



## Gas permeability: A simple and efficient method for testing membrane material/solvent compatibility for membrane contactors applications

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### ABSTRACT

The selection of the most efficient hydrophobic microporous material is a key issue for membrane contactors applications. This holds especially for intensified gas–liquid absorption operations for which stable membrane performances such as non wetting conditions or effective gas permeability are demanded on a long time basis. In this study, the membrane material challenges of post combustion carbon dioxide capture performed in a membrane contactor are developed with a 30% aqueous MEA solution as chemical solvent. A simple, sensitive, non destructive and relevant experimental technique based on gas permeability measurement is proposed in order to estimate membrane / solvent compatibility. The interest of the technique is presented on ten different membrane materials; the materials screening step and the possibility to point out differences in materials ageing issues are shown.

**Keywords:** Membrane contactors; Polymers; Ageing; Carbon dioxide; Capture; Permeability

### 1. Introduction

The essential element in a membrane contactor is obviously the membrane material, which has to ensure maximal interface area, minimal mass transfer resistance and stability in performances. Porous hydrophobic membranes are preferred for this purpose because of their low-cost and wide availability. The key concept of the process is based on the fact that the liquid absorbent cannot penetrate the membrane because its pores are sufficiently small and the membrane material repels the liquid. In practical operation a gas stream is fed along one side of the membrane. The use of a membrane absorber leads to the following advantages:

- Gas and liquid flow are independent, resulting in avoidance of problems encountered in packed/tray columns such as flooding, foaming, channelling and entrainment,
- Equipment will be compact through the use of a hollow fibre membrane leading to specific surface areas in excess of 1,000 m<sup>2</sup>/m<sup>3</sup> compared to 100–200 m<sup>2</sup>/m<sup>3</sup>.

The operational stability relates to the capability of the membrane contactor system to keep the interface for mass exchange intact and its performance stable for a relevant operational period under the normal operational conditions. One important item is that permeation of the solvent through the membrane should not happen throughout the life-time because this will result in the loss of the operational benefits of membrane contactors. Penetration of the membrane pores by the solvent will indeed affect the performances

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of the membrane contactor, because diffusion in gas filled pores is much faster than in liquid filled pores.

This study will more specifically address the membrane stability issues over long time operation, which are determined by:

### 1. Critical entry pressure

The critical entry pressure is the excess liquid side pressure at which the solvent will penetrate the membrane pores. For porous membranes with uniform cylindrical pores, this is governed by the so-called Laplace equation:

$$\Delta P = -2(\gamma/r) \cos \Theta \quad (1)$$

in which  $\Delta P$ : Transmembrane pressure, [N/m<sup>2</sup>]

$\gamma$ : Surface tension of liquid, [N/m]

$r$ : Pore radius, [m]

$\Theta$ : Contact angle, [°]

The contact angle results from the physical affinity between the solvent and the membrane material. A high value indicates a low affinity between the solvent and the membrane material. Eq. (1) shows that the membrane pores will not be wetted if the contact angle is greater than 90°. At contact angles in excess of 90°, Eq. (1) gives the break-through pressure as a function of solvent surface tension and pore size. Consequently, suitable membrane materials for aqueous absorption liquids are non-polar polymers, such as, poly-propylene (PP), poly-ethylene (PE) and poly-tetrafluoroethylene (PTFE). It is important to stress that the critical entry pressure is determined by the largest pores. The knowledge of the pore size distribution (and not only the average pore size) is thus of major importance in that context.

### 2. Chemical interaction between membrane material and solvent

Some solvents will react with the membrane material over a prolonged period of exposure. It is clear that such membrane / solvent combinations are not practical. For instance, polypropylene materials have been occasionally reported to slowly react with DEA [1], which suggests that this membrane material / solvent combination is not feasible.

### 3. Temperature

A higher temperature will lead to a lower surface tension of the solvents and, therefore, a lower critical entry pressure. The chemical stability will also be influenced by temperature. Furthermore, it is known that oxygen induced membrane degradation and at higher temperature, this phenomena is faster.

Apart from the stability issues which have been discussed above, the ideal material for membrane contactor applications should also offer the smallest mass transfer resistance (i.e. the largest mass transfer coefficient). Different methods have been investigated in order to determine the mass transfer coefficient  $k_m$  and three of them are described hereafter.

Generally speaking, the membrane mass transfer coefficient for a specific type of porous membrane depends on the porosity, thickness and tortuosity [2]:

$$k_m = D_{g,e} \cdot \varepsilon / \tau \cdot z \quad (2)$$

where  $D_{g,e}$ : Effective diffusion coefficient in gas filled pores, [m<sup>2</sup>/s]

$z$ : Membrane thickness, [m]

$\varepsilon$ : Membrane porosity, [-]

$\tau$ : Membrane tortuosity, [-]

The effective diffusion coefficient is determined through a combination of bulk diffusion and Knudsen diffusion. Bulk (or molecular) diffusion describes the interaction between the gas molecules; Knudsen diffusion describes the interaction between the gas molecules with the pore walls.

$$1/D_{g,e} = 1/D_{g,b} + 1/D_k \quad (3)$$

where  $D_{g,b}$ : Bulk diffusion coefficient, [m<sup>2</sup>/s]

$D_k$ : Knudsen diffusion coefficient, [m<sup>2</sup>/s]  
which can be written as

$$D_k = 1/3 \cdot d_p (8RT/\pi M)^{0.5} \quad (4)$$

with  $d_p$ : Pore diameter, [m]

$R$ : Gas constant, [J/kmol·K]

$T$ : Temperature, [K]

$M$ : Molecular mass, [kg/kmol]

The membrane mass transfer coefficient is determined by the choice of membrane through the porosity and tortuosity. In general, the porosity should be high and tortuosity should be low, but the membrane should still have sufficient mechanical strength. The pore diameter can also have impact through the Knudsen diffusion coefficient. However, porosity, tortuosity and pore diameter are often unknown because of the difficulty to determine them precisely and of the large pore size distribution.

Given the difficulty to predict the membrane mass transfer coefficient, experimental methods have been developed. A first approach, developed by Qi and Cussler [3] is an indirect method based on permeation of

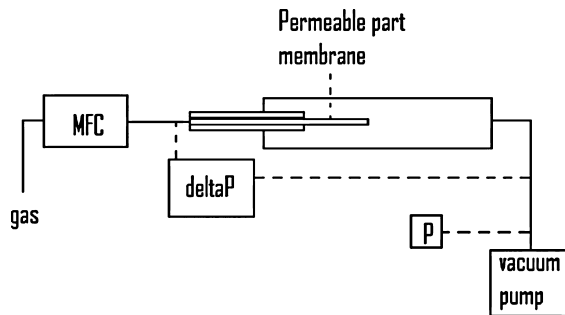


Fig. 1. Experimental set up for direct measurement of membrane mass transfer coefficient in transmembrane distillation operation MFC: mass flow controller; deltaP: pressure drop; P: pressure gauge.

pure gas in a liquid so that the mass transfer resistance in the gas phase  $1/kg$  can be neglected. The mass transfer coefficient in the liquid phase  $k_l$  is then calculated by a selected correlation and  $k_m$  can finally be deduced from the overall mass transfer coefficient  $K$  (which is experimentally determined). The resulting  $k_m$  value combines the imprecision due to the experiments and also the uncertainty of the mass transfer correlations which can induce a large range of  $k_m$ .

Another approach is based on a direct experimental method, only studied for transmembrane distillation operation by Guijt et al. [4]. It considers the permeation of a pure gas in short hollow fibers in order to suppress the influence of the mass transfer resistance in the gas phase, see Fig. 1.

The contribution of both Knudsen diffusion and viscous flow are taken into account in the calculation of the total gas flux through the pores:

$$N = -(1/RT)[K_0 v_M + B_0(P/\mu)]\nabla P \quad (5)$$

with  $N$ : Molar gas flux,  $[\text{mol}/\text{m}^2 \cdot \text{s}]$

$K_0$ : Knudsen diffusion morphology parameter,  $[\text{m}]$

$B_0$ : Viscous flow morphology parameter,  $[\text{m}^2]$

$P$ : Pressure,  $[\text{Pa}]$

$\mu$ : Viscosity,  $[\text{Pa} \cdot \text{s}]$

$\nabla P$ : Derivative from  $P$  in all directions,  $[\text{Pa}/\text{m}]$

$v_M$ : Mean Knudsen molecular velocity,  $[\text{m}/\text{s}]$

with:

$$v_M = (8RT/\pi M)^{1/2} \quad (6)$$

From a practical point of view, a short review of the literature shows a wide range of  $k_m$  data. Indeed, Kumar et al. [5] estimated a value of  $4.62 \cdot 10^{-2}$  m/s, Qi and Cussler [3] measured a value of  $5.1 \cdot 10^{-3}$  m/s and Lin et al. [6] calculated values from  $4.37 \cdot 10^{-4}$  to  $1.14 \cdot 10^{-3}$  m/s for a PP membrane. Yeon and al. [7] reported a value of  $5 \cdot 10^{-4}$  m/s for PTFE and  $10^{-3}$  m/s for PVDF and Lin et al. [6] estimated values from  $10^{-4}$  to  $7 \cdot 10^{-4}$  m/s for the latter material. These results illustrate the diversity of material used for membrane contactors applications and the large uncertainty of the membrane mass transfer coefficient measurements. In a first step, we will address the measurement of membrane mass transfer coefficient of 10 different materials through a simple gas permeability method. The stability of this parameter when the membrane is put into contact with a solvent solution for a long time will be exposed and discussed in a second part.

## 2. Materials & methods

A series of 10 different flat microporous hydrophobic membranes, detailed in Table 1, has been tested for gas permeability (in all experiments, gas stream is air) and ageing evaluation.

The gas permeability set-up, operating under dead end mode and steady state conditions, is schematized on Fig. 2; for each experiment a 10.7 cm diameter flat

Table 1

Characteristics of the different flat microporous hydrophobic membranes tested for the material permeability and ageing tests

Number	Material	Supplier	Reference	Average pore size ( $\mu\text{m}$ )
1	PTFE	Millipore	Fluoropore membrane filters	0.22
2	PTFE	Sartorius	Hydrophobic PTFE membrane filters	0.2
3	PTFE	GE Water	Telsep	0.22
4	PTFE	GE Water	Telsep	5
5	PTFE	GE Water	GE Teflon laminated membrane	0.22
6	PP	GE Water	Polysep Polypropylene	0.1
7	PVDF	Millipore	Durapore membrane filters	0.22
8	PVDF	Pall Gelman	Biotrace transfer membrane PVDF	0.45
9	PVDF	Pall Gelman	Fluorotrans PVDF transfer	0.2
10	Nylon	GE Water	MAGNA Nylon supported	0.1

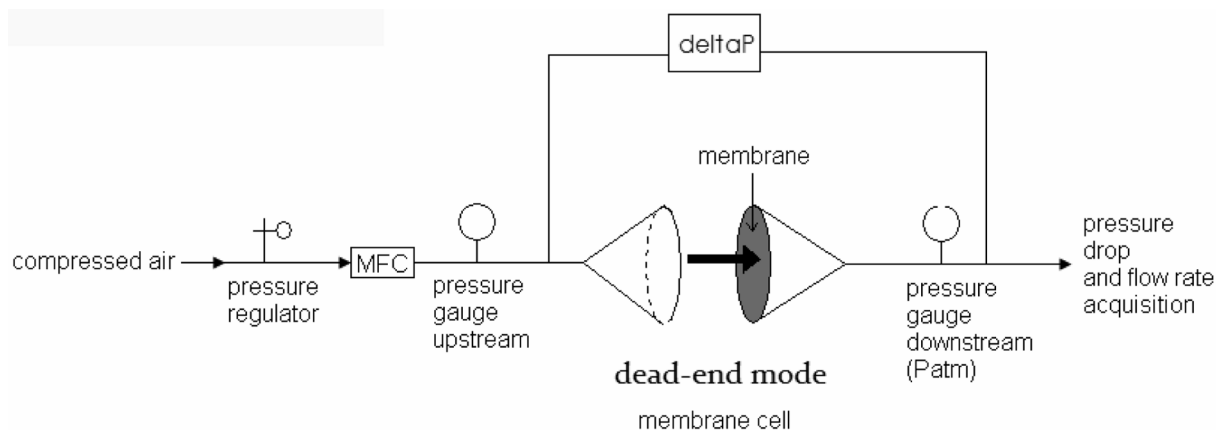


Fig. 2. Experimental set up for direct measurement of membrane mass transfer coefficient and testing of membrane/solvent compatibility MFC: mass flow controller; deltaP: pressure drop.

membrane sample is installed on a support grid and the filter holder cartridge is closed.

The differential pressure drop through the membrane is then measured by a pressure gauge for different controlled air flow rates, regulated by a mass flow meter. The evolution of pressure drop vs. air flow rate, after the support effect is subtracted, gives a direct access to the overall membrane gas permeability. The determination of gas permeability proved to be very sensitive and the measurements, typically repeated five times (for increasing and decreasing air flow rates) for each membrane sample, very reproducible. It can be seen on Fig. 3 that no hysteresis effect takes place. All the experiments reported afterwards have been performed under room temperature conditions.

Two different types of experiments have been performed:

- Dry membrane samples are used as received in order to determine the gas permeability of native materials
- Ageing tests are performed as follows: different 10.7 cm flat membrane samples were immersed in a 30% wt MEA aqueous solution at room temperature. The vessel was closed and after a certain time (ranging from a few days to more than two months), the membrane sample was removed, quickly swept by filter paper, washed in distilled water and dried under vacuum. A gas permeability measurement was performed according to the same procedure than for the native materials. The sample could also be immersed again in case of need in order to follow the evolution with time of the effective membrane permeability.

### 3. Results & discussion

#### 3.1. Membrane mass transfer resistance of native samples through gas permeability measurement

In a first step, the effective gas permeability of the 10 different membranes listed in Table 1 has been determined in order to compare the performances of the native materials. The structure of the different types of membranes used for membrane contactors applications differ in fact largely, as can be seen on the Scanning Electron Microscopy (SEM) pictures shown on Fig. 4.

The four different membrane materials shown on Fig. 4 obviously differ strongly in terms of pore shape, pore size distribution, porosity, connectivity... As a result, the effective gas permeability of the material can hardly be predicted from the membrane thickness, the average pore size and the membrane porosity (expressed in Eq. (2)), even though this assumption is

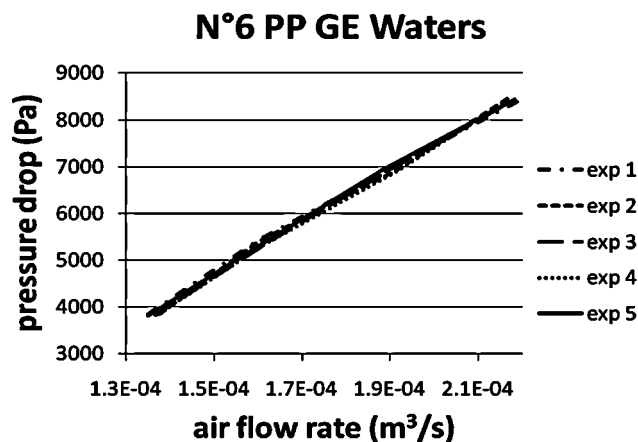
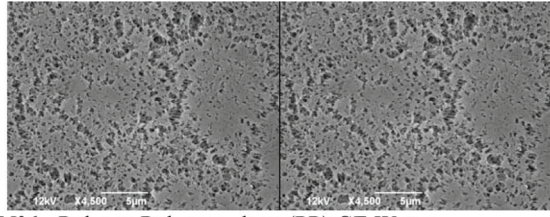
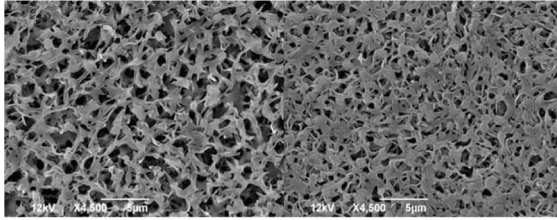


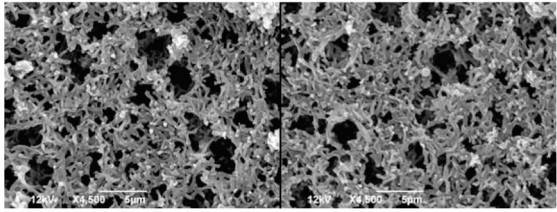
Fig. 3. An example of reproducibility experiments on PP membrane (Details of the membrane in Table 1).



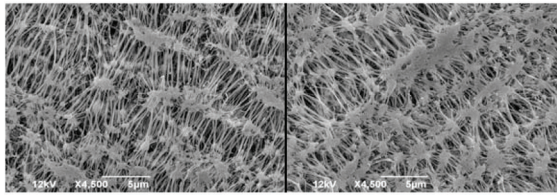
N°6 - Polysep Polypropylene (PP) GE Water



N°8 - PolyVinylidene Fluoride (PVDF) Pall Gelman



N°10 - MAGNA Nylon Supported GE Water



N°5 - Polytetrafluoroethylene (PTFE) GE Water

Fig. 4. SEM pictures of four different membrane materials. Left: native samples. Right: Samples after 2 months exposure in MEA aqueous solutions. Indications correspond to the list given in Table 1.

often proposed for the sake of simplicity. This point will be addressed hereafter.

Fig. 5 shows the experimental effective gas permeability results of the 10 different native membranes. A broad range of permeabilities, covering one order of magnitude and reflected by the different slopes is obtained. This first set of results has been used as the reference material permeability in what follows.

From these experiments, permeability and mass transfer coefficient in the membrane can be tentatively calculated.

The starting point is the effective gas permeability of the membrane material, which can be obtained from the experimental results and is defined as:

$$P' = Qz/(\Delta PS) \quad (7)$$

with  $P'$ : Permeability, [ $m^3 \cdot m / (m^2 \cdot s \cdot Pa)$ ]

$Q$ : Air flow rate, [ $m^3/s$ ]

$z$ : Membrane thickness, [ $m$ ]

$\Delta P$ : Transmembrane pressure, [ $Pa$ ]

$S$ : Surface, [ $m^2$ ]

It is obvious that the permeability calculated by this way depends on the nature of the gas because of difference of viscosity.

The membrane mass transfer coefficient  $k_m$  is often expressed as:

$$k_m = D_{eff}/z \quad (8)$$

with  $D_{eff}$ : Effective diffusion coefficient, [ $m^2/s$ ]

$D_{eff}$  is calculated by equaling these two expressions:

$$N = D_{eff}\Delta c/z \quad (9)$$

$$N = P' \cdot \Delta P / (Vm \cdot z) \quad (10)$$

with  $\Delta c$ : Concentration difference, [ $mol/m^3$ ]

$Vm$ : Molar volume, [ $m^3/mol$ ]

Based on this approach, the membrane mass transfer coefficient for a strict convection mass transfer, expressed in m/s, can be calculated (results are given in Table 2).

It is of interest at this stage to explore how the diffusional mass transfer coefficient of the membrane, which is expected to hold for classical membrane contactors applications (no convection flux), could be estimated. Two sets of porosity/tortuosity data have been taken to calculate  $k_m$  in pure diffusion mode (Eq. (2)) and compare with the data obtained under pure convection mode.

The latter can be estimated based on the permeability of a set of monodispersed capillaries of porosity  $\epsilon$  and tortuosity  $\tau$ :

$$P' = \epsilon \cdot dp^2 / 32\mu \cdot \tau \quad (11)$$

As the porosity is not provided by the supplier, 30% and 85% (which is a very high value) have been used. Additionally, tortuosity cannot be directly determined and it can range between 1 (for straight pore geometry) and 10 in some cases. The pure convection values obtained through this calculation range from  $4.4 \cdot 10^{-4}$  m/s to 0.21 m/s. The agreement of the highest value is rather fair with most of experimental data (Table 2).

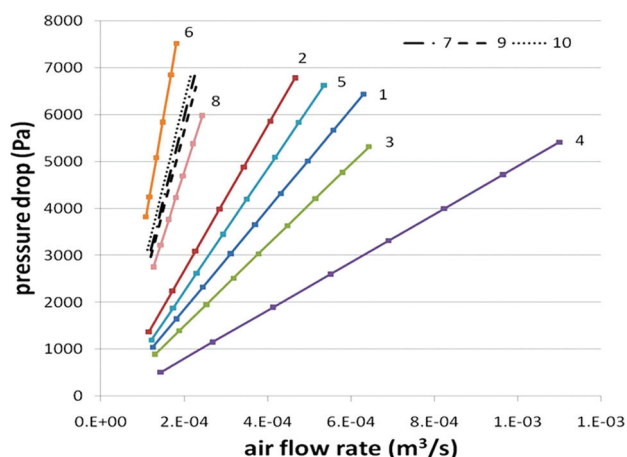


Fig. 5. Gas permeability results for the 10 different membrane materials (Membranes are detailed in Table 1).

Finally, the mass transfer coefficient data for pure diffusion range, which are necessary for real membrane contactors applications, have been calculated through Eq. (2) with a bulk diffusion coefficient of  $10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ . The so obtained values range from 0.002 m/s to 0.13 m/s and logically show larger values than the experimental data reported in the literature, namely  $5 \cdot 10^{-4}$  to  $5 \cdot 10^{-2}$  m/s. The overestimation can be expected to result from the diffusion coefficient since Knudsen contribution has not been taken into account. Unfortunately, similarly to other studies in this field, pore size distribution is required for more rigorous and representative calculation to be performed. We conclude at this stage that direct gas permeability measurement provides a fast and simple method for a comparison between different materials for membrane contactors application. The prediction of a relevant mass transfer coefficient for diffusion conditions remains however difficult. In the next section, we describe the interest of the gas permeability method for a completely different purpose: the identification of

ageing phenomena in membrane contactors applications on a long time basis.

### 3.2. A novel technique for the identification of membrane ageing

In a second step the different membrane materials have been immersed for a given time (between 5 days and 2 months) in MEA solution and possible changes in the effective gas permeability (i.e. in the slope of the curves) have been looked for. The target application of this case study would be the capture of carbon dioxide from flue gases, which has been intensively investigated in the literature. The key concept behind this strategy is to explore the potentialities of a simple, efficient, sensitive, representative and non destructive technique for membrane materials screening. More specifically, it is expected that any change occurring in the material structure due to the chemical solvent influence, be they polymeric matrix swelling, deformation, partial wetting due to a change in the hydrophobicity, leaching, chemical modification of the polymer (e.g. molecular weight distribution), precipitates in the pore... will have an incidence on the effective gas permeability. Any of these effects would suggest that the membrane material shows some limitations and it can be an indication of potential difficulties and problems at the membrane contactor scale. A series of results are shown on Fig. 6 for four different membrane materials: PTFE, PP, PVDF and Nylon. It can be seen that PTFE permeability remains remarkably stable, even after 2 months exposure (no change in the slope  $\Delta P/Q$ ); this is consistent with several observations reported in previous studies. For instance, stable separation performances have been observed by Nishikawa et al. [8] over 6,000 h operation with membrane contactors based on PTFE membranes. A completely different pattern is observed for PP, which shows a significant change in gas permeability after one month exposure.

Table 2

Permeability and mass transfer measurements for the 10 different native membranes. The range of  $k_m$  values have been obtained based on convective conditions (Eqs. 7–9)

Membrane number	Average pore diameter ( $\mu\text{m}$ )	Thickness ( $\mu\text{m}$ )	Permeability ( $\text{m}^3 \cdot \text{m} \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$ )	Permeability (barrer)	$k_m$ (m/s)
1	0.22	150	1.51E-09	2.02E+08	1.02
2	0.2	65	4.38E-10	5.84E+07	0.68
6	0.1	100	2.21E-10	2.94E+07	0.22
7	0.22	120	3.68E-10	4.90E+07	0.31
8	0.45	147	8.51E-10	1.13E+08	0.59
9	0.2	127	3.71E-10	4.95E+07	0.30
10	0.1	120	3.52E-10	4.69E+07	0.30

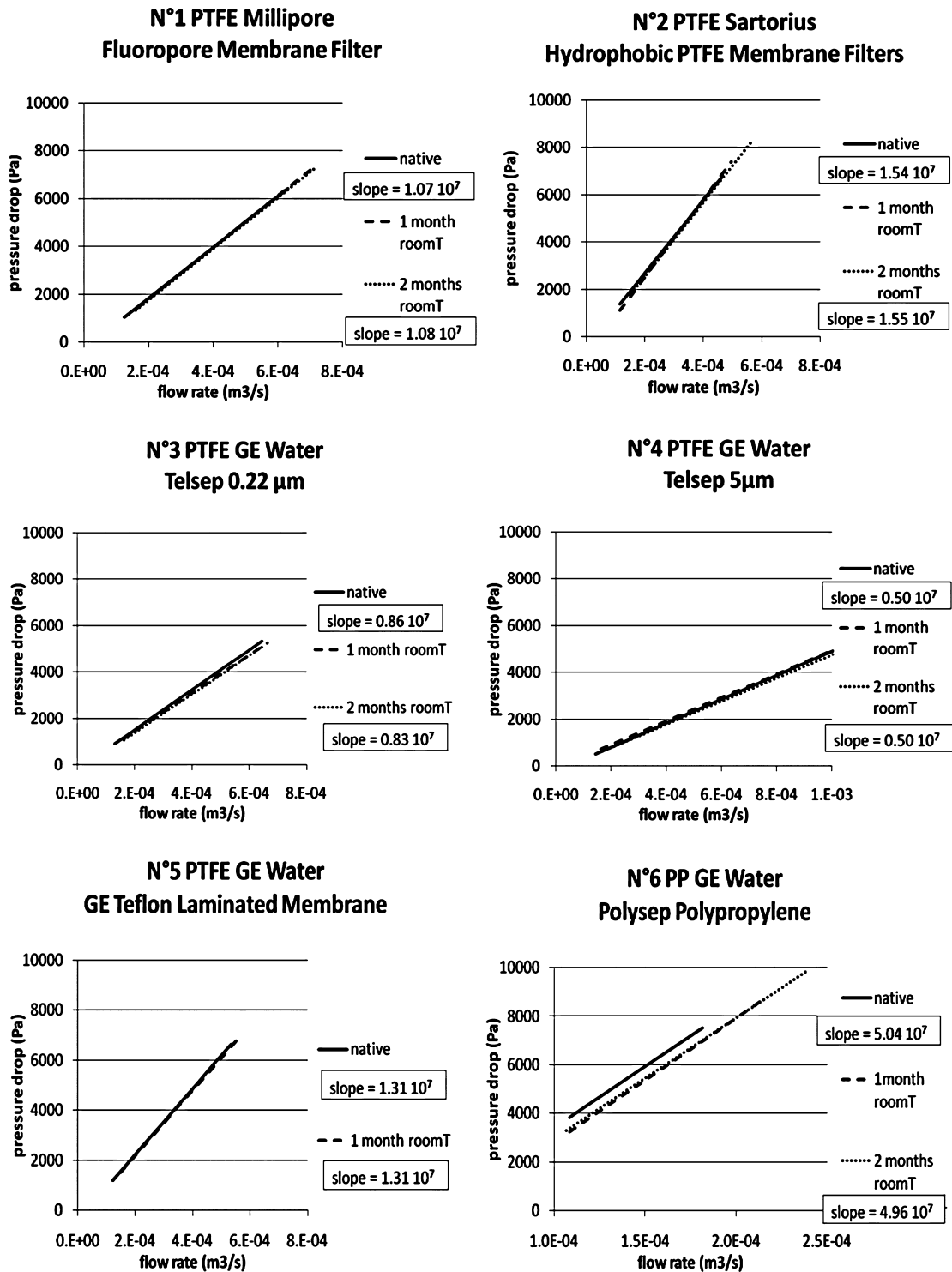


Fig. 6. Characterization of ageing for different membranes after one and two months by gas permeation measurement (slope  $\Delta P/Q$  of native and after ageing membranes are indicated).

Again, this sensitivity of PP towards MEA (or DEA) solutions corroborates the results of Wang et al. [1], based on destructive and complex SEM analysis. PVDF and Nylon, which to our knowledge have not been

investigated in terms of stability with MEA solutions, show a behavior close to that of PP.

More generally, for all the results shown on Fig. 6, it can be seen that no significant change seems to occur

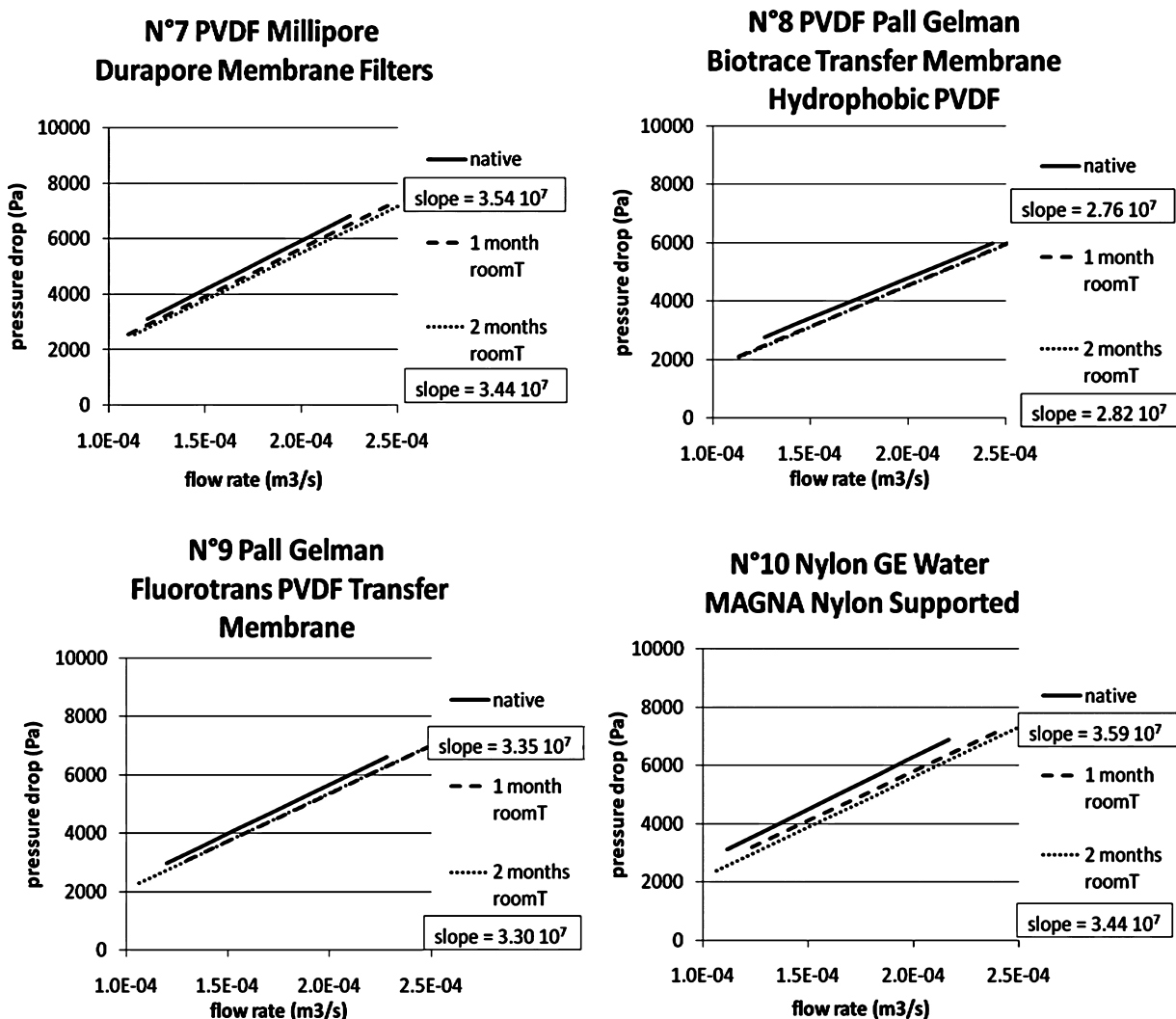


Figure 6 (Continued)

on membrane permeability after one month exposure (the 2 months results are similar to the one month results). This duration might correspond to a reasonable time of exposure and confirms the need to explore the solvent/material compatibility issues on a long term basis. We conclude at this stage that gas permeability measurement offers a novel, simple and quick assessment when the compatibility between a candidate membrane material and a given solvent has to be evaluated. It is important to stress that the only technique which has been reported up to now in order to point out ageing issues in membrane contactors applications is based on SEM analysis; Fig. 4 shows that no visible change could be detected on the four membrane samples after solvent exposure by this approach. This shows the interest of our direct and non destructive method.

#### 4. Conclusion

The objective of this work was to explore, through a case study, the material challenges of membrane contactors. Membrane/solvent compatibility has been investigated on the largely unexplored long term issues on a typical case study of the CO<sub>2</sub> postcombustion capture (MEA aqueous solution). The broad range of gas permeability of native materials has been pointed out by a simple air permeability technique. Furthermore, it has been shown that the evolution of the gas permeability of a candidate material offers a simple and efficient method to identify the stability of the membrane. PP and PVDF have shown significant changes when exposed to MEA solutions for more than one month; PTFE membranes showed a remarkable stability. More generally,



the results suggest that gas permeability changes should be systematically used as an identification technique of the stability of the material. Interestingly, the technique could easily be extended to hollow fibers and modules for which its non-destructive character would be of major interest (i.e. possibility to achieve in place measurement after the modules are rinsed and partly dried by air sweep).

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### Symbols

$B_0$	viscous flow morphology parameter, [m <sup>2</sup> ]
$D_{\text{eff}}$	effective diffusion coefficient, [m <sup>2</sup> /s]
$D_{\text{g,b}}$	bulk diffusion coefficient, [m <sup>2</sup> /s]
$D_{\text{g,e}}$	effective diffusion coefficient in gas filled pores, [m <sup>2</sup> /s]
$D_k$	Knudsen diffusion coefficient, [m <sup>2</sup> /s]
deltaP	pressure drop, [Pa]
$d_p$	pore diameter, [m]
$K$	overall mass transfer coefficient, [m/s]
$K_0$	Knudsen diffusion morphology parameter, [m]
$k_g$	mass transfer coefficient in the gas phase, [m/s]
$k_l$	mass transfer coefficient in the liquid phase, [m/s]
$k_m$	mass transfer coefficient in the membrane, [m/s]
$M$	molecular mass, [kg/kmol]
$N$	molar gas flux, [mol/m <sup>2</sup> .s]
$P$	pressure, [Pa]
$P'$	permeability, [m <sup>3</sup> .m/(m <sup>2</sup> .s.Pa)]
$Q$	air flow rate, [m <sup>3</sup> /s]
$R$	gas constant, [J/kmol.K]
$r$	pore radius, [m]

$S$	surface, [m <sup>2</sup> ]
$T$	temperature, [K]
$V_m$	molar volume, [m <sup>3</sup> /mol]
$z$	membrane thickness, [m]

### Greek letters

$\Delta P$	transmembrane pressure, [N/m <sup>2</sup> ]
$\Delta c$	concentration difference, [mol/m <sup>3</sup> ]
$\gamma$	surface tension of liquid, [N/m]
$\Theta$	contact angle, [°]
$\varepsilon$	membrane porosity, [-]
$\tau$	membrane tortuosity, [-]
$\mu$	viscosity, [Pa.s]
$\nabla P$	derivative from $P$ in all directions, [m/s]
$u_m$	mean Knudsen molecular velocity, [m/s]

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