

Evaluating Water Vapor Permeance Measurement Techniques for Highly Permeable Membranes

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Abstract: The cup method and dynamic moisture permeation cell (DMPC) method are two common techniques used to determine the water vapor permeation properties of a membrane. Often, ignoring the resistance of boundary air layers to the transport of water vapor results in the water vapor permeance of the membrane being underestimated in practical tests. The measurement errors are higher with highly permeable membranes. In this study, the two methods were simulated using COMSOL Multiphysics platform and the extent of the error was evaluated. Initial results showed that the error is equally high in both methods. With the correction for the still air gap, the cup method produces a relatively reduced error. In the DMPC method, reducing the error caused by the boundary air layer by increasing the sweep speed can produce higher instrument error. Highly accurate and precise instrument is needed for DMPC method; however, its error is still higher than that in the cup method. Simulations also show that lowering the test pressure is favorable to both methods.

Keywords: cup method simulation, dynamic moisture permeation cell method simulation, water vapor permeability

1 Introduction

The use of membranes for applications involving the removal or transfer of high amount of water vapor has driven the need for the evaluation of water vapor permeance of membranes with high water vapor permeance (up to 6.8×10^{-6} mol/m².s.Pa [Xing, Rao, TeGrotenhuis, Canfield, Zheng, Winiarski and Liu (2013)]) and high selectivity. Such applications include air dehumidification [Yang, Yuan, Gao and Guo (2015); Metz, Van de Ven, Potreck, Mulder and Wessling (2005)], membrane heat and vapor recovery [Zhang and Jiang (1999)] and vapor/gas separation [Lin,

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Thompson, Serbanescu-Martin, Wijmans, Amo, Lokhandwala and Merkel (2012); Krull, Fritzmann and Melin (2008); Metz, Van De Ven, Mulder and Wessling (2005); Roy, Hussain and Mitra (2013); Scovazzo (2010); Sijbesma, Nymeijer, van Marwijk, Heijboer, Potreck and Wessling (2008)].

There are currently two widespread methods, the cup method [ASTM (2014)] and the dynamic moisture permeation cell (DMPC) method [ASTM (2009)], used for measuring water vapor transmission rates (WVTR) and thus water vapor permeance. Each method has its own variations [Metz, Van de Ven, Potreck, Mulder and Wessling (2005); Gennadios, Weller and Gooding (1994); Huang (2008); Huang and Qian (2008); Zhang (2006)], advantages and disadvantages [Huang and Qian (2008); McCullough, Kwon and Shim (2003)].

In the cup method, a membrane of a specific area covers an upright cup (Figs. 1 a) or inverted cup (Fig. 1 b), with the cup being filled with desiccant, water or salt solution to keep the relative humidity (RH) inside the cup fixed at a certain value. The cup is placed inside a temperature and humidity chamber with air or nitrogen atmosphere at controlled RH. In order to refresh the air/gas outside the cup, the air/gas in the chamber is circulated at a speed higher than 152 m/min as recommended in ASTM E96 - 14 [ASTM (2014); McHugh, Avena-Bustillos and Krochta (1993)]. In this cup method, the change in the mass of the cup's content is monitored and used to quantify the water vapor permeation of the membrane. The advantage of this method is that it employs simple apparatus to conduct the test.

In the dynamic moisture permeation cell (DMPC) method, one side of the membrane is kept at fixed RH by blowing a fast-flowing feed stream of air or gas over the membrane (Figs. 1 c). On the other side of the membrane, a sweep air, helium or nitrogen is passed over. The fluid flows at the opposing sides of the membrane can be countercurrent or concurrent. They can be blown from one end to the opposite end of a rectangular membrane (Fig. 1 c), or radially from the center of a circular membrane surface to the circumference or in the opposite direction [Metz, Van de Ven, Potreck, Mulder and Wessling (2005)]. Water vapor diffuses through the membrane from a space with higher RH to one with lower RH, causing a change in RH of the sweep stream. The RH change is measured and used to calculate the permeability characteristic of the membrane. This method can be coupled with a gas chromatograph system to determine the permeability and selectivity of several gases or vapors at the same time, though the apparatus setup and operations are more complicated [Xing, Rao, TeGrotenhuis, Canfield, Zheng, Winiarski and Liu (2013)]. There are also setups, which are hybrids of the cup and DMPC methods, reported in literature [Huang (2008); Zhang (2006)].

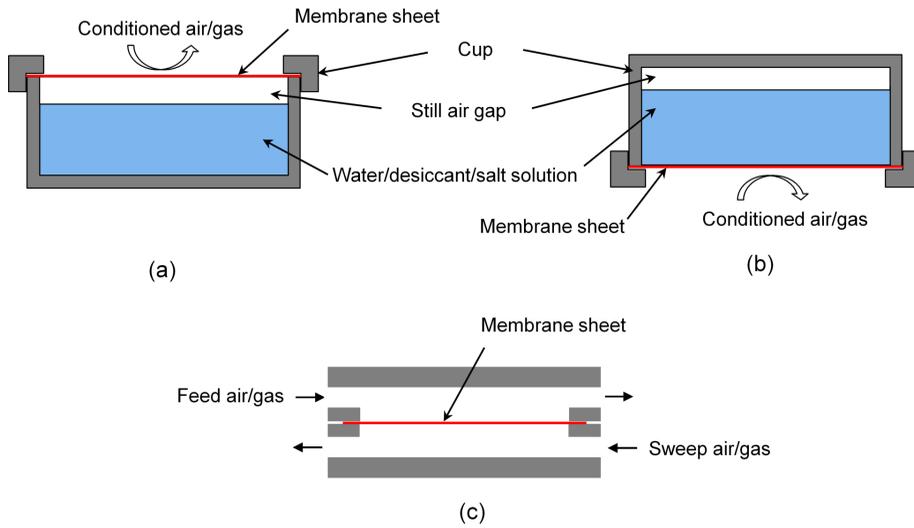


Figure 1: Some examples of experimental setups to determine water vapor permeance: (a) upright cup method; (b) inverted cup method and; (c) counter-current flow DMPC method

For the cup method, the WVTR ($\text{mol}/\text{m}^2 \cdot \text{s}$) is given as [ASTM (2014)]:

$$WVTR = \frac{\Delta m}{t \cdot A \cdot M_w} \quad (1)$$

where Δm is the change in mass of the cup (g), t is the time taken for that change in mass (s), A is the area of the membrane (m^2), and M_w is the molecular weight of water (g/mol).

For the DMPC method, it is determined from [ASTM (2009)]:

$$WVTR = \frac{\delta RH \cdot P_s \cdot V}{A \cdot R \cdot T} \quad (2)$$

where δRH is the change in relative humidity between the incoming and outgoing stream of the sweep air/gas, P_s is the saturation pressure of water vapour (Pa), V is the volumetric flow rate (m^3/s), R is the universal gas constant ($\text{J}/\text{mol} \cdot \text{K}$), and T is the temperature of the measurement (K).

In order to quantify the ease with which water vapor can go through a membrane, vapor permeance (k , $\text{mol}/\text{m}^2 \cdot \text{s} \cdot \text{Pa}$) is employed and defined as the amount of water vapor that goes across a unit area of the membrane under a unit water vapor trans-membrane pressure. With the assumption that the air resistance to the water vapor

transport is negligible, the apparent water vapor transmembrane pressure, and thus apparent permeance, can be determined by

$$k_{app} = \frac{WVTR}{\Delta P_{w,app}} \quad (3)$$

where $\Delta P_{w,app}$ is the apparent water vapor transmembrane pressure (Pa), the driving force for water transmission through the membrane [ASTM (2014)].

For the cup method, the apparent water vapor transmembrane pressure is the water vapor pressure difference between the desiccant/water/salt solution surface inside the cup and the air/gas outside the cup. For the DMPC method, generally, the water vapor transmembrane pressure is the average water vapor pressure difference between feed stream and sweep stream [Metz, Van de Ven, Potreck, Mulder and Wessling (2005); ASTM (2009)].

For both methods, treating the resistance of air as negligible leads to an underestimation of water vapor permeance and causes a certain error for the measurement [Metz, Van de Ven, Potreck, Mulder and Wessling (2005); Gennadios, Weller and Gooding (1994); McHugh, Avena-Bustillos and Krochta (1993); Hu, Topolkarayev, Hiltner and Baer (2001)]. The error is small for a low permeance membrane, whose resistance is much larger than the resistance of air on both sides of the membrane. The error is higher for a high permeance membrane if no correction is appropriately applied. So far, only the resistance of still air has been determined and considered for permeance calculations in the cup method [ASTM (2014)]. The resistance of a moving gas and how it affects the measurement error are still not determined and evaluated in both methods.

Water vapor permeances obtained experimentally are not actual but apparent values. Therefore, although experimental analysis and comparison of the two methods have been done [Gennadios, Weller and Gooding (1994)], the extent of the measurement errors due to air resistance in both methods is not well-known. In particular, the error in the DMPC method, in which no applicable correction has been reported, is completely unknown. Therefore, what is the most appropriate method for a certain membrane and how to minimize the error in each method are questions yet to be addressed.

In this study, computational analysis was utilized to simulate the extent of the errors due to air resistance in a cup setup and a counter current flow DMPC setup. The error after the applicable corrections for the cup method was evaluated. The effects of relative humidity, temperature and pressure on the error of measured apparent and corrected permeances were also simulated. Favorable testing conditions to minimize the error are subsequently discussed.

2 Simulation

2.1 Assumptions

2D simulations for the experimental setup were developed employing the COMSOL Multiphysics 5.0 platform, an engineering modeling software based on the well-developed finite element method [Atluri (2005)]. The consistency between experimental and simulation results for heat and mass transfer involving air flows using COMSOL has been reported in literatures [Bui, Chen, Nida, Chua and Ng (2015); Toujani, Djebali, Hassini, Azzouz and Belghith (2014); Lamloumi, Hassini, Lecomte-Nana, Elcafsi, Smith, Li, Huang, Ai and Tian (2014)]. The following assumptions were made when developing these models:

1. Humid air approximates an ideal gas and the fluid flow of air is plug flow.
2. Water vapor transport is governed by the Fick's law of diffusion and convection. The mass balance equation under isothermal steady state equilibrium is

$$\mathbf{u} \cdot \nabla c + \nabla \cdot (-D\nabla c) = 0 \quad (4)$$

where c is the concentration of the water vapor (mol/m^3), D is the water vapor diffusion coefficient (m^2/s), u is the velocity vector (m/s). c is related to water vapor partial pressure and relative humidity as below:

$$c = \frac{P_w}{RT} = \frac{P_s RH}{100RT} \quad (5)$$

P_w is the water vapor partial pressure (Pa), RH is the relative humidity and P_s is the saturation pressure of water vapor (Pa).

The water vapor diffusion coefficient (D) is calculated from the empirical equation [Massman (1998)]:

$$D = 2.19 \cdot 10^{-5} \left(\frac{P_o}{P} \right) \left(\frac{T}{273.15} \right)^{1.81} \quad (6)$$

where P is the ambient pressure (Pa), P_o is the standard atmospheric pressure (101325 Pa) and T is the temperature (K).

3. The water vapour permeance of the membrane is fixed and does not depend on the air RH. The water vapor transmission rate is proportional to the partial pressure difference between the two sides of the membrane, as in the below equation:

$$WVTR = k |\Delta P_w| = k |P_w^1 - P_w^2| \quad (7)$$

where k is the water vapor permeance of the membrane ($\text{mol}/\text{m}^2 \cdot \text{s} \cdot \text{Pa}$), ΔP_w is transmembrane pressure and P_w^1 and P_w^2 are the water vapor partial pressure at two sides the membrane (Pa).

4. For the cup method, the changes in temperature and air gap due to the water evaporation/absorption are ignored.

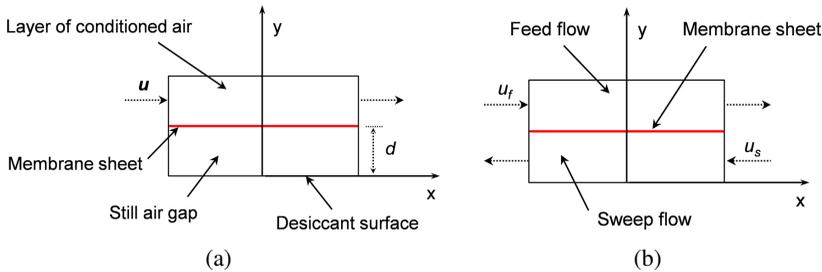


Figure 2: Schematic diagrams for simulations of (a) cup setup and (b) DMPC setup

2.2 Cup method setup

The upright cup setup shown in Fig. 1 a is modeled and simulated. The working membrane is modeled as a 10 mm long thin permeable barrier having constant water vapor permeance (k), placed between an outside layer of conditioned air and a still air layer in the cup as shown in Fig. 2 a. In order to reduce the resistance to mass transfer, the outside air layer is moving at u m/s. Water vapor transport in the outside air is governed by the Fick's law of diffusion and convection. Its mass transfer process is depicted via equation (4). Water vapor transport in the still air gap inside the cup is governed only by the Fick's law of diffusion. Its mass transfer process is simulated using equation (4) without the first term on the left hand side, which accounts for convective transport due to the velocity u .

The apparent water vapor permeance can be determined:

$$k_{app} = \frac{WVTR}{|P_w^{out} - P_w^{in}|} \quad (8)$$

where k_{app} is the apparent water vapor permeance ($\text{mol}/\text{m}^2 \cdot \text{s} \cdot \text{Pa}$), P_w^{out} and P_w^{in} are the water vapor partial pressure of outside conditioned air and at the water/desiccant/salt solution surface respectively (Pa).

The error of k_{app} compared with k is determined as:

$$error = \frac{k - k_{app}}{k} \cdot 100\% \quad (9)$$

The influence of the thickness of the still air gap (d), the conditioned air's RH , temperature and pressure on the *error* is studied judiciously.

2.3 DMPC method setup

The counter flow DMPC setup shown in Fig. 1 c is simulated. The working membrane is a 10 mm long thin permeable barrier between a feed and a sweep flows, as shown in Fig. 2 b. Flow velocities of feed air and sweep gas are u_f and u_s respectively. Water vapor transport in both feed and sweep flows are governed by the Fick's law of diffusion and convection and the mass balance equation for both flows is equation (4).

The apparent water vapor permeance can be determined:

$$k_{app} = \frac{WVTR}{\left| \overline{P_w^{feed}} - \overline{P_w^{sweep}} \right|} \quad (10)$$

where $\overline{P_w^{feed}}$ and $\overline{P_w^{sweep}}$ are the mean partial pressures of water vapor in the feed and sweep flows respectively (Pa), and are calculated with the following equations:

$$\overline{P_w^{feed}} = \frac{\left| P_w^{feed\ in} + P_w^{feed\ out} \right|}{2} \quad (11)$$

$$\overline{P_w^{sweep}} = \frac{\left| P_w^{sweep\ in} + P_w^{sweep\ out} \right|}{2} \quad (12)$$

where $P_w^{feed\ in}$ and $P_w^{feed\ out}$ are the water vapor partial pressures at the inlet and outlet of the feed flow respectively (Pa), and $P_w^{sweep\ in}$ and $P_w^{sweep\ out}$ are the water vapor partial pressures at the inlet and outlet of the sweep flow respectively (Pa).

The error of k_{app} compared with k is determined using equation (9). The influence of the sweep gas velocity, pressure, conditioned air's RH and temperature on the error is analyzed and quantified.

3 Results and discussion

3.1 Transmembrane water vapor partial pressure, ΔP_w

Fig. 3 a shows the water vapor partial pressure along the y axis in a dry cup method setup at different membrane permeances. The relative humidity of the outside conditioned air is set at 70%. A desiccant like silica gel or calcium chloride is modelled in this setup and the relative humidity at the desiccant surface is set at 0%. The temperature is 298K. The outside air layer is moving at $u = 10$ m/s. The water vapor

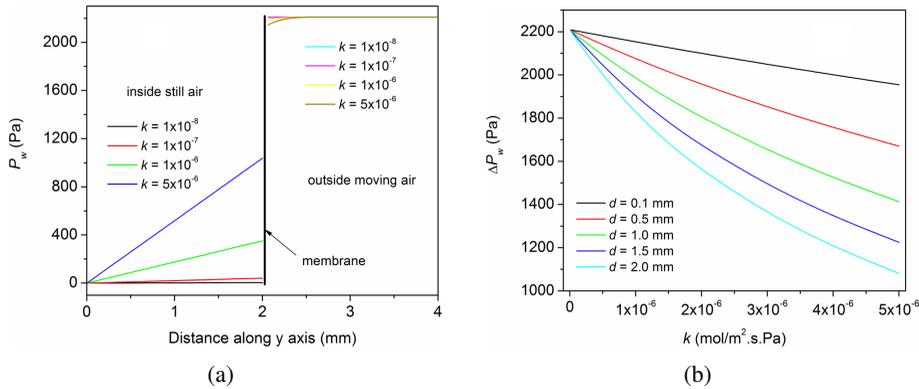


Figure 3: (a) water vapor partial pressure along the y axis and (b) transmembrane pressure in dry cup method setup.

permeance of the membrane (k) is varied from 10^{-8} to 5×10^{-6} mol/m².s.Pa. The result shows that there is an accumulation of water vapor on the inner side of the membrane, causing an increase in water vapor partial pressure near the membrane. This accumulation is small with low water vapor permeance spanning from 10^{-8} to 10^{-7} mol/m².s.Pa and increases sharply with higher water vapor permeance. There is still a significant amount of water vapor dissipation on the outer side of the membrane when water vapor permeance is high, causing a drop in pressure near the membrane, even though the conditioned air is moving at 10 m/s, 4 times higher recommended velocity in ASTM 96 [ASTM (2014)]. As a result, the actual water vapor transmembrane pressure (ΔP_w) is much lower than the water vapor pressure difference between the desiccant surface and outside conditioned air.

Fig. 3 b shows the dependence of the water vapor transmembrane pressure on the membrane permeance and the thickness of the still air (d) between the desiccant surface and the membrane. The actual water vapor transmembrane pressure decreases with higher water vapor permeance and thicker air gap.

Fig. 4 a shows the water vapor partial pressure along the y axis in a DMPC setup. The relative humidity of the feed air and the sweep gas is set at 70% and 0%, respectively. The temperature is 298K. The feed air and the sweep gas are moving at 10 and 0.5 m/s, respectively. The water vapor permeance of the membrane (k) varies from 10^{-8} to 5×10^{-6} mol/m².s.Pa. Similar as the cup method, there are significant water vapor accumulation and dissipation caused by the resistance of air phases on both sides of the membrane in this DMPC setup. This results in a lower water vapor transmembrane pressure compared with the mean water vapor partial pressure difference between the feed air and the sweep gas. As shown in Fig. 4 b, the actual water vapor transmembrane pressure decreases with higher water vapor permeance and slower sweep gas velocity.

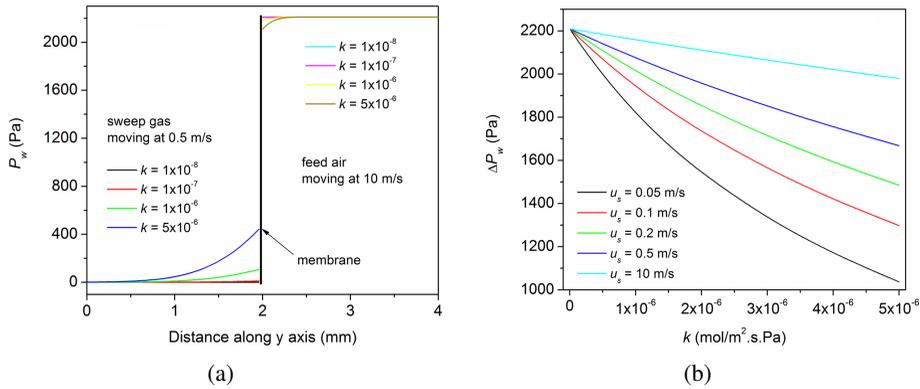


Figure 4: (a) water vapor partial pressure along the y axis and (b) transmembrane pressure in a dynamic moisture permeation cell setup.

3.2 Apparent and actual permeance

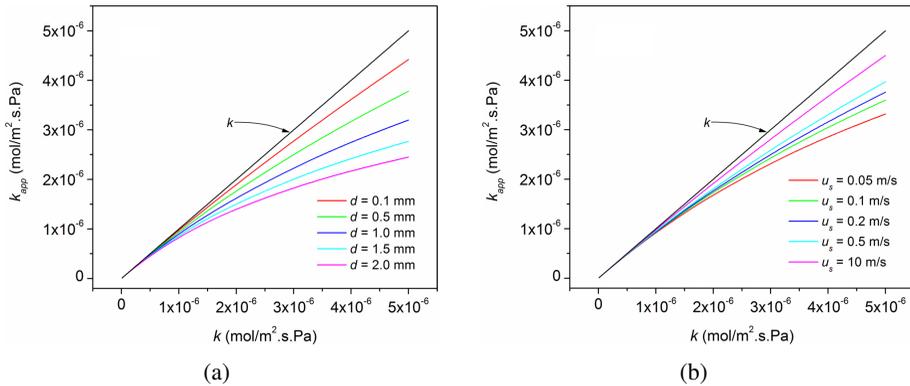


Figure 5: Comparison between apparent and actual permeances in (a) cup setup and (b) DMPC method

During the practical testing of both methods, water vapor permeance is determined based on the assumption that the mass transport resistance of the air on both sides of the membrane is negligible. This leads the apparent permeance computed using equations (8) and (10) to be lower than the actual water vapor permeance. The underestimation of water vapor permeance is shown in Fig. 5. From the two graphs, it is seen that the apparent permeance diverges from the actual permeance as membrane permeance increases. Further, the divergence increases with higher air gap in the cup method and slower sweep velocity in the DMPC method. It is therefore apparent that the resistances to mass transfer in the gas boundary layers on both sides of the membrane cannot be neglected, especially in case of highly water permeable

membranes. Simplifying calculations in practical tests by ignoring this resistance will cause error of the measurement. The errors determined by equation (9) for the two methods are shown in Fig. 6.

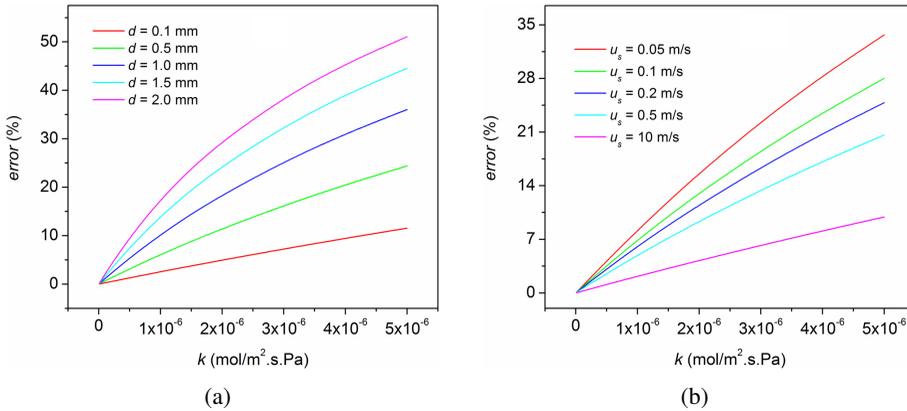


Figure 6: Error caused by ignorance of mass transport resistance of air boundary layers in (a) the cup method and (b) the DMPC method.

As shown in Fig. 6, both methods potentially evolve large errors if the resistances of the air boundary layers are not taken into account. The error increases with higher water vapor permeance. A thicker air gap in the cup method and a lower sweep velocity in the DMPC method also cause higher error.

3.3 Correction for resistance of still air

According to ASTM E96-14 [ASTM (2014)], all measurements that result in permeance values of more than 2-perms ($6.33 \times 10^{-9} \text{ mol/m}^2 \text{ s Pa}$) require corrections. The correction for resistance due to the still air is based on the water vapor permeance of air (k_{air} , $\text{mol/m}^2 \cdot \text{s} \cdot \text{Pa}$):

$$k_{air} = \frac{D}{d \cdot RT} \quad (13)$$

The water vapor permeance with the correction (k_{cor} , $\text{mol/m}^2 \cdot \text{s} \cdot \text{Pa}$) is determined as follows:

$$\frac{1}{k_{cor}} = \frac{1}{k_{app}} - \frac{1}{k_{air}} \quad (14)$$

The error of the measurement with the correction is determined by equation (9) with the replacement of k_{app} with k_{cor} . With this correction, the measured water

vapor permeance skews closer to the actual value and the error becomes smaller as shown in Figs. 7 a and b. Unlike the error of k_{app} shown in Fig. 6 a, the error of k_{cor} shown in Fig. 7 b does not vary significantly with the still air gap. This error is attributed to the resistance of the air boundary layer outside the cup, which has not been taken into account in the calculation of k_{cor} . Determining the resistance of moving air is difficult. One way to reduce this error is by increasing the refreshing velocity of the outer air boundary layer, as shown in Fig. 7 c.

For the inverted cup method in which there is no still air gap, the measured water vapour permeance would be similar to the corrected water vapour permeance values shown in Fig. 7 a. For the convenience of not applying the corrections, the inverted cup method can be used.

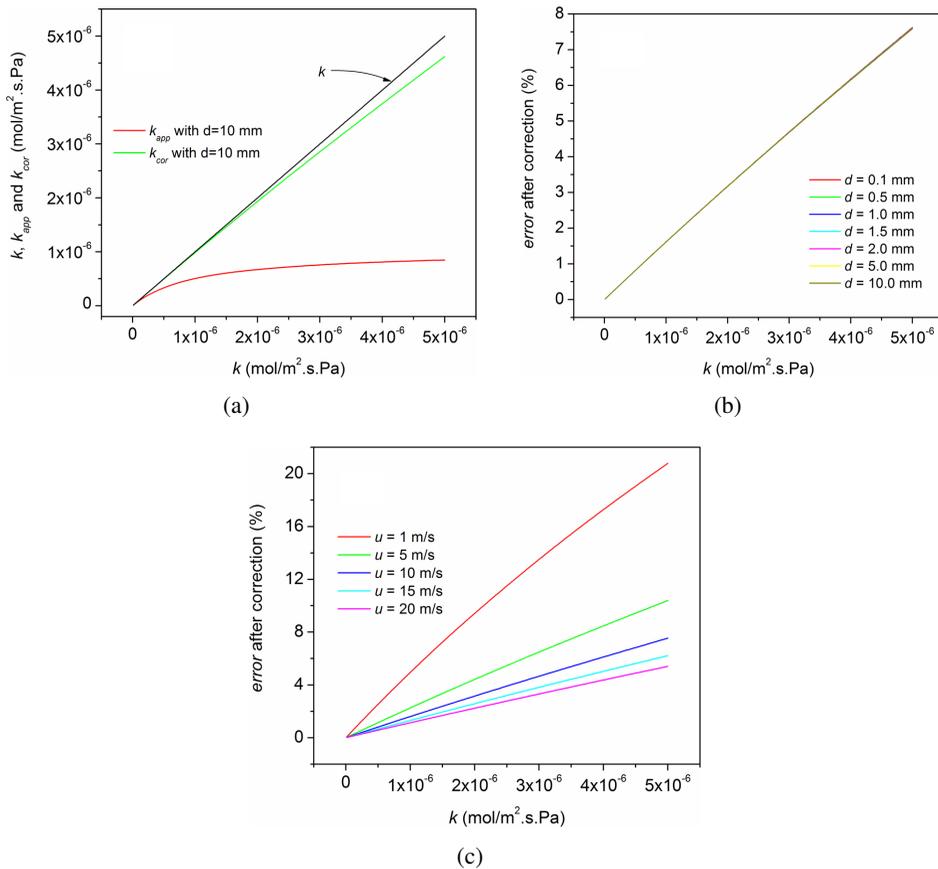


Figure 7: (a) Comparison between k_{app} , k_{cor} and k when the still air gap is 10 mm and refreshing velocity is 10 m/s; (b) error of k_{cor} when varying the still air gap; and (c) error of k_{cor} when varying the refresh air velocity.

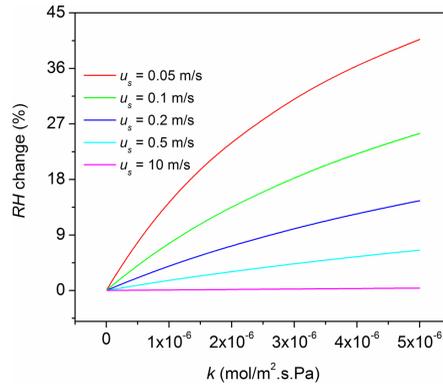


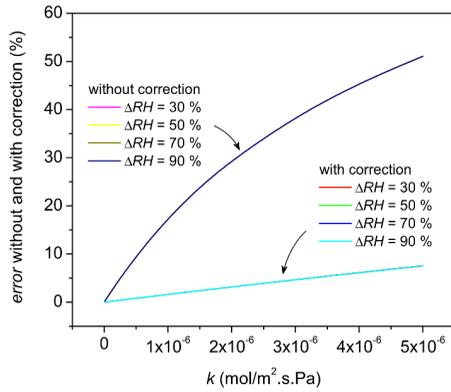
Figure 8: RH change between sweep gas inlet and outlet

In the DMPC method, there has been no reported literature on such a kind of correction for the DMPC method. Therefore, only by increasing the sweeping velocity, the error can be reduced as shown in Fig. 6 b. However, the change in RH also becomes smaller as shown in Fig. 8. The smaller change in water content can magnify the instrument error. This means that there is always a tradeoff between the error caused by the resistance of the air boundary layers and the instrument error with the increase of sweeping velocity. Highly accurate and precise instrument is needed for the DMPC method. However, this does not ensure that the measurement error of the DMPC method is lower than that of the cup method. This is because with the sweep velocity of 10 m/s, at which the sweep stream's RH is almost unchanged (as shown in Fig. 8), the error of the DMPC method (shown in Fig. 6 b) is still higher than the error after correction of the cup method with fresh velocity higher 10 m/s (shown in Fig. 7 c.).

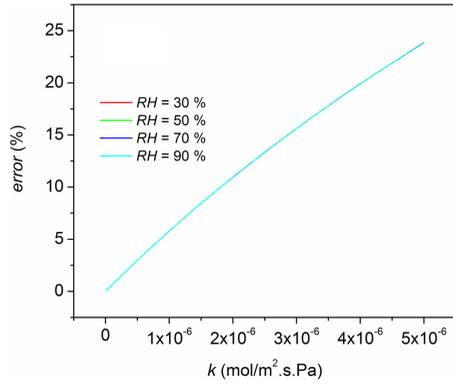
3.4 Effects of RH, temperature and pressure on measurement error

Adopting the assumption that the water vapor permeance of a membrane does not change with RH of the air, the simulation results show that the measurement error also does not depend on the difference in RH values between both sides of the membranes. As shown in Fig. 9 a, the curves for the error of k_{app} and k_{cor} overlap at differing RH values outside the cup while the RH inside the cup controlled using desiccant is kept at 0%. The result is also applicable for the wet-cup method.

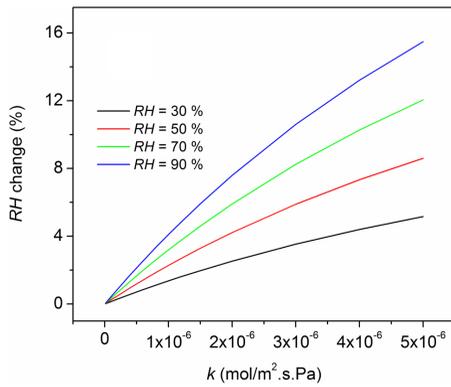
The independence of measurement error from RH is also observed in the case of the DMPC method as shown in Fig. 9 b. The error curves overlap at different feed RH values while the sweep inlet RH is kept at 0%. However, the lowering of the feed RH leads to small RH changes in the sweep stream as shown in Fig. 9 c, causing higher instrument errors. Therefore, when the DMPC method is to be considered, a higher RH difference between the two flows is desirable.



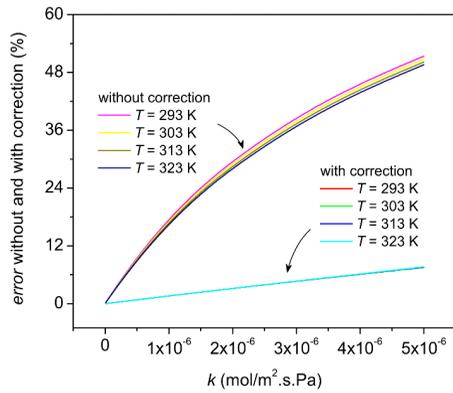
(a)



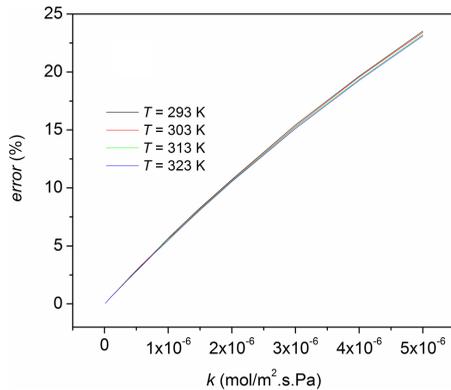
(b)



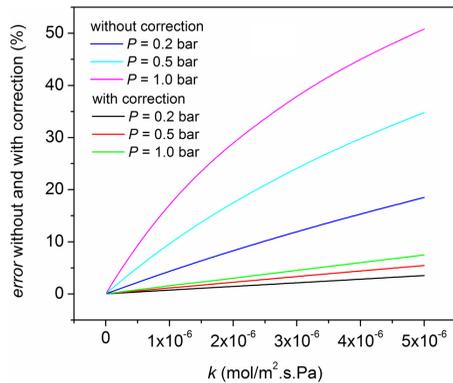
(c)



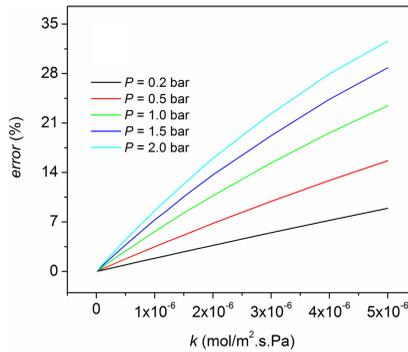
(d)



(e)



(f)



(g)

Figure 9: Effects of RH (a, b and c), temperature (d, e) and pressure (f, g) on measurement errors in the cup (a, d, f) and DMPC (b, e and g) methods and sweep gas's RH change in DMPC method (c).

Adopting the assumption that the water vapor permeance of a membrane is independent of temperature, it follows that a change in temperature just affects the diffusion of water vapor in the air. In both methods, increasing process temperature leads to higher water vapor diffusion and a lower resistance of the boundary air layer. Consequently, lower errors are achieved, as shown in Fig. 9 d and e. However, when the temperature increases from 293 K to 323 K, the error of k_{app} for both the cup and DMPC methods decrease only slightly while the error of k_{cor} is almost unchanged for the cup method.

Pressure affects the water vapor diffusion in air as apparent through equation (6). A decrease in pressure will result in higher diffusion effect while lowering the resistance of air to water vapor transport. In both methods, higher errors are obtained under high pressure conditions as shown in Fig. 9 f and g. This result is consistent with the report that concentration polarization effects cannot be neglected in high pressure applications [Lüdtke, Behling and Ohlrogge (1998)].

4 Conclusions

In this work, two commonly used techniques, the cup and DMPC methods, were studied by numerical analysis to evaluate water vapor permeance of highly permeable membranes. Under room condition and without proper corrective intervention, both methods give high errors due to the effect of water vapor transport resistance of the boundary air layers on both sides of the membrane not being considered. Because the resistance of still air can be determined, the cup method provides a corrected water vapor permeance value closer to the actual one without compro-

mising instrument error. Highly accurate and precise instrument does not ensure a lower error in the DMPC method than the cup method. Both methods can be markedly improved by lowering process pressure.

Acknowledgement: The authors gratefully acknowledge the financial support provided by both Agency for Science, Technology and Research (A*Star) and Ministry of National Development (MND) through their Green Building Joint Grant (no: 112 176 0023) and National Research Foundation (NRF) Competitive Research Programme (CRP) NRF2011NRF_CRP003_003.

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