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### IMPACT OF GAS DIFFUSION LAYER PROPERTIES ON LIQUID WATER BREAKTHROUGH PRESSURE IN POLYMER ELECTROLYTE FUEL CELL

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#### ABSTRACT

Liquid water transport through gas diffusion layers (GDLs) with different thicknesses and different amounts of polytetrafluoroethylene (PTFE) is studied by taking an *ex-situ* approach. Droplet static contact angles on GDLs were also measured. Using a constant flow syringe pump, liquid water was introduced to the surface of GDL sample and the breakthrough pressure at which liquid water passes through the GDL and forms droplet on the other side of the GDL was measured by a differential pressure transducer. Liquid water can pass through the pores when its pressure exceeds the capillary pressure. It was observed that the breakthrough pressure increases with GDL thickness. Also, applying some amount of PTFE to an untreated GDL was observed to significantly increase the breakthrough pressure while more amount of PTFE within the GDL had negligible effect on liquid water breakthrough pressure. Using measured breakthrough pressures and contact angles, maximum pore diameter of GDLs were calculated based on the GDL's single layer model.

#### INTRODUCTION

Water management in polymer electrolyte fuel cell (PEFC) is still one of the most important concerns of scientists of this field. As a PEFC operates, oxygen is reduced in cathode that is accompanied by water production. Produced water in the cathode should be well drained to secure a continuous and optimal

cell performance. Accumulated liquid water within the cathode can fill open pores of the GDL and block the transport of oxygen to the catalyst layer. Reduced flux of oxygen to the catalyst layer decreases the cell performance. Therefore, taking an appropriate approach for preventing liquid water accumulation in GDL pores seems inevitable to render an optimum cell performance. Despite tremendous efforts allocated to improve the performance of PEFCs, water transport mechanism within the GDL is not still quite clear to the researchers.

It is a common practice to load GDLs with some hydrophobic agent such as polytetrafluoroethylene (PTFE) to improve water and gas transport properties [1, 2]. The effect of PTFE treatment of GDL on cell performance have been studied in many works [2–7]. Although loading GDLs with some amount of PTFE was observed to increase the overall cell performance, a comprehensive knowledge about the impact of PTFE content within the GDL on liquid water transport through the GDL is not yet achieved.

There are some parameters that can be used to quantitatively study the water transport behavior within and on the surface of the GDL. Among them, one can refer to the droplet static contact angle that describes GDL's surface energy. Anderson [8] reported that for droplet contact angles of less than 50°, spontaneous imbibition of liquid water occurs into a porous media and for materials showing contact angles greater than 50°, positive displacement pressure is required to force water into the porous media. Since the contact angles that water droplets make

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on both untreated and treated GDL surfaces are higher than this value ( $140^\circ$  and  $150^\circ$ , respectively, in this study), spontaneous imbibition of water never occurs into the GDLs. The external contact angle, on the other hand, only reveals information about the surface energy of the external surface of the GDL and does not provide any information about the surface energy of the inner layers of the GDL [9]. Therefore, contact angle is not an adequate parameter describing liquid water transport through the GDL.

Liquid water transport through the GDL can also be studied by measuring liquid breakthrough pressure. The breakthrough pressure is said to be corresponding to the appearance of a non-wetting phase on the opposite surface of a porous layer such as GDL [10]. Early attempts of measuring liquid water breakthrough pressure of GDL was done by Benziger et al. [11] Using simple water column setup, they were able to measure the breakthrough pressure of both carbon paper and carbon cloth. It was observed that carbon clothes showed lower breakthrough pressure compared to carbon papers. Also, repeating the experiment, they observed much lower breakthrough pressure for initially wet samples compared to virgin dry GDL samples. During last few years there were some studies done on measuring liquid water breakthrough pressure for some few GDL materials but a comprehensive study of measuring such pressure for different combination of Toray carbon paper thickness and PTFE content has not been reported [10, 12–16].

In this work, liquid water breakthrough pressure was measured for Toray carbon papers with different thicknesses and different PTFE contents. Toray carbon papers of TGP\_030, TGP\_060, TGP\_090, and TGP\_120, each with different amount of PTFE content, were used as GDL. In general, it was observed that GDL thickness directly affects the liquid water breakthrough pressure while the PTFE content within the GDL was observed to have negligible effect on this pressure.

## EXPERIMENTAL

The experimental setup used in this study consists of a differential pressure transducer, a syringe pump, and a house-made sample holder designed for this study. Figure 1 shows the experimental setup used in this study. The sample holder was made up of two polycarbonate plates holding GDL sample between and tightened together with eight screws. Liquid water was injected to the surface of the GDL via a rigid capillary tube of  $250\mu\text{m}$  diameter (U\_111, Upchurch). The capillary tube was placed in one of the polycarbonate plates in such a way that the tip of the tube was aligned with the interior surface of the polycarbonate plate. A through-all hole, centered by the position of the capillary tube, with diameter of 5mm was machined on the the other polycarbonate plate to facilitate water breakthrough. The syringe pump injected fresh water at the rate of  $500\mu\text{lh}^{-1}$  on the back side of the GDL. The differential pressure transducer (PX163\_120D5V,

Omega) recorded the variation of liquid water pressure as water was injected. The pressure transducer was connected to a computer and the signals were analyzed using quickDAQ software (developed by Data Translation, Malborough, MA). Sampling was performed at 50Hz to provide enough records for rapid pressure variation. In order to have exact and reliable pressure based on transducer output, the transducer was calibrated using a simple water column setup. Toray carbon papers with different thicknesses and different PTFE contents were used as GDL. Toray carbon papers were loaded with PTFE based on the procedure introduced in [17]. Briefly, GDL samples were dipped in emulsion of PTFE (60 wt.% dispersion in  $H_2O$ , ALDRICH) and deionized water with different ratios (to yield different PTFE content within the GDL) for ten hours and were dried at  $120^\circ\text{C}$  for one hour. Dried samples were then sintered at  $360^\circ\text{C}$  for one hour. GDL samples were cut in  $10 \times 20\text{mm}$  pieces and were put inside the sample holder between two polycarbonate plates in such a way that the capillary tube was positioned on the center of the sample. Teflon sheet was used around the GDL sample as the gasket to provide appropriate sealing within the sample holder. GDL properties tested in this study are listed in Table 1. Three runs were conducted for each case and the results obtained were found reproducible for each test condition.

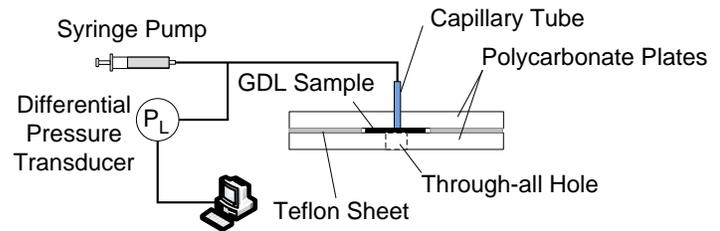


FIGURE 1: Experimental apparatus

## RESULTS AND DISCUSSION

Liquid water breakthrough pressure was measured for Toray carbon papers with different thicknesses and different amount of PTFE content. Liquid pressure was monitored over the time until breakthrough occurred on the surface of the GDL. Liquid pressure increased from some minimal pressure up to the maximal pressure, representing the breakthrough pressure, for each GDL. Obtained results were compared with results published by other groups. Liquid breakthrough pressure was also used to estimate GDL maximum pore diameter based on the simple GDL model

**TABLE 1:** Properties of GDLs used in this study

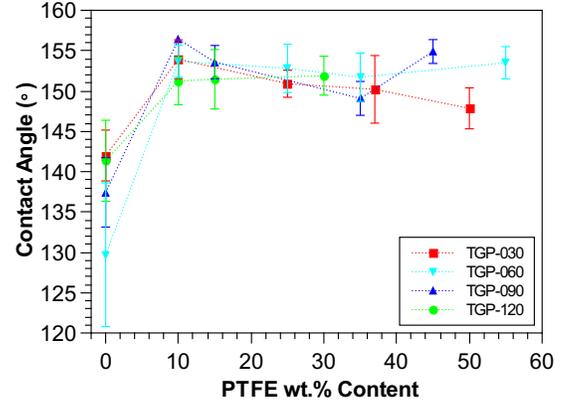
Toray Carbon Paper Type	Manufacturer Specified GDL Thickness ( $\mu\text{m}$ )	Manufacturer Specified Porosity	Air Permeability ( $\text{m}^2$ )	PTFE Content (%)
TGP-030	110	80%	-	0%, 10%, 25%, 37%, 50%, 75%
TGP-060	190	76% (78% [13, 18])	-	0%, 10%, 25%, 35%, 55%
TGP-090	280	78% (80% [19])	$8.9 \times 10^{-12}$ [19]	0%, 10%, 20%, 35%, 47%
TGP-120	370	78%	$8.7 \times 10^{-12}$ [20]	0%, 10%, 15%, 30%, 40%

described by Benziger et al. [11] In order to define GDLs' wettability characteristics, static droplet contact angles on GDLs of different thicknesses and different PTFE contents have also been measured.

### Contact Angle

Droplet static contact angle on GDL samples were measured using a setup specifically designed and made for this purpose [21]. Static contact angle is an indication of wetting ability of a solid surface by a liquid phase material. It also describes the mechanical equilibrium of the droplet under the influence of three phase interfacial tensions [22]. Measured droplet contact angles on GDLs with different amount of PTFE are shown in Figure 2 with error bars representing the standard deviation calculated for different measurements. Figure 2 shows that adding some amount of PTFE to an untreated GDL significantly increases the contact angle while GDLs with different amount of PTFE show almost similar contact angles. Similar observation was reported by some other groups [5, 11, 23]. Similar contact angles for GDLs with different amount of PTFE may be justified by PTFE agglomeration within the GDL rather than sitting on its surface. Fishman and Bazylak [24] measured through-plane porosity distribution of GDL carbon papers and observed decreased local porosity within the GDL and near the surfaces. They attributed this trend to higher PTFE concentration through the GDL and near the surface and lower PTFE concentration in the core region of it. In another study, Rofaiel et al. [25] introduced a novel technique for measuring heterogenous through-plane PTFE distribution within the GDL. They used scanning electron microscopy energy dispersive X-ray spectrometry and analyzed treated GDLs for fluorine signature. Fluorine is the PTFE's high concentration element and also does not exist in carbon fibers of GDL.

Figure 2 also shows that GDL thickness does not affect the droplet contact angle. Contact angles shown in Figure 2 can be divided into two different measurements depending on being treated or not. The contact angles measured are either *low*, that is characteristic property of untreated GDLs, or *high*, belonging to treated GDLs, no matter the PTFE content. For each measurement, the GDL thickness is observed to have no impact on the contact angle measured. This can be justified by the results reported by Whitesides and Laibinis [26]. They reported

**FIGURE 2:** Droplet contact angle on GDL samples

that droplet behavior on a solid surface is mostly governed by top few monolayers of the surface of the GDL.

### Breakthrough Pressure

During the operation of a PEFC, hydrogen is oxidized in the anode and oxygen is reduced in the cathode. Oxygen reduction reaction is accompanied by water production. The liquid water accumulated in cathode side can pass through the GDL only when its pressure exceeds the capillary pressure [27]. For a continuous flow of liquid water through the GDL, liquid water pressure should also remain higher than the capillary pressure [27]. Capillary pressure  $P_c$ , defined as the pressure difference acting on both sides of a curved surface separating two phases of fluid ( $P_L - P_G$ ), is a function of surface tension  $\sigma$  and mean curvature  $H$  of the interface [28]:

$$P_c = 2\sigma H = \sigma \nabla \cdot \vec{n} \quad (1)$$

where  $\vec{n}$  is the unit normal to the surface. Equation 1 can be written as:

$$P_c = \frac{2\sigma_{\text{water}} \cos \theta}{r_{\text{pore}}} \quad (2)$$

As the electrochemical reaction continues, more water is produced in the cathode side. This increases liquid water pressure in cathode and can be a driving force for liquid water penetration into the GDL. Figure 3 shows the breakthrough pressure measured for untreated and 10 wt.% treated GDLs. It can be observed that the breakthrough pressure increases with GDL thickness. Same observation was reported by Tamayol and Bahrami [13] for TGP\_060, TGP\_090, and TGP\_120 Toray carbon papers. Based on such results, they modeled GDL as a multi-layer network of pores connected by throats, and argued that fluid (both liquid and gas phase) are stored in pores and the volume made by throats are negligible. Only throats resist water transport within the GDL and pores do not cause any resistance to the water transport within the GDL [28]. Based on their model, the breakthrough pressure increases with GDL thickness. Gostick et al. [18] studied the relationship between the capillary pressure and liquid saturation for GDLs with different thicknesses and observed that thinner GDLs show higher saturation than thicker ones at a given pressure. This means that intruding liquid water into thinner GDLs occur at lower pressure compared to thicker GDLs. They attributed such behavior to the finite size effect, more amount of pore volume being accessible in a thin GDL compared to a thick one.

Furthermore, it can be noticed from Figure 3 that the breakthrough pressure for PTFE treated GDLs are higher than that for untreated ones. Fairweather et al. [23] measured water-air capillary pressure based on the GDL saturation and observed that adding 5 wt.% of PTFE to the GDL significantly reduces water wetting but adding any more amount of PTFE (from 10 wt.% to 40wt.%) to the GDL hardly affects GDLs' wetting characteristics. Based on liquid-air capillary pressure curves obtained, they noticed that it was easier to intrude water into an untreated GDL rather than a treated one. Similarly, removing liquid water out of treated GDL samples was found to be easier than intruding into GDLs. In other words, higher liquid water pressure is required to intrude water into a treated GDL compared to an untreated one. One possible justification for higher breakthrough pressure for PTFE treated GDLs compared to untreated GDLs can be the higher contact angles measured for treated GDLs (Figure 2) since it increases the capillary pressure (based on Eqn. 1 and Eqn. 2) liquid water need to exceed to be able to penetrate.

### Effect of PTFE content

Breakthrough pressure for untreated GDLs and GDLs with different amount of PTFE content was studied and the result is shown in Figure 4. The error bars shown on the Figure represent the standard deviation calculated for different measurements. It can be noticed that 10 wt.% treated GDLs show significantly higher breakthrough pressures compared to untreated ones while adding more amount of PTFE hardly affects the breakthrough pressure. Same observation was reported by Benziger et al. [11]

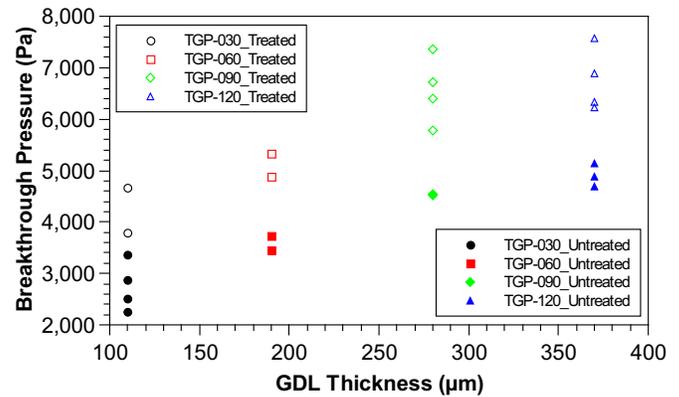


FIGURE 3: Breakthrough pressure measured for untreated and 10 wt.% treated GDLs

for Toray TGP-120 carbon paper.

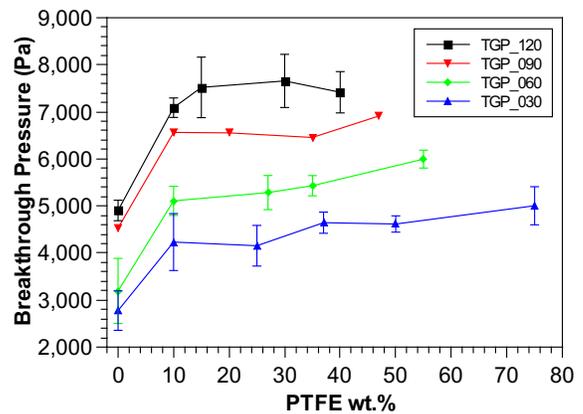


FIGURE 4: The effect of PTFE content on breakthrough pressure

Pressure variation curves from the beginning of water injection to liquid water breakthrough and droplet emergence is shown in Figure 5. Figure 5 shows similar trend in pressure increase for different GDLs over the time. As the Figure shows, the liquid water pressure increases linearly until it reaches to a peak value. The maximum pressure for each curve represents liquid water thoroughly breaking through the GDL and forming a droplet on the surface of the GDL. After the droplet emerged on the surface of the GDL, the pressure sharply decreases. Liu and Pan [29] measured liquid water pressure over time for different injection rate and observed that the slope of such plot differs based on the water injection rate. In current study, however,

water was injected only at  $500\mu l h^{-1}$ . Also, Figure 5 shows a *false peak* of pressure as it increases over the time. For untreated TGP\_030 GDL for instance, this *false peak* was observed to be around 30s. This *false peak* was observed in all of the runs done on the thinnest GDL (TGP\_030) and in most of the runs while using other GDLs. The reason of such minor pressure drop before the ultimate pressure drop (corresponding to the liquid water breakthrough) is not clear to the authors yet. However, it may be an indication of initial penetration of liquid water into the GDL that temporarily decreases the liquid water pressure.

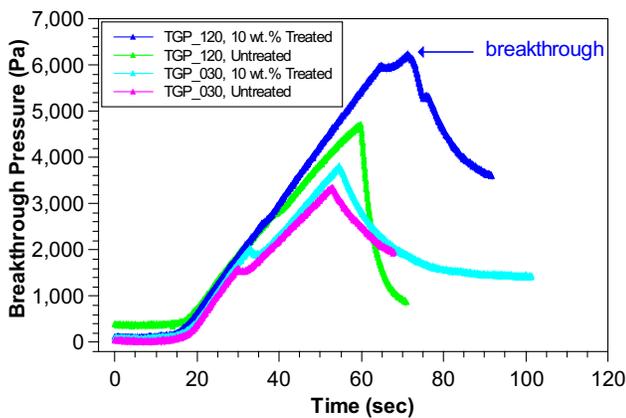


FIGURE 5: Breakthrough pressure profiles for different GDLs

Each pressure curve in Figure 5 can be divided into two regions; one from the point pressure starts increasing ( $\sim 17s$ ) up to *false peak* and one from *false peak* up to breakthrough pressure. The slope of all four curves are identical in the first part while pressure increases with slightly higher slope in second part for treated GDLs. Considering an almost identical overall slope (the whole increasing pressure part) for all four curves, it can be noticed that the GDL thickness is the dominant parameter affecting the breakthrough pressure. This result supports the GDL model introduced by Tamayol [13] as breakthrough pressure increases with GDL thickness.

Breakthrough pressure for Toray carbon paper TGP\_120 had been measured by Benziger et al. [11]. Tamayol and Bahrami [13] also measured breakthrough pressure of Toray carbon paper TGP\_060. The results obtained in this study is compared with their results and is shown in Figure 6. It can be noticed from Figure 6 that the present results are in well agreement with those reported elsewhere.

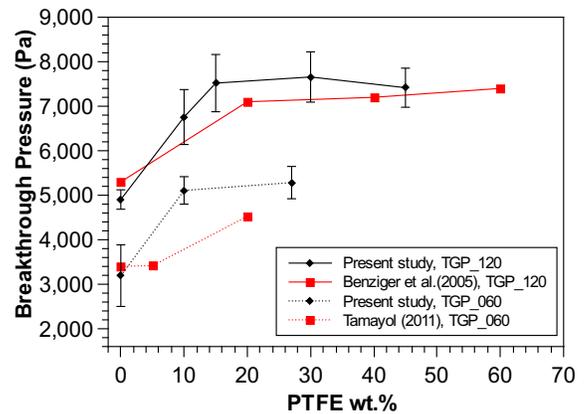


FIGURE 6: Comparing breakthrough pressure measured with results reported by Benziger [11] and Tamayol [13]

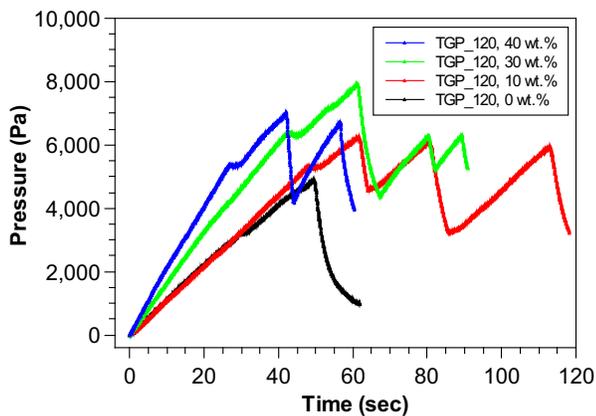
### Multiple Droplet Breakthrough

Pressure variation for subsequent droplets emerging on the surface of the GDL is shown in Figure 7. For the purpose of easier comparison of plots, pressure curves were plotted from the point pressure started increasing. The sawtooth pattern of the curves represent multiple droplets emerging from the surface of the GDL. It can be observed that the breakthrough pressure is slightly higher for the first droplet emerging and it decreases for consequent droplets emerging for each GDL case. The pressure curve for untreated GDL shows only one droplet emergence.

Figure 7 also shows that pressure increases at higher slope as the PTFE content within the GDL increases. The slope of pressure curve for an untreated and 10 wt.% treated GDL is almost identical but the difference becomes more significant for higher PTFE content curves.

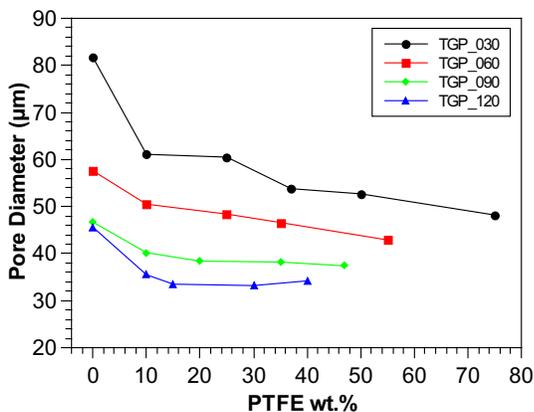
### Pore Diameter Estimation

Benziger et al. [11] considered a simple model to describe GDL porous structure. Studying water transport through different types of GDLs, they described GDL as a single layer of porous media with microchannels of different diameters connecting catalyst layer to the gas channel [11]. Considering this model, maximum GDL pore diameter can be estimated using the breakthrough pressure results obtained along with the contact angle values measured. The calculated results are shown in Table 2. The mean value of measured pressure for different runs are considered for this calculation. Same results are graphically presented in Figure 8 that shows the variation of calculated maximum pore diameter as a function of PTFE content for each GDL thickness. Following Benziger's model [11] and based on the results presented in Table 2 and Figure 8, it can be concluded that adding some amount of PTFE to an untreated GDL signifi-



**FIGURE 7:** Pressure profiles for TGP-120 with different PTFE content

icantly decreases the maximum pore diameter while any more PTFE addition to the GDL hardly affects the maximum pore diameter within the GDL.



**FIGURE 8:** Maximum pore diameter calculated based on breakthrough pressure results

## CONCLUSION

Liquid water breakthrough pressure on Toray carbon papers with different thicknesses and different PTFE contents has been experimentally measured. Injecting fresh liquid water to the surface of GDL samples at one constant injection rate, liquid water

pressure variations were monitored until breakthrough occurred. The experiments were run three times for each GDL case and very reproducible results were obtained. The following conclusions can be drawn from this study:

1. It was observed that droplet contact angle does not depend on the GDL thickness.
2. Although droplet contact angle was observed to increase from an untreated GDL to a PTFE treated GDL, the amount of PTFE content within the GDL was observed to have no effect on droplet contact angle measured.
3. It was observed that liquid water breakthrough pressure increases with GDL thickness ranging from  $\sim 2200$ Pa for untreated TGP\_030 to  $\sim 5000$ Pa for untreated TGP\_120.
4. Liquid water breakthrough pressure was observed to increase by applying some amount of PTFE to an untreated GDL. Applying some amount of PTFE to an untreated GDL increases the droplet contact angle and capillary pressure liquid water needs to exceed to be able to pass through the GDL.
5. Although applying some amount of PTFE to an untreated GDL significantly increased the liquid water breakthrough pressure, it was observed that breakthrough pressure does not change significantly with different amount of PTFE content within the GDL.
6. It was observed that liquid pressure increases linearly with almost identical slope for GDLs of different thicknesses and similar treatment level.
7. It was observed that the pressure slope increased with PTFE content within the GDL.
8. Comparing the breakthrough pressure of multiple droplet emerging from the same location on the GDL, it was observed that the first emerging droplet showed highest breakthrough pressure and the breakthrough pressure decreased for future droplets emerging from the location.

## ACKNOWLEDGMENT

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**TABLE 2:** Pore diameter calculation based on breakthrough pressure measured

Toray Carbon Paper Type	GDL Thickness ( $\mu\text{m}$ )	PTFE Content (%)	Mean Breakthrough Pressure (Pa)	Contact Angle ( $^\circ$ )	Maximum Pore Diameter ( $\mu\text{m}$ )
TGP-030	110	0%	2776	142 $^\circ$	81.7
TGP-030	110	10%	4232	153.9 $^\circ$	61.1
TGP-030	110	25%	4154	150.9 $^\circ$	60.5
TGP-030	110	37%	4644	150.2 $^\circ$	53.8
TGP-030	110	50%	4616	147.8 $^\circ$	52.7
TGP-030	110	75%	5004	NA (considered as 147 $^\circ$ )	48.2
TGP-060	190	0%	3193	129.7 $^\circ$	57.6
TGP-060	190	10%	5108	153.7 $^\circ$	50.5
TGP-060	190	25%	5284	152.8 $^\circ$	48.4
TGP-060	190	35%	5430	151.7 $^\circ$	46.6
TGP-060	190	55%	5995	153.5 $^\circ$	42.9
TGP-090	280	0%	4530	137.4 $^\circ$	46.7
TGP-090	280	10%	6563	156.4 $^\circ$	40.2
TGP-090	280	20%	6553	NA (considered as 151 $^\circ$ )	38.4
TGP-090	280	35%	6455	149 $^\circ$	38.2
TGP-090	280	47%	6913	NA (considered as 154 $^\circ$ )	37.4
TGP-120	370	0%	4904	141.3 $^\circ$	45.6
TGP-120	370	10%	7088	151.2 $^\circ$	35.5
TGP-120	370	15%	7519	151.4 $^\circ$	33.5
TGP-120	370	30%	7657	151.9 $^\circ$	33.2
TGP-120	370	40%	7416	NA (considered as 152 $^\circ$ )	34.2

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