electricity generation with reported value of 13 TWh in 2000 to 92 TWh in 2019. However, the biogas trend shows a slow rise from 2016, most probably due to the fast developments of other renewables as compared to biogas (Rafiee et al., 2021).

Fig. 2 shows the installed biogas electricity capacity of several countries in 2019. Europe is the global player according to the number of installed biogas plant capacity and thus biogas production, followed by North America and Asia regions (IRENA, 2021). In 2019, 62.3 billion m^3 of biogas was produced globally with an equivalent energy content of 1.43 EJ, with an annual growth rate of 9%, between the years 2000 – 2019. Europe itself contributes 30.6 billion m^3 of biogas, accounting for almost half of the global biogas production in 2019 (World Bioenergy Association, 2021).

The future of biogas in the energy vector line is undeniable because of the high scope of supply and demand. To ensure that the utilization of biogas does not affect the security supply, the feedstock or substrate for the biogas from wastes is a feasible solution, be it biogas from palm oil mill effluent (POME), landfills, agricultural wastes, etc. In summary, biogas is a sustainable energy vector with several benefits, including renewable energy sources, lowering greenhouse gases discharge into the atmosphere due to lower dependency on fossil fuels, and generation of by-products of anaerobic digestion digestate for the potential use of fertilizers.

3. Biogas upgrading technologies

Biogas upgrading refers to removing carbon dioxide (CO_2) from the biogas. To fulfill the market specifications of biomethane, CO_2, which is the most significant pollutant in biogas, must be removed in various degrees to meet the methane product quality, which typically needs a methane purity of greater than 95%. Upgraded biogas has a higher calorific value and is known as biomethane as a final gas product, similar to natural gas specifications.

The biogas upgrading technologies are principally adapted from the decarbonization technology in the petroleum and gas refining industry. The biogas upgrading plant adds investment and operating costs to the overall biogas production process. As such, it is essential to choose the most suitable biogas upgrading technology. The upgrading costs of the established technologies depend on the technology itself, and most prominently, on the capacity of the plant (Rafiee et al., 2021). Currently, the most employed technology for this purpose is water scrubbing, PSA, chemical absorption, and membrane permeation. This section will briefly discuss the four main biogas upgrading technologies, including water scrubbing, chemical absorption, membrane permeation, and PSA.

3.1. Water scrubbing

Water scrubbing basically removes carbon dioxide by using water due to the fact that carbon dioxide is more soluble in water than methane. This technique is based on the physical separation (absorption) of gases component using water. Theoretically, at standard temperature and pressure (STP), the solubility of carbon dioxide in water is 26 times higher than that of methane (Muñoz et al., 2015). In addition to carbon dioxide, water scrubbing can simultaneously remove H_2S because CO_2 and H_2S are more soluble in water than CH_4, as shown in Table 3. However, pre-separation of H_2S is typically required prior to CO_2 removal as dissolved H_2S is very
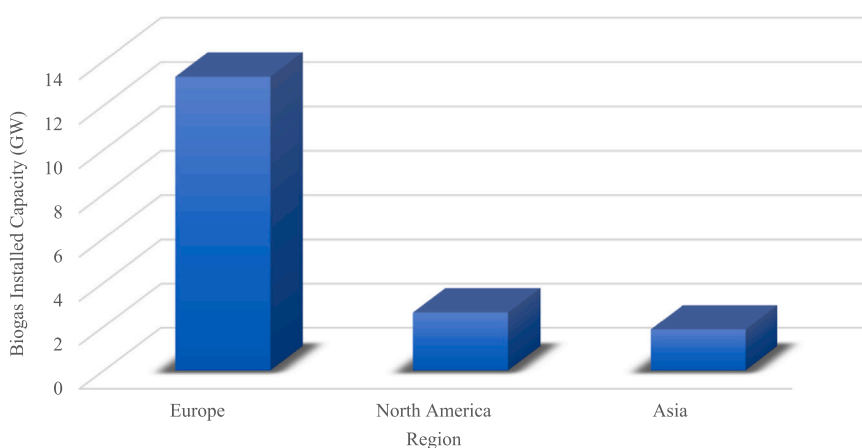
**Fig. 2 – Biogas installed capacity across different region in the world.
Data source taken from IRENA (2021).**

Table 3 – Solubility of several biogas component's in water at STP conditions (Aghel et al., 2022).	
Components	Solubility in water (mmol/kg.bar)
CH_4	1.32
CO_2	34
H_2S	102
Ammonia	28

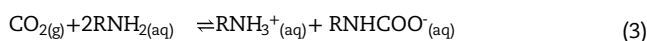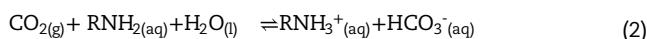corrosive, subsequently, can cause operational problems (Awe et al., 2017).

Depending on the water reusability, two configurations are available for water scrubbing: single-pass scrubbing, which operates without recirculation of the water, and regenerative absorption, which operates a recirculating water system to regenerate and reuse the water for the next cycle (Angelidaki et al., 2018). Nowadays, the latter configuration is widely adapted due to large quantities of water that are required for this technology and a more stable operation (Bauer et al., 2013), as depicted in Fig. 3. Thus, the availability of a low-cost water supply is a determining factor in selecting this technology (Muñoz et al., 2015).

3.2. Chemical absorption

The chemical absorption upgrading process is fundamentally similar to water scrubbing but has a simpler process configuration due to enhanced process performance derived from using CO_2-reactive absorbents (Rafiee et al., 2021). Chemical absorption involves reactions between the absorbed substance (CO_2) and the absorbing component (amine) (Fig. 4). The absorption is called a chemical absorption process since covalent bonds are formed between the chemical (usually amines) and carbon dioxide. The most common amines used in the CO_2 chemical absorption are alkanoamines, including monoethanolamine (MEA) (Abu-Zahra et al., 2007), digylcolamine (DGA) (Al-Juaied and Rochelle, 2006), diethanolamine (DEA) (Xue et al., 2016), triethanolamine (TEA) (Ghiasi and Mohammadi, 2014), methyldiethanolamine (MDEA) (Y. Zhang and Chen, 2011), and piperazine (PZ) (Bougie and Iliuta, 2011).

Chemical absorption is an efficient technology for removing CO_2 from gas mixtures owing to the excellent solubility of CO_2 in chemical solvents. The CO_2 loading capacity of the tertiary amine is higher than those of primary and secondary amines, where the loading capacity lies around 0.5–1.0 mol of CO_2 per mole of amine (Baena-Moreno et al., 2019; Kadam and Panwar, 2017; Yu et al., 2012). The following reactions, as in Eqs. (2) and (3), occur in the CO_2 absorption using alkanoamines (Abdeen et al., 2016).

$$CO_{2(g)} + RNH_{2(aq)} + H_2O_{(l)} \rightleftharpoons RNH_3^+{}_{(aq)} + HCO_3^-{}_{(aq)} \quad (2)$$

$$CO_{2(g)} + 2RNH_{2(aq)} \rightleftharpoons RNH_3^+{}_{(aq)} + RNHCOO^-{}_{(aq)} \quad (3)$$

3.3. Membrane permeation

This technique works on the principle of selective permeation of biogas components through a semi-permeable membrane. The driving force for membrane permeation is the differential permeability of different gas components from the feed side to the permeate side (Baena-Moreno et al., 2020). In biogas upgrading, the CO_2 (the highest permeability rates) passes through the membrane to the permeate side, whereas the CH_4 (the lowest permeability rates) remains on the inlet side as retentate (Noroozi and Bakhtiari, 2019). In order to avoid damaging the membrane fibers, the incoming gas is filtered before entering the membrane to remove water and oil droplets and aerosols. It is much easier to separate CH_4/CO_2 using membrane, as the raw biogas generally contains a high amount of CH_4. The CH_4 remains on the same side of the membrane due to its larger size, which makes it unable/difficult to pass through the selective membrane. At the end, the CH_4 is recovered at high pressure in the retentate side, while the CO_2 is recovered at low pressure in the permeate side, as depicted in Fig. 5. Generally, the membrane fibers have three modules configurations suitable for biogas upgrading, including hollow fiber modules, spiral wound modules, and envelope modules (Brunetti et al., 2010; Scholz et al., 2013).

The modern commercial membrane gas separation is mainly carried out using polymeric membranes. They are typically made from organic materials such as polysulfone, polyimide, polycarbonate, polydimethylsiloxane, and cellulose acetate (Baena-Moreno et al., 2020; Basu et al., 2010), due to their low manufacturing cost, and abundant resources with attractive separation properties (Ullah Khan et al., 2017). For biogas upgrading applications, cellulose acetate is the first to be commercialized and has been in the market for over 30 years (Scholz et al., 2013). In practice, there are two main arrangements of membrane modules available in the
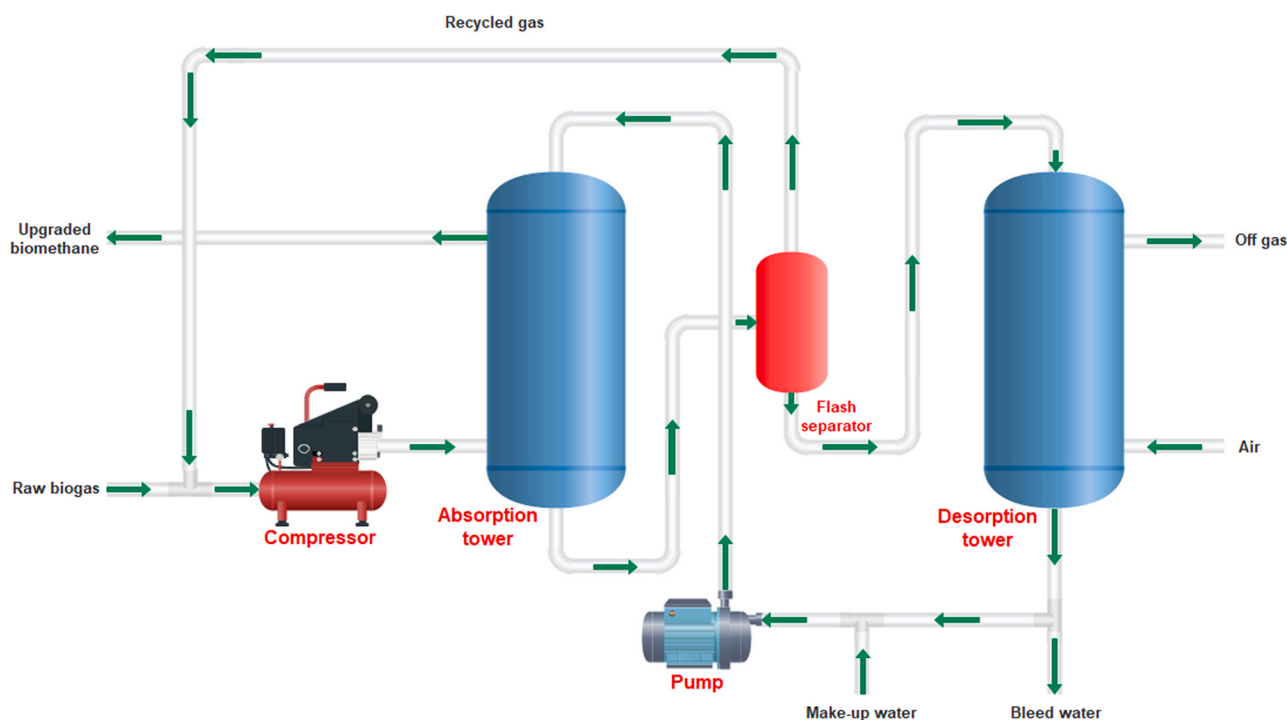
Fig. 3 – Schematic flowsheet of circulating water scrubbing for biogas upgrading.

market: single-stage membrane modules and multistage membrane modules (Baena-Moreno et al., 2020). The latter configuration, especially with recycle, offers better separation efficiency with higher CH_4 purity and lower CH_4 loss (Deng and Hägg, 2010).

3.4.   Pressure swing adsorption (PSA)

The working principle of PSA for biogas upgrading is a dry method based on the selective adsorption of CO_2 over CH_4 onto porous solid adsorbents materials (Rafiee et al., 2021), packed in column as illustrated in Fig. 6. Prior to CO_2 removal using PSA, pre-treatment aims for H_2S removal is required as the adsorbents in PSA can be irreversibly damaged by the H_2S. Pressure swing adsorption (PSA) is one of the cyclic adsorption separation process that is widely adapted for gas separation applications (Marcinek et al., 2021; N. Zhang et al., 2021).

In principles, PSA process consists of two major steps: (1) the adsorption step, at when the solid adsorbent adsorbs the strongly adsorbable component until it reaches saturation, and (2) the desorption step, during which the previously adsorbed component is released from the adsorbent and the adsorbent is regenerated and prepared for the next repeating cycle (Kamin et al., 2022). Fig. 7 depicts the schematic diagram of a conventional Skarstrom 2-bed, 4-step PSA unit design. The four steps cycle involves are (1) Pressurization (2) Adsorption (3) Countercurrent blowdown, and (4) Countercurrent purge. The two connected beds follow the sequence of steps in such a way that there is a continuous flow of product gas. In order to operate such unit cyclically, the columns experience a series of "step" events like opening and closing of valves and changing of flow direction. The red parts in Fig. 7 indicate the PSA parts involved or in the "on" mode during the steps in the cycle.

The core of the PSA system lies in the choice of good adsorbent material for the selectivity to be higher to CO_2 as it is
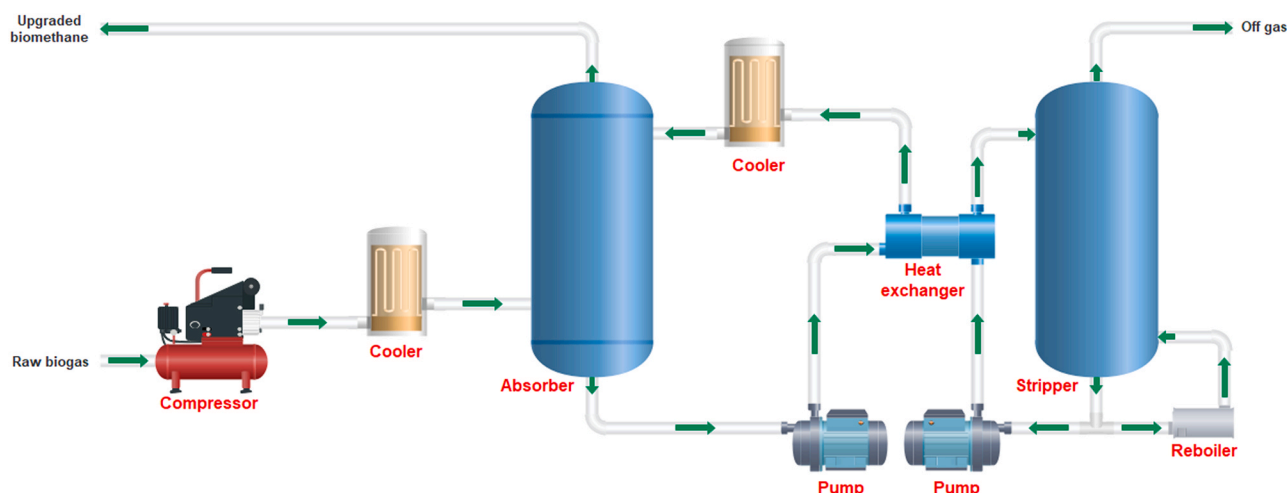

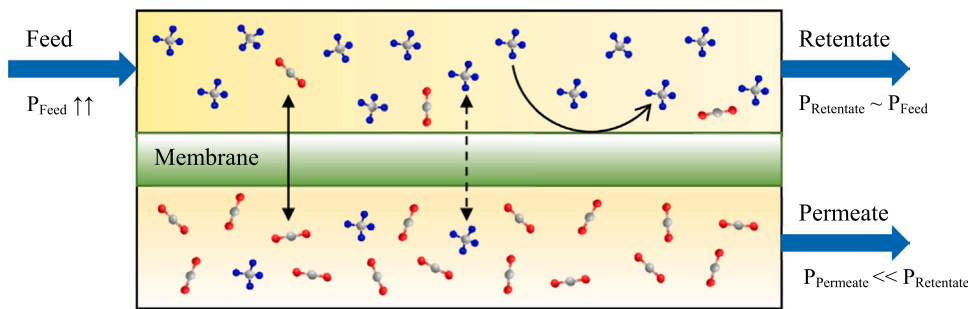Fig. 4 – Schematic diagram of chemical scrubbing process for biogas upgrading.

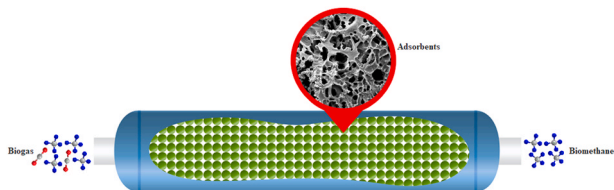**Fig. 5 – An illustration diagram of biogas upgrading by membrane separation.**

Fig. 6 – An illustration of a bed column packed with solid adsorbents.
Adapted from ref. Jawing et al. (2021)

the most crucial determinant of PSA efficiency. The PSA separates the CO_2 from a gas mixture varies depending on their molecular characteristics and affinity for adsorbent. They may either be equilibrium-type adsorbents, which adsorb more CO_2 than CH_4, or they can be kinetic-type adsorbents, which adsorb CO_2 faster than CH_4 due to controlled diffusional rates. Fig. 8 illustrates the difference between equilibrium-controlled and kinetic-controlled adsorbents for CH_4/CO_2 separation using PSA. As illustrated, the equilibrium-controlled adsorbent shows a larger working capacity based on the adsorption isotherm plot. In contrast, the kinetic-controlled adsorbent has a smaller working capacity, thus, it relies on the diffusional rate between the smaller (CO_2) and larger (CH_4) molecules across the adsorbent to achieve the separation.

Table 4 summarizes the comparison of each previously discussed biogas upgrading technologies, in terms of their working principles, advantages and disadvantages. Detailed comparisons can be found in many recent review publications, providing an in-depth comparison (Abd et al., 2021; Adnan et al., 2019; Ahmed et al., 2021; Angelidaki et al., 2018).

As of the year 2015, the water scrubbing remains the dominant CO_2 capture or removal for biogas upgrading method in the biogas global player region that is Europe, most probably due to easy scale up and low cost. While, the chemical absorption and PSA contribute to the second- and third-highest market share for upgrading technologies (Angelidaki et al., 2018; Rafiee et al., 2021). Regardless of which carbon capture or removal method is used, the collected CO_2 must be purified, compressed, and transported for subsequent treatments. However, considering most of the biogas plant is relatively small, it is important to select the biogas upgrading technology that is compact, non-complex and cost-effective when operating at a small to medium range of production capacity. As such, the PSA technology seems to be an appealing option for biogas upgrading application. Hence, in the rest of this review, the focus will be given solely on using PSA as a CO_2 removal technology for biogas upgrading to produce high purity biomethane.

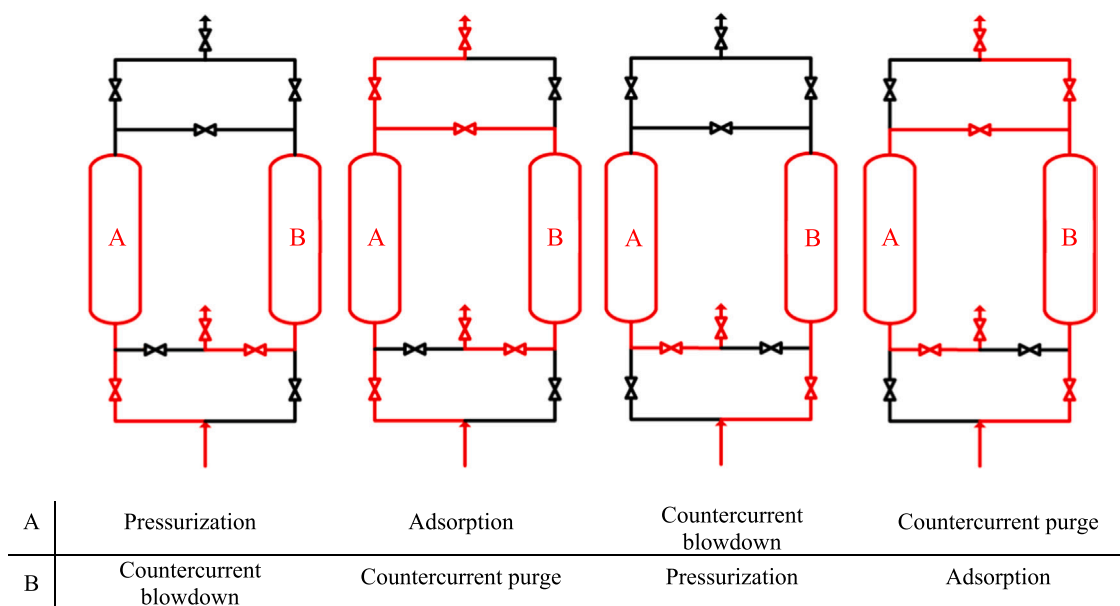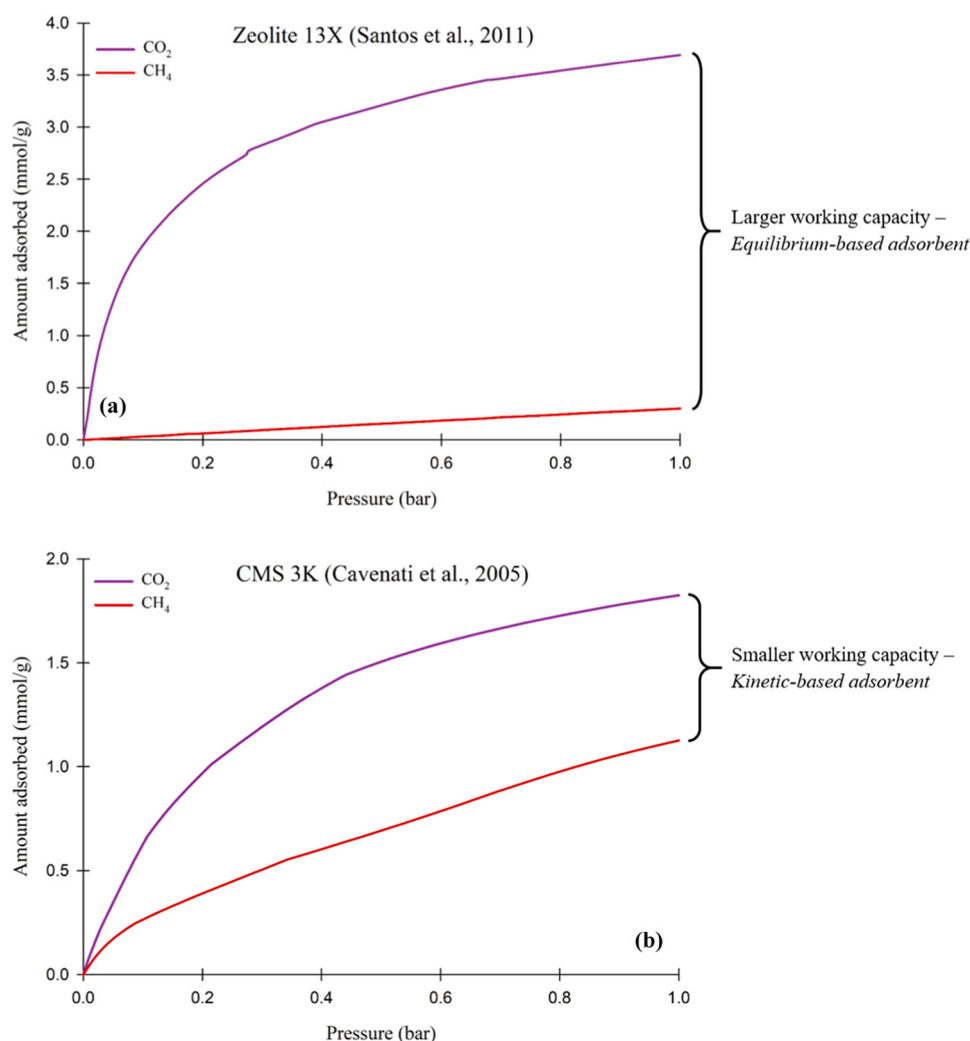| A	Pressurization	Adsorption	Countercurrent blowdown	Countercurrent purge
B	Countercurrent blowdown	Countercurrent purge	Pressurization	Adsorption

Fig. 7 – Classical PSA configuration of 2-bed,4-step Skarstrom cycle.

Fig. 8 – The difference in PSA working capacity of the (a) equilibrium- and (b) kinetic-controlled adsorbents. Adapted from ref. Santos et al. (2011) and Cavenati et al. (2005)

4. Solid adsorbent materials for biogas upgrading

The adsorbent choice is the first and fundamental consideration for achieving high separation efficiency in developing the adsorption system. Different classes of adsorbent materials have been identified over the years, particularly on the CH_4/CO_2 adsorption. Generally, two distinct classes of adsorbents being classified: physical and chemical adsorbents. Between these two classes, physical adsorbents are more attractive due to low energy demand, which in turn requires a lower energy penalty for regeneration. Due to that, most commercially available adsorbents for CO_2 separation belong to the physical adsorbents family, such as activated carbons (AC), carbon molecular sieves (CMS), zeolites, metal-organic frameworks (MOF), etc. The properties of the adsorbents influence the efficiency of these methods, and improvement of adsorbents can improve the technique itself.

Carbon molecular sieves (CMS) have played an important part in the successful development and commercial use of gas separation using PSA technology. CMS are microporous carbon materials, attributed by the uniform slit-shaped pore openings, similar in size to small molecules, usually between 3 and 7 Å (Rodríguez-Reinoso and Sepúlveda-Escribano,

2001). In particular, it is essential that the micropores of the CMS be carefully regulated between 3.2 and 3.7 Å (Shah, Ahmad et al., 2021), in order to provide the sieving effect to CH_4 and CO_2, in which the larger CH_4 (3.8 Å) molecules become slow in comparison with the smaller CO_2 (3.3 Å) molecules, which assure a kinetically-controlled separation.

It is depicted in Fig. 9 that CMS has a sieving effect towards CH_4 and CO_2, as well as the generalized fractional uptake of CH_4 and CO_2 by CMS, which confirms the presence of "bottleneck" in the pore structure of CMS, through which the smaller CO_2 diffuses more quickly than the slightly larger CH_4. Rocha et al. (2017) reported the highest kinetic selectivity by CMS, with kinetic CO_2/CH_4 selectivity of 100 at the defined conditions, as reported in Table 5.

Activated carbons (ACs) and CMS differ mainly in the pore size distribution (Lemcoff, 1999). The ACs have disordered and distributed broader range of pore sizes, ranging from 0.4 to 4 nm (Gu and Yushin, 2014), making them unsuitable for kinetic separation of gas mixtures. The ACs have been widely reported as one of the efficient adsorbents in removing CO_2 because of their excellent specific surface area for high adsorption capacity, particularly at higher pressures. These materials, however, have lower capacities for CO_2 compared with zeolites at lower pressures due to relatively uniform

Table 4 – Summary of different biogas upgrading technologies for removing CO_2.

Property	Water scrubbing	Chemical absorption	Membrane permeation	Pressure swing adsorption
Working principles	The difference in the component solubility	The difference in the component reactive solubility	The difference in the component permeation rate	The difference in the component adsorption or/and diffusion rate
Advantages	– No chemicals is required – Easy operation – Cheap and green operation (water is cheaper and green)	– Chemical solubility higher than water – Small absorption tower than water scrubbing – Low CH_4 loss	– No chemicals or solvent is required – Non-complex process operation – Low CH_4 loss	– No chemicals or solvent is required – Easy scale-up by adding extra unit columns – Can treat small capacities (<500 Nm^3/h)[a]
Disadvantages	– Large volume of water is needed	– Chemicals or solvent is needed – Corrosion issues due chemicals usage – Low CH_4 loss	– Prone to fouling of membrane module over time – Cannot obtain two product gases simultaneously, both at high purities	– Requires high cycle control – Conventional PSA cannot produce simultaneous CH_4 and CO_2 at high purity and recovery

[a] Reference from Syakdani et al. (2019)

electric potential on the surfaces of activated carbons leading to a lower enthalpy of adsorption for CO_2. Since raw biogas is generally obtained at low pressure, the surface chemistry modification of ACs often studied as a strategy for the preparation of carbon capture adsorbents, for the efficiency to be higher at low pressures (Peredo-Mancilla et al., 2019). Wang et al. (2016) demonstrated that at low pressure, the selectivity is highly influenced by the surface chemistry, in particular, the presence of basic functionalities is reported to increase CO_2 adsorption capacity at low pressures (Peredo-Mancilla et al., 2019).

Zeolites are inorganic crystalline materials with diverse structures that exist naturally and can be synthesized in the laboratory. Zeolites possess regular pore sizes in the range of 0.3 – 1.5 nm, consisting of a chain of channels and cavities to capture the gas molecules (Cundy and Cox, 2003). They separate the gas mixtures by means of dipole-quadrapole interactions between the gas molecules and the alkali metal cations in the zeolite frameworks. Depending on the alkali metal cations and the Si/Al ratio, several zeolites are commonly and widely used for CO_2 adsorption, such as zeolite 13X, NaX, LiX, NaUSY, CaX, CaA, etc (Arya et al., 2015; Jiang et al., 2018; Y. Li et al., 2013; Santos et al., 2011). The most popular zeolite used in PSA for various gas separation applications is the zeolite 13X (Y. F. Chen et al., 2021; Shokroo et al., 2016; Xiao et al., 2008). The efficiency of zeolite adsorbents depends on several parameters, including the shape and size of the framework, Si/Al ratio, cationic presence, and molecular polarity (Matito-Martos et al., 2014). Of all the listed zeolites in Table 5, zeolite NaUSY appears to be one of the most efficient adsorbents for CH_4/CO_2 separation, thus a fit candidate for biogas upgrading application, based on its equilibrium selectivity at the defined conditions i.e. temperature and pressure.

5. Pressure swing adsorption for biogas upgrading

Many upgrading technologies presented in the literature revolve around analyzing the biomethane purifying and conditioning considering the benefits of utilizing biomethane as fuel for heat and transportation. The focus of this current review will be centered on the usage of PSA in upgrading biogas to produce high purity biomethane (> 90%).

5.1. Carbon dioxide removal from biogas

Carbon dioxide, CO_2 being the major impurity of biogas, must be removed to the satisfaction of methane level in the biogas, usually greater than 95%. PSA is a mature technology, has become a vital technology for CO_2 separation. Many recent publications have attempted to investigate various aspects of the PSA process for removing carbon dioxide from the biogas stream. Table 6 summarizes the carbon dioxide removal from biogas using PSA studies by various recent publications.

It is observed from Table 6 that the most dominating type of adsorbents for CO_2 removal in biogas upgrading are from the family of CMS and zeolites, mainly because of their highly porous structure, high surface area, with attractive separation efficiency. The commercial PSA technology employs adsorbents from these families. While some recent publications have attempted to produce in-house adsorbents for this purpose, as reported by Ammar Ali Abd, Othman, Shabbani et al. (2022), Noelia Álvarez-Gutiérrez et al. (2016);
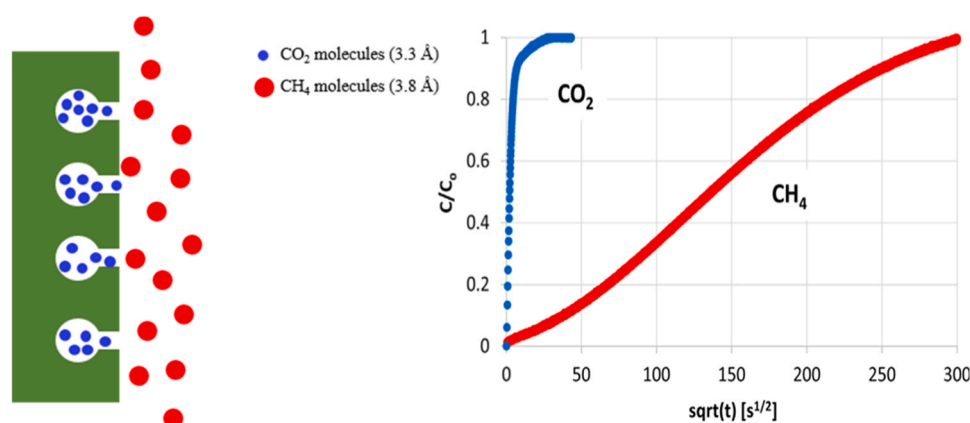
Fig. 9 – (a) Schematic diagram of kinetic sieving effect of CMS towards CH$_4$ and CO$_2$ (b) Fractional uptake of CH$_4$ and CO$_2$ by CMS due to sieving effect.
Taken from ref. Rocha et al. (2017).

N. Álvarez-Gutiérrez et al. (2016), and Durán et al. (2022). In fact, the scope for improvement of the PSA system fundamentally lies in the selection of adsorbent materials. Subsequently, more research for in-house adsorbent synthesis for this application is demanding, especially on green materials originating from waste agriculture, biomasses, etc., thus contributing to a sustainable circular economy principle.

Within the domain of this review, the CH$_4$ purity obtained using PSA technology are in the range of 92–99 + %, having the most frequent CH$_4$ purity in the range between 98 and 99 + %. The lowest CH$_4$ loss was reported by Jiang et al. (2018) using zeolite NaUSY as adsorbent in a 3-bed, 9-step configuration, with CH$_4$ loss of 3.10% (or CH$_4$ recovery of 96.9%). It is worth mentioning that most of the authors in biogas upgrading using PSA technology did not report the PSA performance in terms of productivity and energy consumption. While these indicators are equally important as the product purity and recovery, they should be included for a better image of overall PSA performance at particular configuration, adsorbent and process conditions (pressure ratio (P$_a$/P$_d$), cycle time, purge-to-feed flowrate ratio (P/F) etc.). For example, to obtain a high purity product, the adsorption pressure (or the adsorption/desorption pressure ratio, P$_a$/P$_d$) should be as high as possible, thereby leading to the increasing power requirement (Jain et al., 2003). Though desired high CH$_4$ product purity is achieved, higher power consumption leads to higher compression costs, subsequently making PSA an energy-intensive and expensive technology. Hence, the energy consumption indicator is essential for the overall picture of the PSA performance.

5.2. Parameters affecting PSA performance in biogas upgrading

There are a number of parametric analyses of PSA systems for biogas upgrading that can be found in the scientific literature. This section will address several factors for parametric analysis to establish the influence of different parameters on the process performance with brief explanation on the phenomenon based on the literature. The summary of each parameter affecting discussed in this section is tabulated in Table 7, and the details of it are described in Table 8.

5.2.1. Adsorption pressure
Adsorption pressure is among the widely investigated for parametric analysis of PSA system, because of its direct influence on the capacity of PSA to produce high purity product gas. The basis of the selection of adsorption pressure depends on the equilibrium relationship between adsorbate and adsorbent (Shah, Ahmad et al., 2021). The amount of CO$_2$ adsorbed is directly proportional to the increment of adsorption pressure, thus at higher pressures, the maximum amount of CO$_2$ gets adsorbed. Subsequently, higher purity of CH$_4$ product gas could be obtained at higher adsorption pressure due to high amount of CO$_2$ that gets adsorbed and cause less CO$_2$ in the mixture gases in the outlet. However, the CH$_4$ recovery reduces as the adsorption pressure increases, mostly due to CH$_4$ loss in the feed end during countercurrent depressurization (Moon et al., 2014).

5.2.2. Desorption pressure
Desorption pressure is the opposite of adsorption pressure. Desorption pressure refers to the pressure at which the bed is regenerated for use in the next cycle. It is usually in the blowdown step in a PSA cycle. The selection of desorption pressure also depends on the equilibrium isotherm between the adsorbate and adsorbent. Generally, decreasing desorption pressure will increase the CH$_4$ purity, while reducing its recovery. This is because the adsorption column will hold less CH$_4$ at lower desorption pressure (Xu et al., 2018). In terms of recovery, reduced CH$_4$ recovery is obtained at lower desorption pressure.

5.2.3. Purge to feed flowrate (P/F) ratio
Purging is an important step in PSA cycle as it functions to regenerate the solid adsorbent materials in the bed column, which is applied simultaneously to the blowdown, usually in a counter-current manner, during which a specified volume of product gas (CH$_4$ in biogas upgrading domain) is reversed into the adsorption bed column. The degree of bed regeneration depends on the amount of the purge gas, which is directly related to the purity of the product (Li et al., 2019). Higher P/F ratio translates to a higher purge gas amount at constant feed flowrate, which means better bed regeneration, resulting in higher CH$_4$ product purity. However, increasing the P/F ratio will cause a reduction in CH$_4$ recovery, as more CH$_4$ product gas was used to purge the adsorbent

Table 5 – Literature survey on various adsorbents used for biogas upgrading using adsorption process.

Adsorbent	Temperature, T (K)	Pressure, P (bar)	Adsorptive capacity, q (mmol/g)	Uptake rate (s^{-1})	Equilibrium/Kinetic selectivity	Ref.
CMS 3 K	308	1.00	CO_2 = 1.49; CH_4 = 0.80	–	1.86/-	Cavenati et al. (2005)
CMS KP 407	298	5.00	CO_2 = 3.04; CH_4 = 1.75	$CO_2 = 1.00 \times 10^{-2}$, $CH_4 = 1.00 \times 10^{-4}$	1.74/100	Rocha et al. (2017)
Pine sawdust AC	303	1.20	CO_2 = 2.00; CH_4 = 0.37	–	5.41/-	Durán et al. (2022)
CS-CO_2	303	1.00	CO_2 = 2.32; CH_4 = 1.12	–	2.07/-	Noelia Álvarez-Gutiérrez et al. (2016); N. Álvarez-Gutiérrez et al. (2016)
CS-H_2O			CO_2 = 2.30; CH_4 = 1.02		2.25/-	
Spent coffee	303	1.00	CO_2 = 1.86; CH_4 = 0.98	–	1.90/-	Abd, Othman, Shabbani et al. (2022)
Zeolite 13X	303	1.00	CO_2 = 3.69; CH_4 = 0.30	–	12.3/-	Santos et al. (2011)
Zeolite NaUSY	303	1.00	CO_2 = 4.02; CH_4 = 0.29	–	13.86/-	Arya et al. (2015)
Zeolite 5 A	303	1.00	CO_2 = 3.33; CH_4 = 0.34	–	9.79/-	Ahn et al. (2012)
Zeolite APGI	313	1.10	CO_2 = 3.72; CH_4 = 0.52	$CO_2 = 1.35 \times 10^{-2}$, $CH_4 = 5.09 \times 10^{-2}$	7.15/0.27	Jiang et al. (2018)
Zeolite NaUSY			CO_2 = 3.56; CH_4 = 0.25	$CO_2 = 1.28 \times 10^{-2}$, $CH_4 = 3.90 \times 10^{-2}$	14.24/0.33	
Zeolite NaX	303	1.00	CO_2 = 4.13; CH_4 = 0.73	–	5.66/-	Li et al. (2013)
Zeolite CaX			CO_2 = 3.37; CH_4 = 0.63		5.35/-	
Silica gel	298	1.00	CO_2 = 1.02; CH_4 = 0.10	–	10.20/-	Shen et al. (2018)

bed and then flowed out with the CO_2 (Li et al., 2019). It is worth mentioning that the rate of CH_4 purity rise is substantially slower than the rate of the decrease in CH_4 recovery (Kim et al., 2015; Santos et al., 2011).

5.2.4.   *Adsorption time*
In practice, the adsorption time should be less than the breakthrough time of CH_4 to avoid the mass transfer zone of the CO_2 approaching the end of the bed, thus causing a decline in the product purity. An optimum adsorption time is a function of the adsorption bed length and operating conditions (Zhang et al., 2021). At longer adsorption time, the CH_4 product purity decreases but CH_4 product recovery increases. This is because a longer adsorption time leads to an increased presence of CO_2 during the adsorption process, which reduces the CH_4 purity but increases the CH_4 recovery. However, there is a certain time limit that increasing adsorption time leads to CH_4 purity to decrease, which is at the adsorption time beyond the breakthrough time of CH_4, and at when the rise of CO_2 mass transfer occurs and approaches toward the end of the column (Abd and Othman, 2022; Yang et al., 2008).

5.2.5.   *Blowdown time*
Blowdown time determines the preliminary regeneration time of the bed column in removing the impurities attached to it. The blowdown step is usually carried out in a counter-current manner with or without the use of inert gas. In principle, longer blowdown time means better bed regeneration. Consequently, it leads to higher CH_4 product purity. When the blowdown time increases, the strongly adsorbable component, which is CO_2 has a greater chance to escape from the adsorbent bed because the reduced pressure with longer time forced the CO_2 to detach from the adsorbent surfaces, leading to greater regeneration of the bed (Canevesi et al., 2019; Shah, Sahota et al., 2021). Thus, the efficiency of the adsorbent bed regeneration affects the amount of adsorbate remaining in the bed at the beginning of the next cycle (Durán et al., 2022).

5.2.6.   *Purge time*
The purging time also determines the degree of bed column regeneration in removing the impurities attached to it. The fundamental difference between blowdown and purge lies in the presence of product for bed regeneration, in which purge requires CH_4 product to displace the CO_2 impurities attached to the adsorbent bed. At a longer purge time, the CH_4 purity increases, but its recovery decreases. A longer purge time indicates that more CH4 product is being used, as the CH_4 product is purging the bed, thereby better regeneration of bed for the next cycle (Shah, Sahota et al., 2021). At the same time, the recovery decreases because more CH_4 product fraction was used to purge the adsorbent bed and then flowed out with the CO_2.

5.3.   *Optimization of CH4/CO_2 separation from biogas using PSA*

Since the understanding of important parameters affecting the PSA performance on the separation of CH_4/CO_2 from biogas was established in the previous section, it is imperative to discuss the parameters optimization for maximum performance of the process. The optimization of the PSA performance is extremely substantial for the design and

Table 6 – Literature summary of carbon dioxide removal from biogas using PSA.

System configuration	Adsorbent	Feed composition	Operating conditions			Performance		Productivity	Energy	Reference
			Temperature, T (°C)	P_a/P_d	Cycle time (s)	CH_4 purity (%)	CH_4 loss (%)			
2-bed, 6-step	Zeolite 13X	67:33% CH_4-CO_2	50.0	13.3	850	99.84	16.48	5.43 mol/kg.h	0.123 kW/mol	Santos et al. (2011)
2-bed, 4-step	Zeolite NaUSY	58:42% CH_4-CO_2	30.0	15.0	336	99.28	14.06	15.58 mol/kg.h	0.123 kW/ mol-CH_4	Arya et al. (2015)
4-bed, 7-step	CMS-FB	54.9:45.1% CH_4-CO_2	25.0	19.0	1200	99.60	32.7	N. A.	N. A.	Kim et al. (2015)
2-bed, 6-step	Zeolite 13X	67:33% CH_4-CO_2	50.0	40.0	N. A.	98.60	22.50	N. A.	N. A.	Wu et al. (2015)
4-bed, 6-step	Zeolite 5 A	60:40% CH_4-CO_2	N. A.	30.0	760	98.90	16.10	N. A.	10.2 kW	Augelletti et al. (2017)
3-bed, 9-step	Zeolite NaUSY	50:50% CH_4-CO_2	40.0	20.0	N. A.	99.40	3.10	N. A.	0.27 GJ/ton-CO_2	Jiang et al. (2018)
4-bed, 9-step	Silica gel	55:45% CH_4-CO_2	N. A.	22.2	N. A.	99.56	43.05	N. A.	18.98 kJ/ mol-CH_4	Shen et al. (2018)
2-bed, 6-step	CMS KP 407	60:40% CH_4-CO_2	30.0	50.0	N. A.	99.36	16.20	3.46 mol/kg.h	N. A.	Canevesi, Andreassen et al. (2019)
3-bed, 9-step						98.40	7.90	2.56 mol/kg.h	N. A.	
2-bed, 6-step	CMS-3 K	55:45% CH_4-CO_2	35.0	13.0	560	92.08	18.37	N. A.	N. A.	Kottititum et al. (2020)
2-bed, 8-step	COSMO zeolite 13X	64:35:0.01% CH_4-CO_2-H_2S	25.0	13.3	850	95.47	8.73	N. A.	0.68 GJ/ton-CH_4	Chen et al. (2021)
3-bed, 6-step	CMS	55:45% CH_4-CO_2	30.0	40.0	N. A.	98.03	16.83	N. A.	N. A.	Shen et al. (2021)
2-bed, 4-step	Silica gel	55:45% CH_4-CO_2	30.0	4.0	228	97.17	12.74	N. A.	N. A.	Abd and Othman (2022)
2-bed, 4-step	Spent coffee	68:32% CH_4-CO_2	27.0	2.0	N. A.	97.00	4.57	N. A.	N. A.	Abd et al. (2022)
1-bed, 4-step	Pine sawdust AC	50:50% CH_4-CO_2	30.0	2.5	1680	96.50	41.50	3.9 mol/kg.h	N. A.	Durán et al. (2022)

CH_4 loss (%) = 100 – CH_4 recovery (%)

Table 7 – Effect of process parameters on the CH_4 purity and recovery in biogas upgrading using PSA.

Process Parameters	Features	Explanation
Adsorption pressure	CH_4 purity increases with increasing adsorption pressure, while its recovery decreases with increasing adsorption pressure.	When the adsorption pressure increases, more CO_2 is attracted to the adsorbent materials, thus maximizing the CH_4 purity. In contrast, lower CH_4 recovery ratio is observed at increased adsorption pressure due to longer retention of CH_4 product in the adsorption column (Wu et al., 2015).
Desorption pressure	CH_4 purity decreases with increasing desorption pressure, while its recovery increases with decreases desorption pressure.	As the desorption pressure increases, the pressure ratio, P_a/P_d decreases. Lower P_a/P_d means lower adsorption working capacity, resulting in less CH_4 and CO_2 product gases are being disposed, thus lower the CH_4 purity. Subsequently, increases the CH_4 recovery at higher desorption pressure.
P/F ratio	CH_4 purity increases with increasing P/F ratio, while its recovery decreases with increasing P/F ratio	When the P/F ratio increases, the efficiency of adsorbent bed regeneration increases, thereby resulting in higher CH4 product purity. Consequently, at higher P/F ratio, more of the CH_4 product gas are being used for regenerating the bed column (Moon et al., 2014). Thus, leads to the decrease in the CH_4 recovery as it flows out with the CO_2 at the waste stream (H. Li et al., 2019).
Adsorption time	CH_4 purity decreases with increasing adsorption time, while its recovery increases with increasing adsorption time	When the adsorption time increases beyond a certain time limit, CO_2 get adsorbed because the CO_2 mass transfer zone rise prematurely and approaches the end of the column, subsequently reducing the CH_4 purity (Ammar Ali Abd and Othman, 2022). On the other hand, CH_4 recovery increases due to longer residence time at increased adsorption time.
Blowdown time	CH_4 purity increases with longer blowdown time, while its recovery decreases with longer blowdown time	At longer blowdown time, the strongly adsorbed component, which is CO_2 in the case of biogas upgrading, is experiencing reduced attraction force with the adsorbent as the pressure is reduced, and also the presence of inert gas forced the CO_2 to detach and escape from the adsorbent bed, leading to greater regeneration of the bed (Canevesi et al., 2019; Shah, Sahota et al., 2021).
Purge time	CH_4 purity increases with longer purge time, while its recovery decreases with longer purge time	As the purge time increases, more CH_4 product is being used for purging the saturated bed column, which implies better regeneration of the adsorbent, hence further increasing the CH_4 product purity. In contrast, more fraction of CH_4 product being used at longer purge time subsequently reduces its recovery ratio.

operation of the PSA process. This section presents the optimization of PSA parameters of CO2 adsorption from biogas, compiled from the available literature.

5.3.1. *Techniques on the design of experiment for optimizing PSA process*

Experimental design plays an important role in the field of science and engineering. Design of experiment, referred to as DOE, is a powerful statistical and mathematical tool for systematic experimentation, by generating optimal experimental conditions (Rodriguez-Granrose et al., 2021), thus efficiently determining the cause and effect relationships. Compared to classical experimental methods, the DOE has several merits, including a quicker method, requires smaller sample volume (less experimental set), and systematic data analysis, which can reveal the statistical information from the experimental results (Morali et al., 2019). When used correctly, DOE can provide the answers to specific questions about the behaviour of a system, using an optimum number of experimental observations.

Three key steps determine the successful implementation of DOE: (i) planning, (ii) execution, and (iii) data analysis (Farooq et al., 2016; Jankovic et al., 2021), as described in Fig. 10. As shown, the design of experimental falls under the planning step, at which the selection of the DOE is crucial for producing meaningful results, especially the interacting relationship among the factors or parameters investigated. Therefore, it is important to have the right experimental design to successfully find the optimal value for the factors or parameters, with respect to the objective function(s) of the optimization.

Basically, four designs are widely adapted in the DOE, including factorial, response surface, mixtures, and Taguchi designs. Literature survey on the DOE techniques for PSA process investigation reveals that most authors in the field have employed the response surface design, by employing either the Box-Behnken Design (BBD) or the Central Composite Design (CCD), as listed in Table 9. As expected, none PSA investigation is made using the mixtures design, as it is only suitable for investigation involving formulations and mixture of ingredients.

5.3.2. *Optimization strategy for PSA process*

Optimizing the decision variables to achieve the objective function(s) is another important topic in pressure swing adsorption as the PSA is bound to various configurations, from the simplest 2-bed, 4-step to up to more than 10-bed PSA process. The major decision variables on the PSA process optimization are adsorption pressure, desorption pressure, P/F ratio, adsorption time, blowdown time, and purge time, as

Table 8 – Detailed summary on the effect of process parameters on the PSA performance (purity and recovery). The green arrow indicates increasing trend, while the red arrow indicates decreasing trend.

Process parameter, X	Parameter varied	System configuration	Adsorbent	Feed mixture	Operating conditions			Performance, Y				Ref.
					Temp., T (°C)	P_a/P_d	Cycle time (s)	CH_4 purity (%)	CH_4 recovery (%)	CO_2 purity (%)	CO_2 recovery (%)	
Adsorption pressure, X_1	$2.0 \le X_1 \le 7.0$	4-bed, 7-step	CMS-FB	54.9:45.1% CH_4-CO_2	25	X_1/0.213 bar	1200	$95.3 \le Y \le 99.6$ –	$78.2 \le Y \le 68.9$ ↓	N. A.	N. A.	Kim et al. (2015)
	$4.0 \le X_1 \le 12.0$	2-bed, 6-step	Zeolite 13X	67:33% CH_4-CO_2	50	X_1/0.3 bar	N. A.	$96.8 \le Y \le 98.6$ –	$88.2 \le Y \le 77.5$ ↓	N. A.	N. A.	Wu et al. (2015)
	$5.0 \le X_1 \le 8.0$	2-bed, 6-step	CMS-3 K	55:45% CH_4-CO_2	35	X_1/0.5 bar	560	$91.5 \le Y \le 92.5$ –	$85.3 \le Y \le 81.2$ ↓	N. A.	N. A.	Kottititum et al. (2020)
	$2.0 \le X_1 \le 4.0$	1-bed, 4-step	Pine sawdust AC	50:50% CH_4-CO_2	30	X_1/1 bar	1680	$86.5 \le Y \le 98.8$ –	$72.0 \le Y \le 42.9$ ↓	$69.1 \le Y \le 56.1$ ↓	$83.6 \le Y \le 97.5$ –	Durán et al. (2022)
Desorption pressure, X_2	$0.1 \le X_2 \le 1.0$	4-bed, 7-step	CMS-FB	54.9:45.1% CH_4-CO_2	25	4.052 bar/X_2	1200	$99.4 \le Y \le 89.8$ ↓	$71.0 \le Y \le 77.9$ –	N. A.	N. A.	Kim et al. (2015)
	$0.1 \le X_2 \le 0.5$	2-bed, 6-step	Zeolite 13X	67:33% CH_4-CO_2	50	8.0 bar/X_2	N. A.	$98.9 \le Y \le 97.5$ ↓	$82.6 \le Y \le 88.3$ –	N. A.	N. A.	Wu et al. (2015)
	$0.1 \le X_2 \le 0.5$	4-bed, 9-step	Silica gel	55:45% CH_4-CO_2	N. A.	4.0 bar/X_2	N. A.	$99.5 \le Y \le 95.9$ ↓	$70.4 \le Y \le 75.8$ –	$73.4 \le Y \le 76.1$ –	$99.5 \le Y \le 95.4$ ↓	Shen et al. (2018)
	$0.4 \le X_2 \le 2.0$	1-bed, 4-step	Pine sawdust AC	50:50% CH_4-CO_2	30	3.0 bar/X_2	1680	$99.3 \le Y \le 90.7$ –	$52.8 \le Y \le 62.3$ –	$61.3 \le Y \le 63.1$ –	$99.2 \le Y \le 85.6$ ↓	Durán et al. (2022)
P/F ratio, X_3	$0.15 \le X_3 \le 0.30$	1-bed, 6-step	Zeolite 13X	67:33% CH_4-CO_2	50	13.3	800	$98.7 \le Y \le 99.7$ –	$81.8 \le Y \le 70.3$ ↓	N. A.	N. A.	Santos et al. (2011)
	$0.0 \le X_3 \le 0.2$	4-bed, 7-step	CMS-FB	54.9:45.1% CH_4-CO_2	25	19.0	1200	$97.3 \le Y \le 99.6$ –	$78.7 \le Y \le 67.3$ ↓	N. A.	N. A.	Kim et al. (2015)
	$0.0 \le X_3 \le 0.2$	4-bed, 9-step	Silica gel	55:45% CH_4-CO_2	N. A.	13.3	N. A.	$95.1 \le Y \le 99.1$ –	$82.6 \le Y \le 63.3$ ↓	$81.6 \le Y \le 68.9$ ↓	$94.5 \le Y \le 99.1$ –	Shen et al. (2018)
	$0.067 \le X_3 \le 1.000$	1-bed, 4-step	Pine sawdust AC	55:45% CH_4-CO_2	30	3.0	1680	$94.3 \le Y \le 97.0$ –	$46.9 \le Y \le 62.9$ –	$63.4 \le Y \le 63.0$ ↓	$95.5 \le Y \le 94.9$ ↓	Durán et al. (2022)
Adsorption time, X_4	$100 \le X_4 \le 200$	4-bed, 9-step	Silica gel	55:45% CH_4-CO_2	N. A.	13.3	N. A.	$99.2 \le Y \le 96.4$ ↓	$57.8 \le Y \le 81.1$ –	$65.6 \le Y \le 80.9$ –	$98.5 \le Y \le 96.6$ ↓	Shen et al. (2018)
	$200 \le X_4 \le 480$	2-bed, 6-step	CMS-3 K	55:45% CH_4-CO_2	35	13.0	$310 + X_4$	$92.0 \le Y \le 61.0$ ↓	N. A.	N. A.	N. A.	Kottititum et al. (2020)
	$110 \le X_4 \le 300$	2-bed, 4-step	Silica gel	55:45% CH_4-CO_2	30	4.0	$118 + X_4$	$97.2 \le Y \le 69.4$ ↓	$87.3 \le Y \le 62.3$ ↓	N. A.	N. A.	Abd and Othman (2022)
Blowdown time, X_5	$420 \le X_5 \le 1260$	1-bed, 4-step	Pine sawdust AC	50:50% CH_4-CO_2	30	3.0	$1260 + X_5$	$96.5 \le Y \le 99.3$ –	$58.5 \le Y \le 53.1$ ↓	$63.1 \le Y \le 61.4$ ↓	$95.0 \le Y \le 99.4$ –	Durán et al. (2022)
Purge time, X_6	$180 \le X_6 \le 220$	1-bed, 6-step	Zeolite 13X	67:33% CH_4-CO_2	50	13.3	$600 + X_6$	$98.0 \le Y \le 99.3$ –	$82.8 \le Y \le 80.7$ ↓	N. A.	N. A.	Santos et al. (2011)

Adsorption and desorption pressure in unit bar; adsorption, blowdown and purge time in unit second.
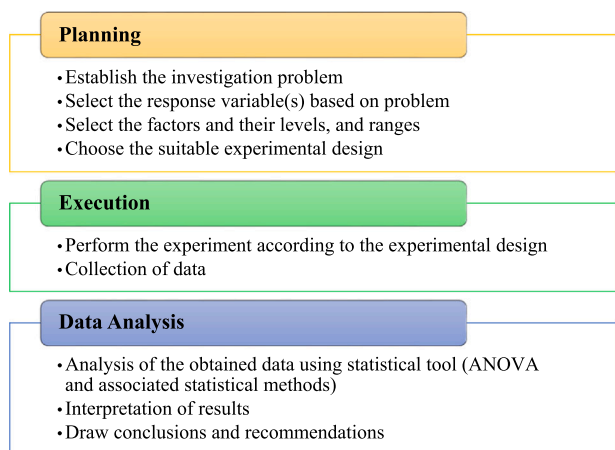
Fig. 10 – Key steps in successful implementation of the design of experiment (DOE).

discussed in Section 5.2, though some studies also included other decision variables as well such as feed flowrate, effect of adsorbents, and sizing of the column (Abd et al., 2021; Shah, Ahmad et al., 2021). Nevertheless, the selection criteria for a suitable combination of these parameters for optimization for a particular problem depends upon various considerations like cost, the existence of a current system, plant capacity, etc.

The research on the optimization of PSA has been carried out by aiming various single objectives of optimization i.e. maximization of product purity (Noelia N. Álvarez-Gutiérrez et al., 2016; Noelia Álvarez-Gutiérrez et al., 2016), recovery (Yáñez et al., 2020), productivity (Zhang et al., 2021), and minimization of power requirement (Xu et al., 2019; Zhang et al., 2008) and cost (Zheng and Lee, 2017), as well as multiple objectives optimization (Haghpanah et al., 2013; Ko et al., 2005; Subraveti et al., 2019). Several techniques of optimization models that are widely used are the response surface methodology (RSM), artificial neural network (ANN), Gaussian process regressions, etc. (Pai et al., 2020), with the first two being the most applied.

The popularity of the statistical model optimization methodologies (RSM and ANN) is due to their nature of reduced-order or surrogate models; a type of data-driven statistical algorithms to represent complex models (S. H. Kim and Boukouvala, 2019). It is a very efficient approach for optimizing systems through regression of surrogate models. Once the surrogate model is trained, analytical representations become available, and the optimization can be done. In the case of biogas upgrading, although the PSA route for

biogas upgrading has been the object of several studies, none have examined the optimization of PSA processes. This creates necessity for applying the abovementioned optimization strategy to the biogas upgrading application.

For example, N. Zhang et al. (2021) demonstrated the PSA optimization strategy for hydrogen purification based on the RSM through the BBD framework. Three decision variables were considered in their work, namely adsorption time (t_{AD}), pressure equalization time (t_{PE}), and P/F ratio, with three objective functions, including hydrogen purity, recovery, and productivity. In one of their case, they optimized the system by aiming to maximize the hydrogen purity, subjected to the constraint of the hydrogen recovery and productivity, and able to obtain the maximum hydrogen purity of 99.99% at t_{AD} of 168 s, t_{PE} of 14 s, and P/F ratio of 0.11, with a prediction error between the obtained optimum decision variables value and the Aspen Adsorption simulation of only 0.03%.

In addition, Tong et al. (2021) have examined the optimization of a PSA system for hydrogen purification using ANN, by considering the adsorption pressure and adsorption step time as the decision variables, which target for multi-optimization of both hydrogen purity and recovery. The values of the objective functions (hydrogen purity and recovery) from the trained dataset of the ANN were confirmed by the Aspen Adsorption simulation, with a regression coefficient (R^2) of 0.99995 for the whole dataset. Therefore, the above examples of optimization strategy using RSM and ANN for hydrogen purification application, though limited, they can represent the capability of the optimization strategy, with excellent prediction from the surrogate model, and should be applied the same for PSA optimization in biogas upgrading studies.

6. Mathematical modeling of pressure swing adsorption process

To achieve a suitable and effective design of adsorption process, there is a need for an appropriate model to describe the dynamics of the adsorption system (Dantas et al., 2011). Mathematical modeling is an essential tool in chemical process design and optimization. So if no experimental facility is available or time is limited, modeling and simulations are commonly applied for investigating the dynamic behavior of the PSA process. It is capable of predicting the dynamics of adsorption systems by coupling the partial differential equations representing the mass, momentum and energy transfer within the control volume (Ben-Mansour et al., 2016). In this section, the mathematical model of the packed bed adsorption column is presented with some sample of simulations results on CH_4/CO_2 separation.

DOE techniques	Author (s)
Table 9 – Authors using various design of experiment (DOE) techniques on PSA process investigation.	
Factorial	Chen et al. (2021), Liu and Ritter (1997), Mulgundmath and Tezel (2010)
Response surface	Saberimoghaddam and Nozari (2017), Zhang et al. (2021), Relvas et al. (2018), Karimi and Fatemi (2021), Shen et al. (2018), Yáñez et al. (2020)
Mixtures	Not found
Taguchi	Sureshkannan et al. (2021)

6.1. Theoretical framework and model assumptions

The prediction of the dynamic performance of a packed bed column involves the simultaneous solution of a set of partial differential equations (PDEs) that represents the material, momentum, and energy balances across a packed bed with proper boundary conditions. Several general assumptions were listed in the following, which serve as the foundation in developing the model.

(i) The gas in the bulk phase behaves following the ideal gas law, $P = CRT$
(ii) The bed properties are assumed to be constant for the bed porosity and bulk solid density
(iii) There are no radial variations in gas concentration, temperature and pressure
(iv) The mass transfer kinetics within the solid phase is described by the lumped linear driving force (LDF) model
(v) Constant mass transfer coefficients along the column bed in axial direction

6.2. Principles of conservation

Three occurring conservation principles are involve in describing the complete adsorption process in a packed bed column, including mass, momentum, energy.

6.2.1. Conservation of mass
The mass balance includes convection, axial dispersion, gas accumulation in the interparticle void space, and adsorption flux to the solid surface. The overall mass balance equation is shown in Eq. (4).

$$-\varepsilon_b D_{ax}\frac{\partial^2 c_i}{\partial z^2}+\frac{\partial(v_g c_i)}{\partial z}+\left[\varepsilon_b+(1-\varepsilon_b)\varepsilon_p\right]\frac{\partial c_i}{\partial t}+(1-\varepsilon_b)\rho_s\frac{\partial q_i}{\partial t}=0 \tag{4}$$

6.2.2. Conservation of momentum
Due to the presence of solid adsorbent particles inside the column, resulting in the reduction of pressure and the gas flows through the packed bed column. Just like flow through a pipe, the frictional force relies on the flow's laminarity and certain gas characteristics. To account for a wider range of flow regime, the Ergun equations is used as it can handle both laminar and turbulent flows. The Ergun equation is shown in Eq. (5).

$$\frac{\partial P}{\partial z}=-\left(\frac{1.50\times10^{-3}\mu(1-\varepsilon_b)^2}{\varepsilon_b^3(2r_p\psi)^2}v_g+1.75\times10^{-5}\frac{(1-\varepsilon_b)M\rho_g}{2r_p\psi\varepsilon_b^3}v_g^2\right) \tag{5}$$

6.2.3. Conservation of energy
Adsorption is an exothermic process, while the desorption is an endothermic process. The release and absorbed of energy leads to temperature variations throughout the process. In the conservation of energy, three control volumes may be accounted for the energy transfer: gas-phase, solid-phase, and column wall.

The energy equation for the gas-phase can be written as shown in Eq. (6).

$$-k_{ga}\varepsilon_i\frac{\partial^2 T_g}{\partial z^2}+C_{vg}\nu_g\rho_g\frac{\partial T_g}{\partial z}+\varepsilon_B C_{vg}\rho_g\frac{\partial T_g}{\partial t}+P\frac{\partial\nu_g}{\partial z}+HTCa_p$$
$$(T_g-T_s)+\frac{4H_w}{D_B}(T_g-T_o)=0 \tag{6}$$

While the energy equation for the solid-phase can be written as in Eq. (7).

$$-k_{sa}\frac{\partial^2 T_s}{\partial z^2}-\rho_s C_{ps}\frac{\partial T_s}{\partial t}+\rho_s\sum_{i=1}^{n}(C_{pai}w_i)\frac{\partial T_s}{\partial t}+\rho_s$$
$$\sum_{i=1}^{n}\left(\Delta H_i\frac{\partial w_i}{\partial t}\right)-HTCa_p(T_g-T_s)=0 \tag{7}$$

And the energy equation for the bed column wall can be expressed as the following Eq. (8).

$$-k_w\frac{\partial^2 T_w}{\partial z^2}+\rho_w c_{pw}\frac{\partial T_w}{\partial t}-H_w\frac{4D_B}{(D_B+W_T)^2-D_B^2}(T_g-T_w)+H_{amb}$$
$$\frac{4(D_B+W_T)^2}{(D_B+W_T)^2-D_B^2}(T_w-T_{amb})=0 \tag{8}$$

6.3. Adsorption isotherms

The adsorption equilibrium isotherms and their corresponding equations characterize the adsorption processes comprehensively and thoroughly. The adsorption isotherm describes and predicts the amount of adsorbed material as a function of pressure (or concentration) at a constant temperature (Mozaffari Majd et al., 2022). Several adsorption isotherms are widely adapted to describe the amount of adsorbed CO_2 and/or CH_4 onto the adsorbent material as a function of pressure, including the Langmuir isotherm (Shen et al., 2018), Sips isotherm (Abdeljaoued et al., 2018; Noelia Álvarez-Gutiérrez et al., 2016; N. Álvarez-Gutiérrez et al., 2016; Durán et al., 2022; Surra et al., 2022), Extended Langmuir 2 (Ammar Ali Abd, Othman, Shabbani et al., 2022), and Dual-Site Langmuir (Jiang et al., 2018; Rocha et al., 2017), as listed in Table 10. It is noticed that the Sips isotherm is among the widely adapted in fitting the equilibrium data of gas components, most probably due to the ability to cover the Langmuir and Freundlich isotherms at low and high pressures, which means that at low pressure, the Sips isotherm reduces to the Freundlich isotherm, but at high pressure, it becomes the Langmuir isotherm.

6.4. Sample of simulation result for CH_4/CO_2 separation

Here in this section, sample of simulation results for CH_4/CO_2 adsorption taken from the literature are presented, particularly on the validation of the mathematical modelling, by comparing the simulation results with the experimental breakthrough curves.

The work of Durán et al. (2022) presents breakthrough curve experiments for CH_4/CO_2 adsorption onto pine sawdust AC. Fig. 11 (a) – (c) show comparison of experimental data and LDF simulation for the breakthrough curves for the adsorption of CO_2 from binary gas mixture of CH_4/CO_2 at various pressures (1.2 – 10 bar), flowrates (15 – 50 cm³/min), and feed binary mixtures (30 – 65% CO_2). These figures show the ratio of the gas components at the column end to the feed mixture (C_t/C_0) versus the normalized time. The normalized time was obtained by dividing the time of the simulation to the total mass of the adsorbent in the column. Roll-up phenomena are observed from those figures, indicating competitive adsorption between CO_2 and CH_4 for the adsorption sites, which is a common behavior in multiple component mixtures adsorption. Overall, the model agrees very well to all the breakthrough curve experiments, at different pressures, flowrates, and feed mixtures, indicating the

Table 10 – Various adsorption isotherms for CO_2 adsorption. The fitting parameters presented here is for CO_2.

Adsorption isotherm	Adsorbent	Equation	Fitting parameters		Ref.
Sips	CS-CO_2	$q = q_s \dfrac{(bP)^{\frac{1}{n}}}{1+(bP)^{\frac{1}{n}}}$	At 303 K, $q_s = 10.88$ mol/kg $b = 0.0017$ kPa $n = 1.38$	$Q = 19.12$ kJ/mol $\alpha = 0.37$	Noelia Álvarez-Gutiérrez et al. (2016); N. Álvarez-Gutiérrez et al. (2016)
Sips	MCW(PA)3 h	$q = q_s \dfrac{(bP)^{\frac{1}{n}}}{1+(bP)^{\frac{1}{n}}}$	At 303.15 K, $q_{s,0} = 8.32$ mol/kg $b_0 = 0.27$ 1/bar $\alpha = 0.10$	$n_0 = 1.39$ $Q = 17.1$ kJ/mol $T_0 = 303.15$ K	Surra et al. (2022)
Langmuir	Silica gel	$q = \dfrac{q_{m,i} b_i P_i}{1+b_i P_i}$	Temperature dependent, $q_{m,i} = 6.006$ mmol/g $b_{0,i} = 8.609 \times 10^{-6}$ 1/bar $-\Delta H_i = +24.967$ kJ/mol		Shen et al. (2018)
Sips	Pine sawdust AC	$q = \dfrac{IP_1 IP_2 P_i^{IP_3} e^{\frac{IP_4}{T_s}}}{1+IP_5 P_i^{IP_3} e^{\frac{IP_6}{T_s}}}$	Temperature dependent, $IP_1 = 6.107 \times 10^{-3}$ mol/g $IP_2 = 6.312 \times 10^{-4}$ 1/bar $IP_3 = 7.315 \times 10^{-1}$	$IP_4 = 2.108 \times 10^3$ K $IP_5 = 8.507 \times 10^{-4}$ 1/bar $IP_6 = 1.982 \times 10^3$ K	Durán et al. (2022)
Extended Langmuir 2	Spent coffee	$q = \dfrac{IP_1 IP_2 e^{\frac{IP_4}{T}} P}{1 + IP_3 e^{\frac{IP_4}{T}} P}$	Temperature dependent, $IP_1 = 1.274 \times 10^{-5}$ mol/g $IP_2 = 1839$ 1/bar	$IP_3 = 0.2486$ $IP_4 = 626.8$ K	Abd, Othman, Shabbani et al. (2022)
Sips	CNS activated carbon	$q = q_s \dfrac{(bP)^{\frac{1}{n}}}{1+(bP)^{\frac{1}{n}}}$	Temperature dependent, $q_s = 14.25$ mol/kg $b = 0.0016$ kPa	$n = 1.30$	Abdeljaoued et al. (2018)
Langmuir	Zeolite NaUSY	$q = \dfrac{q_s bP}{1 + bP}$	Temperature dependent, $q_s = 5.30$ mmol/g	$b = 12.24$ 1/bar	Arya et al. (2015)
Langmuir	Zeolite 5 A	$q = \dfrac{q_m BP}{1 + BP}$	Temperature dependent, $k_1 = 10.03$ mol/kg $k_2 = -0.01858$ mol/k	$k_3 = 1.5781$ 1/bar $k_4 = 207$ K	Ahn et al. (2012)
Dual-Site Langmuir	Zeolite NaUSY	$q = \dfrac{m_1 B_0 P}{1 + B_0 P} + \dfrac{m_2 D_0 P}{1 + D_0 P}$	Temperature dependent, $m_1 = 4.45$ mol/kg $m_2 = 1.15$ mol/kg $b_0 = 1.5 \times 10^{-7}$ 1/kPa	$Q_1 = 31.79$ kJ/mol $d_0 = 4.2 \times 10^{-10}$ 1/kPa $Q_2 = 38.84$ kJ/mol	Jiang et al. (2018)
Sips	Zeolite NaX	$q = \dfrac{q_m b P^C}{1 + bP^C}$	At 303 K $q_m = 5.9774$ $b = 10.4457$	$c = 0.6680$	Li et al. (2013)
Dual-Site Langmuir	CMS KP407	$q = \dfrac{q_{m1} K_1 P}{1 + K_1 P} + \dfrac{q_{m2} K_2 P}{1 + K_2 P}$	Temperature dependent, $q_{m1} = 2.8442$ mol/kg $k_{01} = 1.560 \times 10^{-10}$ 1/Pa $-\Delta H_1 = 25{,}565$ J/mol	$q_{m2} = 1.068$ mol/kg $k_{02} = 8.970 \times 10^{-10}$ 1/Pa $-\Delta H_2 = 27{,}948$ J/mol	Rocha et al. (2017)

robustness and reliability of the model in predicting the dynamic adsorption separation of CH_4/CO_2 at various conditions.

7. Challenges and future research

Based on the current review of the CO_2 removal using pressure swing adsorption (PSA) for biogas upgrading research, several challenges have been identified which may lead to more possibilities for future research in this field.

Even though the biogas production technology is commercially-ready and mature, the biogas usage itself is still limited, especially as a fuel, due to higher purification standards before its usage (CH_4 purity > 95%). This review found that many researchers are still working on achieving ultra-high CH_4 purity, while at the same time increasing the CH_4 product recovery coming out from the PSA unit. The CH_4 recovery is generally low, mostly reported within the range of 30–50%, which means that the exhaust of the PSA units contains an appreciable amount of CH_4. This triggers, not only the economic perspectives but also the environmental concerns as CH_4 is 25 times more greenhouse effect than CO_2. In addition, the cost-competitiveness of the PSA technology remains one of the challenges faced by the researchers and the industrial practitioner. Though PSA is proven to produce ultra-high CH_4 purity, the cost associated with the process is usually high owing to work needed to be supplied to increase-decrease the system's pressure.

Future research and development are needed to address the aforementioned challenges. The primary emphasis of a PSA is on the improved adsorbent capacity, less CH_4 loss and efficient use of energy, while achieving the required CH_4
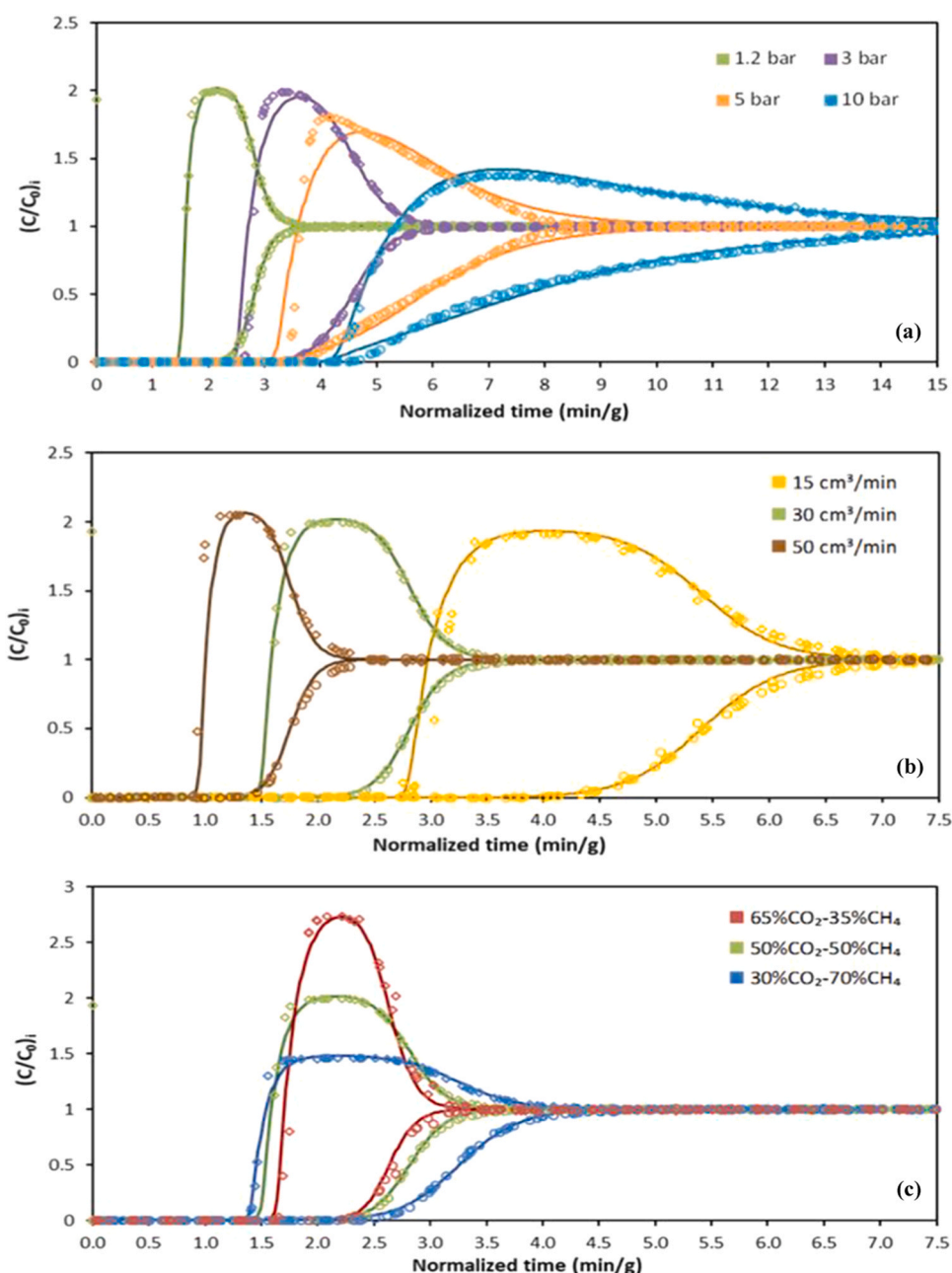
Fig. 11 – Validation of adsorption breakthrough curve for CH_4 (diamond) and CO_2 (circle) on pine sawdust AC (a) at different total pressures, (b) at different flowrates, and (c) at different feed mixtures.
Taken from ref. Durán et al. (2022).

purity. Hence, the research and development are interested in developing novel adsorbents and optimize the PSA cycles/configurations. The current latest research on zeolites, carbon nanotubes (CNTs), metal-organic frameworks (MOFs), and zeolitic imidazolate frameworks (ZIFs) has the potential to enhance the efficiency of the adsorbents. The adsorbents' capacity and selectivity towards a particular component molecule are among the important indicators for adsorbents' performance. Several notable research works on adsorbents screening for PSA application mostly use these two criteria (capacity and selectivity) as index measures in choosing appropriate and viable adsorbents (N. Álvarez-Gutiérrez et al., 2018; Pirngruber et al., 2012). Not only that, the decision on the PSA configuration itself plays an important role in determining the final product purity and recovery, as well as

the energy required. Optimizing the PSA configuration in terms of the number of columns, step cycle, cycle time, P_a/P_d, flowrates, energy required etc., has always been the most researched investigation in the PSA. The ability of systematic DOE and statistical surrogate models eases the optimization of PSA configuration through mathematical modeling and simulation approach.

Likewise, a state-of-the-art dual-reflux PSA (DR-PSA) configuration should be explored for particular purposes, in this case is biogas upgrading. To the best of our knowledge, there is no reported literature on applying the DR-PSA for biogas upgrading purposes. Though DR-PSA has been feasibly proven to simultaneously produce two product components with higher purity and lower loss (for binary mixture), the investigation in biogas upgrading should be

made to contribute to the knowledge and bridging the gap between the theory and practical feasibility (through experiment and/or modeling investigations).

8. Concluding remarks

Biogas production from waste materials and renewables is an ideal solution to address the world's energy and environmental concerns. Biogas is widely recognized to be one of the major energy carriers in this modern era, and it is expected to grow exponentially in the near future year. Upon the formation of biogas, many by-products are formed from the side reactions of anaerobic digestion. Impurities content in the raw biogas such as CO_2 must be removed in order to produce a natural gas substitute (biomethane). Although biogas production technology is well-established globally, commercial applications of biogas are still limited due to the necessity for the biogas purification before on-site use. Water scrubbing, chemical absorption, membrane permeation, and PSA are among the widely adapted technologies in biogas upgrading for removing CO_2 to produce high purity biomethane. Among others, PSA has been widely applied to remove carbon dioxide from the biogas stream. Until today, the PSA still uses CMS- and zeolites-type of adsorbents for this purpose. In-house adsorbent synthesis and testing on the PSA system should be carried forward to progress. In fact, PSA has shown feasibility in producing ultra-high biomethane, exceeding 99% CH_4 purity. Even though PSA is a well-developing technology in both research and commercial domains, there is still scope for improvement, especially on the infinite number of possible cycles or configuration and process conditions of the PSA system itself. As such, it is crucial to understand the effect of important parameters that affect the PSA performance for better optimizing the system.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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