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Keywords: CO<sub>2</sub> capture; anion exchange membranes; CO<sub>2</sub> separation; diffusion; sorption



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*Article*

# Assessment of Anion-Exchange Membranes for CO<sub>2</sub> Capture Processes: A Focus on Fumasep<sup>®</sup> and Sustainion<sup>®</sup>

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**Abstract:** Anion exchange membranes are utilized in cutting-edge energy technologies including electrolyzers and fuel cells. Recently, these membranes have also emerged as a promising tool in CO<sub>2</sub> capture techniques, such as moisture-driven direct air capture and the separation of CO<sub>2</sub> from other gases, leveraging the moisture-induced sorption/desorption and diffusion of CO<sub>2</sub> in its ionic forms. In this study, we examine the absorption and permeation of CO<sub>2</sub> and CH<sub>4</sub> in two commercially available anion exchange membranes, Fumasep<sup>®</sup> and Sustainion<sup>®</sup>, under dry conditions. With the exception of CO<sub>2</sub> sorption in Fumasep<sup>®</sup>, these measurements have not been previously reported. These new data points are crucial for evaluating the fundamental separation capabilities of these materials and for devising innovative CO<sub>2</sub> capture strategies, as well as for the simulation of novel combined processes. In a dry state, both materials demonstrate similar CO<sub>2</sub> absorption levels, with a higher value for Sustainion<sup>®</sup>. However, Fumasep<sup>®</sup> exhibits higher CO<sub>2</sub>/CH<sub>4</sub> ideal solubility-selectivity and higher diffusivity. CO<sub>2</sub> diffusion coefficient increases with CO<sub>2</sub> concentration in both membranes, due to swelling of the matrix, while the solubility coefficient of CO<sub>2</sub> shows the typical behavior of glassy polymers. CO<sub>2</sub> permeability vs. pressure curves exhibit a minimum at a pressure of approximately 2-3 bar. The CO<sub>2</sub> permeability in the dry state is higher in Fumasep<sup>®</sup> than in Sustainion<sup>®</sup>, and for both polymers has similar order of magnitude to the value measured in fluorinated ion exchange membranes such as Nafion<sup>®</sup>.

**Keywords:** CO<sub>2</sub> capture; anion exchange membranes; CO<sub>2</sub> separation; diffusion; sorption,

## 1. Introduction

Escalating CO<sub>2</sub> emissions from a broad range of industrial sectors have disrupted Earth's natural carbon cycle, exacerbated the greenhouse effect and led to climate change. In response to this pressing environmental challenge, CO<sub>2</sub> capture and utilisation strategies have garnered significant interest due to their potential to mitigate anthropogenic greenhouse gas emissions and convert CO<sub>2</sub> into high-value products. Within the spectrum of carbon capture and conversion technologies, ion exchange materials have emerged as crucial components. Such membranes are used in devices such as electrolyzers and fuel cells, which will be important players in the energy transition in place in the industrial and energy sectors. Furthermore, they have been evaluated in recent years also as materials to assist CO<sub>2</sub> capture processes. Their high perm-selectivity is particularly beneficial in striving toward net-zero greenhouse gas emission goals. Such membranes have been evaluated mostly to support electrochemical CO<sub>2</sub> abatement processes such as electrodialysis (ED) and CO<sub>2</sub> electrochemical reduction, which are instrumental in the capture and utilization of CO<sub>2</sub> [1]. In these applications, the ion exchange membrane, characterized by a high charge density and high ion perm-selectivity, facilitates the directional migration, separation, and categorization of ionic substances

under a direct current field. In CO<sub>2</sub> reduction it has been observed that anion exchange membranes (AEM) may perform better than proton exchange membranes [1].

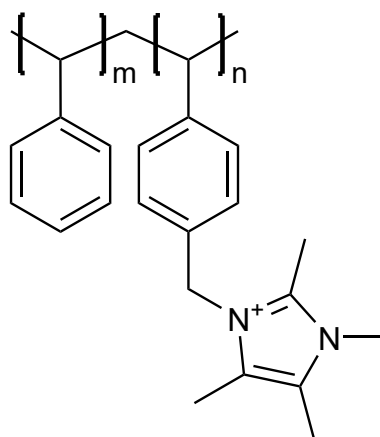
More recently, the use of anion exchange membranes or resin beads has been proposed in a non-electrochemical process, i.e. the moisture-driven carbon capture from air or other sources. In such a process the presence of humidity gradients allows for the flow or adsorption/desorption cycle of carbon in the forms of carbonate and bicarbonate ions through the material [2,3]. These processes exploit the fact that the CO<sub>2</sub> uptake in the AEM material changes as a function of hydration degree: it is maximum in dry conditions and decreases with increasing humidity. Therefore, if an appropriate difference of humidity is provided, via a switch or a gradient, a cyclic or continuous CO<sub>2</sub> removal process can be sustained. This process is a cost-effective approach for CO<sub>2</sub> capture, leveraging water as the driving force: the energy required to concentrate CO<sub>2</sub> is provided by the evaporation of water. The design of appropriate direct air capture schemes within a membrane or cyclic adsorption/desorption layout requires for the knowledge of a number of parameters including the CO<sub>2</sub> sorption and diffusion levels inside the membranes, at different humidity levels. In this paper we provide a direct estimation of the CO<sub>2</sub> uptake in the dry state at 30°C. In order to assess the usability of such membranes for a wide range of CO<sub>2</sub> capture processes, e.g., in the removal of CO<sub>2</sub> from natural gas and biogas, we have tested the behavior of the membrane as a function of pressure in the range between 0 and 8 bar. Furthermore, we have interpolated the experimental data using a correlative model, the Dual Sorption Model, which allows us to extrapolate the CO<sub>2</sub> uptake outside the region experimentally probed. In addition, we have assessed the sorption of CH<sub>4</sub> under the same pressure conditions. The results allow us to evaluate the gas sorption and sorption-based selectivity values in a range of pressures of industrial interest in processes such as biogas upgrading and natural gas refinement.

## 2. Materials and Methods

### 2.1. Materials

Two common anion exchange membrane materials with covalently bonded quaternary ammonium functional groups were analysed in the present work. Fumasep® FAA-3-50 was purchased from the Fuel Cell Store, Bryan, Tx, USA; Sustainion® X37-50 Grade RT was purchased from Dioxide Materials, Boca Raton, FL, USA. The chemical structure of Sustainion® is shown in **Figure 1**: it contains the imidazolium group as the cation instead of the usual quaternary ammonium group. Fumasep® is based on a proprietary polymer backbone with covalently bonded quaternary ammonium functionality.

Both membranes were ion-exchanged into OH-form by soaking in 1 M KOH solution for 24 h at room temperature. The membranes were then rinsed with deionized water and dried overnight at 105 °C under vacuum for the complete removal of water prior to further characterisation.



**Figure 1.** Chemical formula of Sustainion® [4,5].

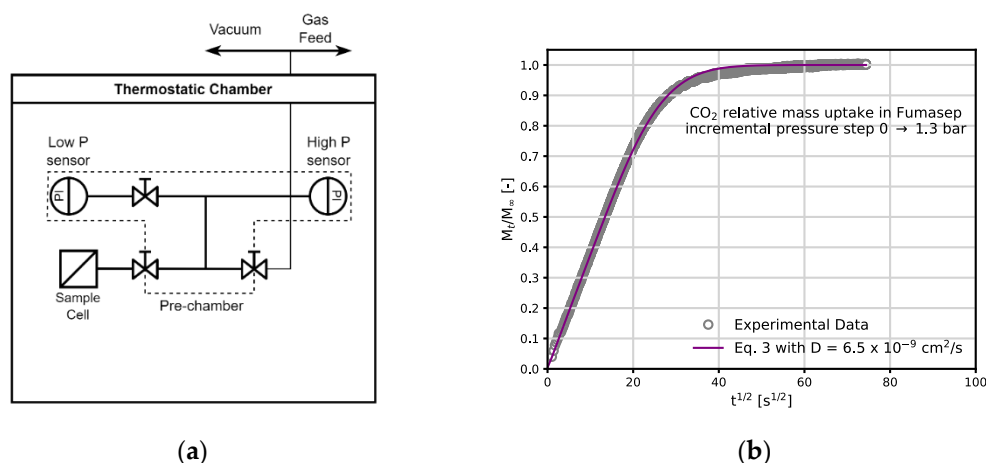
The thickness was measured to be  $50.5 \pm 1.4 \mu\text{m}$  for Fumasep® and  $50.0 \pm 1.5 \mu\text{m}$  for Sustainion®, consistent with the declared commercial value of  $50 \mu\text{m}$  of the dry membranes. The density values of the dry membranes were, according to the literature,  $1.3 \text{ g/cm}^3$  for Fumasep® [3,6] and  $0.9 \text{ g/cm}^3$  for Sustainion® [4,7]. Different literature sources show rather different density values for these materials, depending on the humidity conditions in which they were measured, and on the counterion. The density value does not affect the estimate of diffusivity performed in this work, and affects the solubility value, expressed per unit mass of polymer, only to a limited extent, estimated to be to within 5% using the range of densities reported in the literature.

$\text{CO}_2$  and  $\text{CH}_4$ , used for gas sorption tests, were purchased from BOC UK as specialty grade gas cylinders (Research Grade) with guaranteed minimum purities  $\geq 99.995\%$ .

## 2.2. Gas Sorption

The solubility of  $\text{CO}_2$  and  $\text{CH}_4$  in two anion exchange membranes in the dry state was determined at  $30^\circ\text{C}$  at pressures up to 8 bar in a manometric apparatus, built in-house according to ASTM D1434 [8]. The measurements were repeated at least twice for each gas.

The full equipment set up is reported in **Figure 2a**. A known mass of polymer sample is placed into a sample cell and the sample is conditioned at the test temperature under dynamic vacuum overnight prior to the experiment. The sample chamber is then isolated, by closing the interconnecting valve, and the pre-chamber is filled with the desired amount of gas and left until equilibration. The gas is then left to expand into the sample cell, and the mass uptake by the sample is evaluated by measuring the pressure decrease in a closed and calibrated volume over time until a constant value of pressure is reached. The amount of gas absorbed inside the polymer at such pressure is calculated through a mass balance, given the knowledge of the amount of gas in the sample cell prior to expansion, the amount of gas loaded into the pre-chamber, and the residual amount of gas in the gaseous phase in the sample cell after reaching equilibrium. To perform the next incremental step of the isotherm, the cell is once again isolated, the pre-chamber is filled with a higher amount of gas, the gas is left to expand into the sample cell. The procedure is repeated in such stepwise manner until the desired equilibrium pressure is reached, and the whole sorption isotherm is obtained. The system is placed in a thermostatic chamber with forced air convection (Binder KT series), where the temperature is fixed to within  $\pm 0.1^\circ\text{C}$ . The pressure is monitored by two pressure sensors (Druck UNIK 5000) with full scale of 11 and 36 bar, and an accuracy of  $\pm 0.04\%$  of the full-scale value. The two sensors can be connected separately to the sample cell, to allow for an accurate reading and good level sensitivity in different pressure intervals. Pressure data are collected using the Eurotherm Nanodac™ recorder, controlled via Eurotherm iTools proprietary software (version 9.87).



**Figure 2.** (a) Schematics of the pressure-decay apparatus for gas sorption. (b) Sorption kinetics of  $\text{CO}_2$  in Fumasep® at  $30^\circ\text{C}$  and fitting of  $D$  with Eq. (3). The step initial pressure was 0 and the final pressure was 1.3 bar.

The solubility coefficient,  $S$ , at each equilibrium pressure,  $p$ , is determined as the ratio between the concentration of the gas in the sample,  $c$ , and the equilibrium pressure, as follows:

$$S = \frac{c}{p} \quad (1)$$

Gas concentration dependence on pressure can then be described by several models, based on the nature of the penetrant and the state of the polymer [9].

Sorption isotherms which are concave to the pressure axis, typical for more condensable gases absorbed in glassy polymers, can be described by applying the Dual-Mode Sorption (DMS) model. The DMS model assumes two populations of gas in equilibrium with one another absorbed in the polymer: one absorbed into the dense equilibrium matrix and described by the Henry's law, and one absorbed into the non-equilibrium excess volume associated with the glassy state and described by the Langmuir curve. The gas concentration variation with pressure is then described as sum of these two contributions:

$$c = k_D p + \frac{C'_H b p}{1 + b p} \quad (2)$$

where  $k_D$  is the Henry's law constant,  $C'_H$  is the Langmuir capacity constant, and  $b$  is the Langmuir affinity parameter. For light gases, linear sorption isotherms, typical of Henry's law valid in dilute conditions is retrieved as  $c = k_D p$ .

The penetrant diffusivity in the film,  $D$ , can be evaluated from the sorption kinetics at each sorption step in the isothermal run, by assuming Fickian diffusion and accounting for the variation of penetrant concentration at the film interface [10].

The expression for the mass uptake as a function of time,  $M_t$ , for the mass sorption in a limited volume, where the variation of the interfacial concentration is due to mass sorption in the membrane, is given by [10]:

$$\frac{M_t - M_0}{M_\infty - M_0} = 1 - \sum_{n=1}^{\infty} \frac{2\alpha(1 + \alpha)}{1 + \alpha + \alpha^2 q_n^2} \exp\left(-D \frac{q_n^2 t}{l^2}\right) \quad (3)$$

where  $M_0$  and  $M_\infty$  are the initial and final mass uptake, respectively, and  $\alpha$  is the ratio between the volume of solution and that of the membrane, corrected for the partition coefficient of vapor between the gaseous phase and the polymer,  $l$  is the semithickness of the membrane, while  $q_n$  variables are the positive, nonzero, solutions of the equation:  $tg(q_n) = -\alpha q_n$ . By fitting the experimental data of mass uptake versus time to the aforementioned equation, one obtains the average diffusivity value within the concentration interval inspected in the differential sorption step. An example of the data output from a sorption step and the relative fitting of data to Eq. (4) to obtain  $D$  is reported in **Figure 2b**.

When swelling is associated with the sorption of penetrant into the polymer, the diffusion coefficient can increase with concentration. Oftentimes, such dependence can be described through an exponential correlation:

$$D = D_0 \exp(\beta c_{av}) \quad (4)$$

where  $D_0$  is the pre-exponential coefficient,  $\beta$  the exponential factor, while  $c_{av}$  is the average concentration of the penetrant in the membrane during the sorption step.

The CO<sub>2</sub> diffusion coefficients were determined for the anion exchange membranes in the present work and correlated to the CO<sub>2</sub> concentration. Due to the low sorption levels, it was not possible to determine diffusivity values for CH<sub>4</sub> with a reasonable accuracy.

Given the dense and homogeneous nature of the investigated materials, the CO<sub>2</sub> permeability coefficient,  $P$ , was estimated as a product of its average solubility coefficient and diffusivity coefficient in each step, under the solution-diffusion framework [11,12]:

$$P = \bar{S} \cdot D \quad \text{with} \quad \bar{S} = \frac{c_{i+1} - c_i}{p_{i+1} - p_i} \quad (5)$$



The ideal permselectivity,  $\alpha_{ij}$ , between gas  $i$  and  $j$  is, under negligible downstream pressure conditions, the ratio between pure-gas permeabilities, and can be seen as the product of solubility-selectivity,  $\alpha_{ij}^S$  and diffusivity-selectivity,  $\alpha_{ij}^D$ :

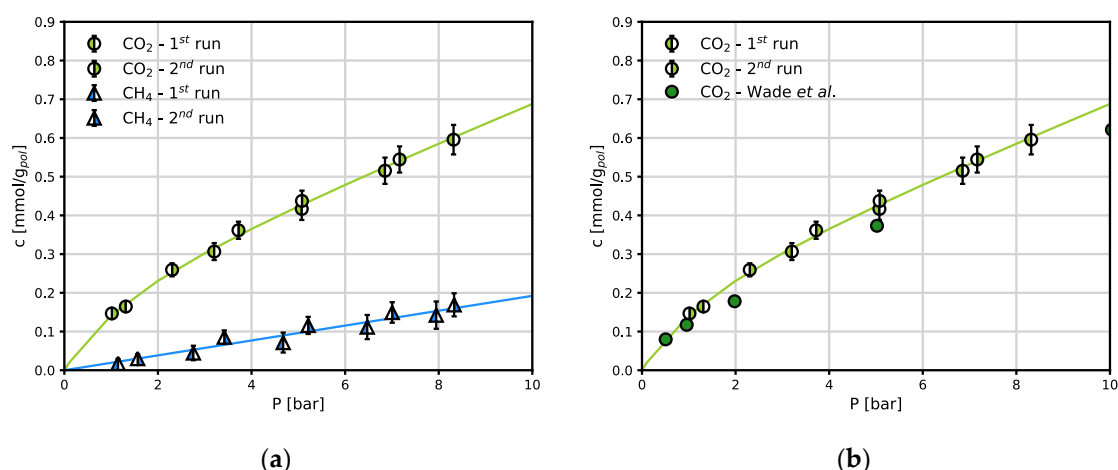
$$\alpha_{ij} = \frac{P_i}{P_j} = \frac{S_i}{S_j} \cdot \frac{D_i}{D_j} = \alpha_{ij}^S \cdot \alpha_{ij}^D \quad (6)$$

The ideal permselectivity is generally used as a first estimate of the separation performance of new materials and its comparison with existing literature data. In the present work, we were able to determine CO<sub>2</sub>/CH<sub>4</sub> solubility-selectivity for the two anion exchange membranes as function of pressure, which gives an initial indication on the separation capability of these materials.

### 3. Results

#### 3.1. CO<sub>2</sub> and CH<sub>4</sub> Sorption Isotherms

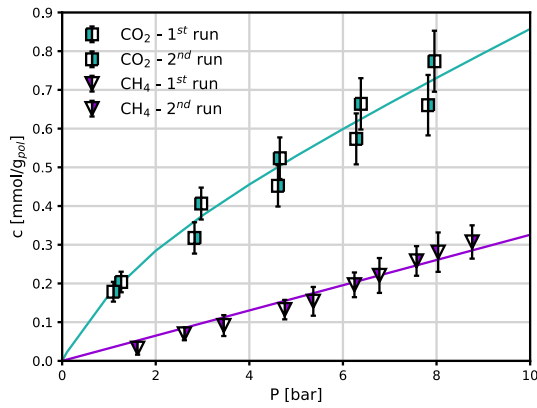
The amount of CO<sub>2</sub> and CH<sub>4</sub> absorbed at the end of an isothermal pressure step is reported in **Figure 3a**, in terms of mmol/kg of dry polymer.



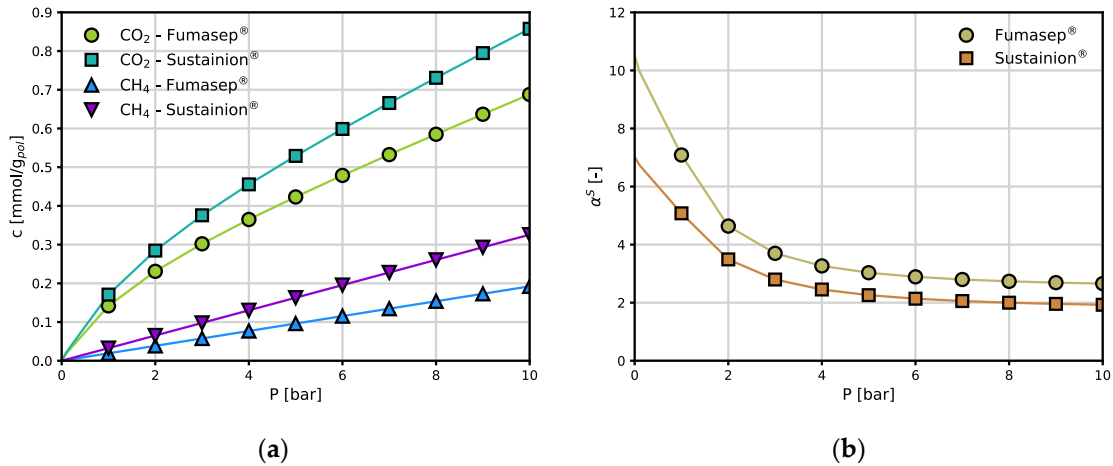
**Figure 3.** Sorption isotherms in Fumasep® at 30 °C: (a) CO<sub>2</sub> and CH<sub>4</sub> sorption isotherms; (b) CO<sub>2</sub> sorption in comparison with the literature [3]. The continuous lines are obtained by fitting the DMS (Eq. 2) or linear Henry's law model.

CH<sub>4</sub> sorption follows Henry's law linear behaviour in the Fumasep® membrane, while CO<sub>2</sub> sorption in the same material follows a convex shape, (**Figure 3a**). For the case of CO<sub>2</sub> sorption in dry Fumasep®, experimental data are available at the same conditions used here, and are shown in **Figure 3b** for comparison. It is clear that the data obtained in this work are in excellent agreement with those obtained by independent researchers who reported CO<sub>2</sub> sorption in Fumasep® [3].

CO<sub>2</sub> and CH<sub>4</sub> sorption isotherms measured under the same conditions in Sustainion® are reported in **Figure 4**. For this polymer, these are the first experimental data of this kind, and there are no literature data to use as benchmark. Sustainion® is characterized by larger CO<sub>2</sub> sorption levels than Fumasep® (**Figure 5a**), and the same qualitative behavior is followed. CH<sub>4</sub> sorption follows a linear trend, as for the case of Fumasep®. The DMS parameters for both gases in both polymers are reported in Table 1: linear isotherms are represented simply by a Henry's law term and the coefficient  $k_D$ . CH<sub>4</sub> uptake is slightly lower in Fumasep® than in Sustainion®.



**Figure 4.** CO<sub>2</sub> and CH<sub>4</sub> sorption isotherms in Sustainion® at 30 °C in the dry state. The solid lines are obtained by fitting the DMS (Eq. 2) or a linear model.



**Figure 5.** Comparison of CO<sub>2</sub> and CH<sub>4</sub> sorption in Fumasep® and Sustainion®: (a) CO<sub>2</sub> and CH<sub>4</sub> sorption isotherms as determined by the DMS (Eq. 2) and linear models; (b) CO<sub>2</sub>/CH<sub>4</sub> solubility-selectivity  $\alpha^S_{CO_2/CH_4}$  as function of pressure, evaluated using DSM models for each gas.

**Table 1.** The DMS model (Eq. 2) correlation parameters for the CO<sub>2</sub> and CH<sub>4</sub> sorption isotherms in the anion exchange membranes investigated, together with the coefficient of determination.

Material	CO <sub>2</sub>				CH <sub>4</sub>	
	$k_D \times 10^2$ mmol g <sup>-1</sup> bar <sup>-1</sup>	$C'_H \times 10^1$ mmol g <sup>-1</sup>	$b$ bar <sup>-1</sup>	$R^2$	$k_D \times 10^2$ mmol g <sup>-1</sup> bar <sup>-1</sup>	$R^2$
Fumasep®	4.80 ± 0.15	2.41 ± 0.24	0.64 ± 0.01	0.997	1.92 ± 0.21	0.934
Sustainion®	5.78 ± 0.02	3.34 ± 0.75	0.51 ± 0.12	0.959	3.26 ± 0.04	0.961

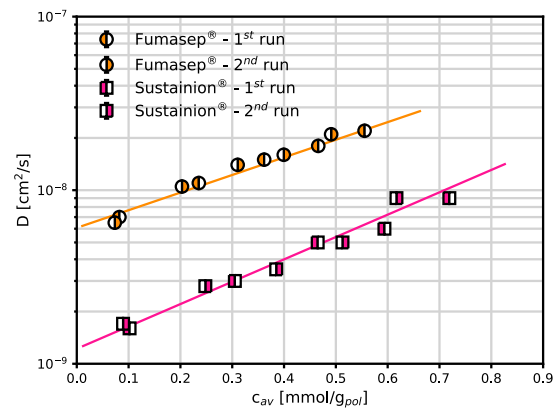
a CO<sub>2</sub> diffusion.

Due to the shape of the solubility isotherm, the solubility coefficient ( $S = c/p$ ) of CO<sub>2</sub> in both materials decreases rapidly with pressure and thus the ideal solubility-selectivity  $\alpha^S_{CO_2/CH_4}$  reported in **Figure 5b**, decreases from values of approximately 10 to above 2. Interestingly, the best separation performance is exhibited at low pressure, that is the one of interest for several CO<sub>2</sub> capture processes.

Overall, the differences between the two materials are limited and quantitative, rather than qualitative: Fumasep® sorbs slightly less CO<sub>2</sub> but is more selective with respect to CH<sub>4</sub> than Sustainion®, in pure gas conditions.

The diffusion coefficients of CO<sub>2</sub> were estimated from the transient mass uptake in the polymers, as described in the methods section. These are the first experimentally obtained CO<sub>2</sub> diffusion values in these polymers: a value was given in the literature for Fumasep® ( $3.8 \cdot 10^{-9}$  cm<sup>2</sup>/s [3]) but it was estimated as the ratio between independent permeability and solubility data. In our case the diffusion

coefficient was obtained directly by fitting the transient sorption data with Eq. 3. The same procedure was also attempted with CH<sub>4</sub> but, due to the small level of sorption in this polymer and the low amount of sample available, it did not yield reliable results. Interestingly, CO<sub>2</sub> diffusion coefficients in both materials follow an exponential dependence on concentration. The CO<sub>2</sub> diffusivity in Sustainion® increases from 1.6 to 9.0·10<sup>-9</sup> cm<sup>2</sup>/s within the range examined, while Fumasep® is significantly higher and goes from 0.7 to 2.2·10<sup>-8</sup> cm<sup>2</sup>/s. These differences could be due to a smaller initial free volume of Sustainion® compared to Fumasep®. However, as it is typical of membranes characterised by smaller initial free volume, the dependence on concentration represented by the parameter  $\beta$  is higher for the Sustainion® membrane than for Fumasep®. The slope is significant and allows the diffusion coefficients to increase by one order of magnitude by increasing the CO<sub>2</sub> pressure from 0 to 8 bar, indicating an appreciable effect of CO<sub>2</sub>-induced swelling on the transport behavior, which should be taken into account when designing the separation process.



**Figure 6.** CO<sub>2</sub> diffusion coefficients in Fumasep® and Sustainion®, determined from sorption transient at 30 °C in the dry state, as function of average CO<sub>2</sub> concentration. The continuous lines are obtained from a best fit to the exponential correlation (Eq. 4).

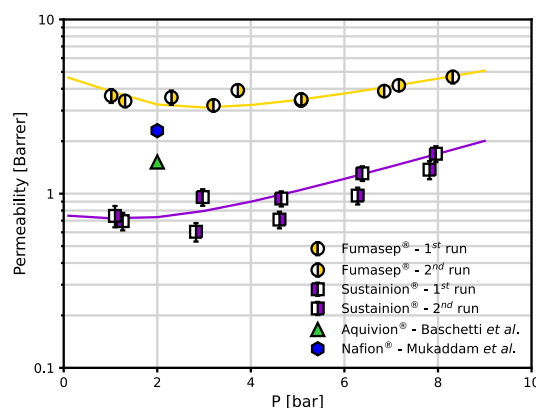
**Table 2.** The exponential fitting (Eq. 4) correlation parameters for CO<sub>2</sub> diffusion at 30 °C in the AEM investigated, together with the coefficient of determination.

Material	$D_0 \times 10^9$ cm <sup>2</sup> s <sup>-1</sup>	$\beta$ mmol g <sup>-1</sup>	$R^2$
Fumasep®	6.07 ± 0.19	2.34 ± 0.15	0.977
Sustainion®	1.22 ± 0.11	2.96 ± 0.31	0.921

a      CO<sub>2</sub> permeation.

Finally, the permeability coefficients were evaluated as the product between diffusivity and solubility for CO<sub>2</sub>, in the two materials (Eq. 5) and reported in **Figure 7**. Due to the combination of solubility and diffusivity effects, Fumasep® shows a higher CO<sub>2</sub> permeability than Sustainion®. Permeability varies from 3.4 to 4.7 Barrer and from 0.7 to 1.7 for Sustainion. Also, as  $S$  decreases sharply in the low-pressure range while  $D$  continues to increase exponentially as  $S$  flattens out at medium pressures, the permeability shows a minimum (3.2 Barrer for Fumasep® at about 3 bar, 0.6 Barrer for Sustainion® at 2.8 bar). This threshold is sometimes indicated as the plasticization pressure in the membrane literature and is typically observed in the CO<sub>2</sub> permeation through glassy polymers.





**Figure 7.** CO<sub>2</sub> permeability coefficients in Fumasep® and Sustainion® at 30°C in the dry state, evaluated as the product of solubility and diffusivity coefficients. The continuous lines are reported as guide to the eye. CO<sub>2</sub> permeability in Aquivion® [13] and Nafion® [14] at 35 °C at 2 bar transmembrane pressure is reported for comparison.

Finally, we report CO<sub>2</sub> dry permeability values obtained in the literature for other ion exchange membranes, namely two fluorinated matrices which go under the trade name of Aquivion® [13] and Nafion® [14]. In particular, Nafion® was tested after being dried at 80°C for 48 h [14], at 35 °C and 2 bar upstream pressure, while Aquivion® was pre-dried at 100°C for 24 h, tested at 35 °C and 2 bar [13]. The permeability of these two materials lies in between the values obtained in this work for Sustainion® and Fumasep®, respectively.

#### 4. Conclusions

In this study, we conducted a detailed analysis of the dry gas transport properties of two commercially available anion exchange membranes, Fumasep® and Sustainion®. Both membranes feature covalently attached quaternary ammonium groups and show potential for use in various clean energy and carbon-neutral applications. Their increasing application in fuel cells, electrolysis, and notably in CO<sub>2</sub> capture and utilization processes makes understanding their basic transport properties such as sorption and diffusion coefficients vital for process design and simulation.

We carried out dry CO<sub>2</sub> and CH<sub>4</sub> sorption tests at 30 °C and pressures up to 8 bar. These tests revealed that CO<sub>2</sub> exhibits a convex-shaped sorption isotherm in both types of membrane, with Sustainion® displaying a marginally higher sorption capacity than Fumasep®. The sorption isotherms for CO<sub>2</sub> can be described using the DSM model. In contrast, CH<sub>4</sub> displayed a linear sorption isotherm, consistent with Henry's law, and showed a lesser mass uptake compared to CO<sub>2</sub>. From these findings, we calculated the ideal solubility selectivity for CO<sub>2</sub> over CH<sub>4</sub> in each membrane. Fumasep® demonstrated a higher selectivity, which decreased with increasing pressure from 10 to 3 over the pressure range examined.

The diffusion coefficients for CO<sub>2</sub> were determined from the transient phase of the sorption tests at varying concentrations. In both membranes, CO<sub>2</sub> diffusivity followed an exponential relationship with its concentration, suggesting significant swelling effects within the membrane structure induced by CO<sub>2</sub>. Notably, Fumasep® exhibited considerably higher CO<sub>2</sub> diffusivity compared to Sustainion®, likely due to a greater free volume.

Permeability curves, calculated as a function of pressure using the solution-diffusion model, showed that dry CO<sub>2</sub> permeabilities of both membranes had a comparable order of magnitude to that observed in fluorinated ion exchange polymers like Nafion® and Aquivion®. The permeability decreased initially with increasing pressure, reaching a minimum at around 2-3 bar, and then increased exponentially. This pattern is typical for glassy polymers, highlighting the complex interplay of solubility and diffusivity under varying pressures.

In conclusion, the results of this study establish a solid groundwork for the development of advanced CO<sub>2</sub> capture and utilization technologies utilizing the selective properties and high charge

density of anion exchange membranes. This includes promising applications such as moisture-driven direct air capture and other innovative processes.

**Author Contributions:** Conceptualization, M.G.D.A.; Methodology, S.K. and K.P.; Software, K.P.; Investigation, K.P.; Data Curation, K.P.; Writing – Original Draft Preparation, M.G.D.A.; Writing – Review & Editing, M.G.D.A. and S.K.; Supervision, M.G.D.A.

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**Conflicts of Interest:** The authors declare no conflicts of interest.

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