Droplet and Percolation Network Interactions in a Fuel Cell Gas Diffusion Layer

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The transport of liquids through porous media is a crucial phenomenon in many fields, such as geology, chemistry and energy technologies.\(^1\)–\(^3\) One specific application used in energy conversion is the polymer electrolyte fuel cell (PEFC) in which this transport process plays an essential role. During the electrochemical reaction of hydrogen and oxygen, water is generated in the cathode catalyst layer. The water needs to be removed continuously to allow access of the reactant gas to the catalytically active sites and sustain the electrochemical reaction. A porous material, termed a gas diffusion layer (GDL), is used to transport the product water from the catalyst layer to the gas channels in the flow field plate (FFP), where the water is carried with the reactant gas stream towards the cell outlet. The porous GDL materials are commonly papers, felts or woven materials made of carbon fibers with diameters in the range of 7 to 10 \(\mu\)m and contain varying amounts of binder and hydrophobic coating to optimize their water management capabilities.\(^4\)–\(^6\)

There are two main mechanisms of water transport through the GDL from the catalyst layer towards the flow field plates; (i) vapor phase transport\(^7\) via diffusion and convection towards the flow field plates, where the water vapor is removed by the gas stream, and (ii) liquid phase transport\(^8\)–\(^9\) driven by the formation of a percolation network within the GDL. If the percolation networks grow too large, resulting in a high liquid water saturation of the GDL, gas transport in the remaining free pore space is not sufficient to sustain high rates of the electrochemical reaction. This phenomenon is commonly referred to as flooding. The main factors determining the interaction of the GDL with the water cluster are the pore morphology and the contact angle between the water and fibers forming the pore surface. While the pore morphology is a direct result of the fiber arrangement, hydrophobic coatings are applied to increase the water contact angle, thereby confining the water clusters to keep the GDL liquid saturation low and maintain continuity of the gas phase, especially at high current density operation. Understanding and managing the liquid water clusters inside the GDL is required to improve PEFC technology.

However, studying the water percolation networks is not trivial, as many methods of investigation are inapplicable in the confines of a fuel cell. As such, \textit{ex situ} experiments, isolating the GDL domain and emulating the fuel cell environment have proven to be a very effective method of investigation.\(^10\)–\(^12\) Studies investigating the injection of water into fuel cell GDLs, showed that establishing the percolation network as well as consecutive droplet formation and detachment events exhibit a characteristic pressure evolution.\(^13\)–\(^16\) During formation of the percolation network, the pressure of the water phase (\(p_w\)) increases while the water spreads until it emerges on the GDL surface (breakthrough). After emerging, a decline in water pressure is observed as the droplet forms on the surface and increases in size up to the point when the droplet is detached by the gas flow in the channel. Following the detachment, the water pressure increases again as a new droplet has to form resulting in a periodic pressure fluctuation. While the effect of this pressure evolution on the droplet can be studied with visual instrumentation, the response and behavior of the percolation network is largely obstructed by the GDL and as such little is known regarding the behavior of the percolation network within the GDL during droplet release.

To investigate the liquid water in GDLs of PEFCs, many research groups have successfully utilized X-ray tomographic microscopy (XTM) and X-ray radiography on a variety of time and length scales.\(^16\)–\(^23\) With these methods, 2D and 3D information about the internal structure of the GDL and the water distribution in its pores can be obtained. While many projections have to be recorded during XTM, inherently lowering the temporal resolution compared to radiography, developments in fast XTM of PEFCs with scan times of about 1 s\(^2\)\(^4\)–\(^25\) make it possible for dynamic effects to be investigated in 3D with tomography.

Using a combination of XTM and X-ray radiography, we aim to obtain a more complete picture of the droplet formation and detachment cycle and its impact on the percolation network inside the GDL. The 3D information of the water cluster that can be deduced from XTM in the range of seconds linked to information about the dynamics of droplet detachment in the gas channel recorded at a higher temporal resolution via X-ray radiography, providing an unprecedented level of insight. As pressure equalization in the water phase is the main way of interaction between the percolation network and the droplet, the pressure inside the water phase is determined through interfacial curvature analysis of the segmented XTM data.

**Experimental**

An \textit{ex situ} liquid injection cell was used to investigate the behavior of the percolating water in the GDL, droplet evolution and
detachment in the gas channel and their interaction (Fig. 1a). It consisted of a GDL sandwiched between a flow field plate, emulating a channel section of a fuel cell cathode bipolar plate, and a water injector. The injector (PEEK) featured a pathway, through which demineralized water was supplied to the bottom of the GDL while it simultaneously served as a stable mount for the other components. To prevent the injected water from spreading uncontrollably between the injector field and the GDL, a 50 μm thick hydrophobic gasket with a circular, laser cut hole (Ø 500 μm) was placed between them. The hole in the gasket was centered under the gas channel to increase the likelihood for the generated percolation network to evolve droplets in the gas channel without them getting in contact with the channel walls. A single rectangular gas channel with a depth of 300 μm and a width of 800 μm (Fig. 1b) was supplied with dry nitrogen at 25 °C to establish a constant gas channel flow with an average velocity of 15 m s⁻¹. A GDL sample from Sigracet (SGL 24 BA) with 5 wt% PTFE was used in this study. It was determined to have an average porosity of 0.75 (based on XTM segmentation) which can not consider binder porosity compared to 0.74. Advancing and receding contact angle values of 159° and 119° respectively, were measured using the sessile droplet method on a GDL sample of the same type. It was positioned between the injector and flow channel and compressed from its original thickness of 190 μm to 180 μm in the gas channel and 150 μm under the ribs. For the water injection a 5 ml Hamilton syringe in a syringe pump (KD Scientific 110) was used, feeding water at a constant injection rate of 350 nl min⁻¹ to the injector. Using Faraday’s law, this can be determined to be equivalent to a current density of 32 A cm⁻² relative to the injection area (0.196 mm²). This high rate of injection was necessary to ensure the formation of droplets in the presence of evaporation into the dry nitrogen gas stream in the channel.

An analysis of the capillary number

\[ Ca = \frac{v \mu_n}{\gamma} \]  

with the liquid velocity \( v \), the viscosity of the non-wetting phase \( \mu_n \) and the gas-water interfacial tension \( \gamma \) yielded a capillary number of \( \log_{10} Ca = -4.85 \) or lower which is still well within the regime of capillary fingering and does not result in a loss of generality in the results.

To verify that the vertical orientation of the gas channel does not influence the behavior of the water structures, the Bond number (\( Bo \)) was calculated for the GDL and the gas channel domain:

\[ Bo = \frac{\Delta \rho g R^2}{\gamma} \]  

where \( g \) represents the gravitational constant, \( R \) the radius of the water cluster, \( \Delta \rho \) the density difference between nitrogen and water and \( \gamma \) the gas-water interfacial tension. The Bond numbers for the largest possible spherical water cluster in the GDL and the gas channel domain are given in Table I.

Even when assuming water droplets large enough to fill the respective domains (\( R_{\text{Max}} \)), the values for the Bond number are far below 1, indicating that the effect of interfacial tension largely outweighs the impact of gravity on the droplet shape and behavior. This confirms the viability of this experiment in a vertical arrangement without any loss of generality. For completeness, the droplet radius required to have a balance of the effects of interfacial tension and gravity is 2750 μm.

**Image acquisition and processing.**—X-ray imaging was performed at the TOMCAT beamline of the Swiss Light Source (SLS) with a GigaFRoST high speed camera in combination with a microscope from Elya Solutions. Before the tomographic microscopy, radiograms were recorded for 20 s with a frame time of 10 ms to gather detailed dynamics of the droplet formation and detachment. During the acquisition of these radiograms, the GDL was oriented relative to the imaging cell comprised of injector and flow field plate (FFP) with a single gas channel (GC). (b) Cuts along and through the channel, indicating the positioning of the hydrophobic gasket and the GDL as well as the channel geometry. (c) Schematic depictions of the changing state of the waterfront with changing water pressure before and after droplet formation in a cross-section of the cell (top) and between two fibers (bottom).
perpendicular to the beam, resulting in an image showing a top down view of the droplet in the channel.

The radiograms were segmented using a combination of median filtering for denoising and a manually selected global threshold. An automatic fitting was used to extract the projected 2D area of the droplet, $A_{2D}$ from every frame. The circularity $\omega$ of the segmented droplet shape was computed as a measure of droplet deformation for each frame using the 2D perimeter $P_{2D}$:

$$\omega = \frac{4\pi A_{2D}}{P_{2D}^2} \quad [3]$$

It was assumed that for small droplets that don't reach far into the channel domain, the shape is not dominated by the gas flowing in the channel and is thus close to spherical ($\omega \sim 1$). Under this assumption, the volume of water contained in the droplet $V_{3D}$ was calculated by using the radius $R_{3D}$ of a circle with equivalent area to $A_{2D}$:

$$V_{3D} = \frac{4}{3}\pi R_{3D}^3 = \frac{4}{3}\pi \left( \frac{A_{2D}}{\pi} \right)^{\frac{3}{2}} \quad [4]$$

The XTM imaging of the water containing GDL was performed with an exposure time of 3 ms for each of the 251 projections resulting in a scan time of 0.75 s for a full tomogram similar to Eller et al.24 This comparably fast scan procedure was used to image the water during the continuous injection to obtain a set of 10 XTM images. After each XTM, a 1.8 s period was needed for back rotation of the rotation stage of the beamline to be ready to acquire the next scan resulting in a scanning period of 2.55 s. A slower, higher quality scan was employed to obtain the dry structure. The dry structure tomogram was obtained after the injection experiment was performed to reduce the impact of radiation dose. A summary of all relevant parameters for both radiography and tomography can be found in Table II below.

The image processing and segmentation (Fig. 2) was performed using an in house methodology implemented in ImageJ consisting of image subtraction, thresholding and morphological operations (see flow chart representation in Fig. 2). As a first step, the high quality, dry, data (Fig. 2b) was aligned to Cartesian coordinates and the water containing data (Fig. 2a) registered to it. Next, the dry data was subtracted from the water containing data after median filtering to obtain a difference image, enhancing the water signal (Fig. 2c).

Figure 2. Schematic representation of the imaging pipeline with the water containing data (a), dry data (b), difference image of water and dry data (c), segmented water mask (d), the segmented dry mask of fiber and binder (e) and the combined segmented image (f).
denotes the observed water-solid contact angle measured by analysis.

Therefore channel droplets were excluded from the curvature analysis.

Smeared gray scale (Figs. 2a and 2c). This made it difficult to show motion artefacts because expansion during the scan lead to a growth rate. The solid line exemplary indicates the water injection rate into the setup. Two frames show radiographic images of the droplet 1 s and 2.5 s after breakthrough.

The difference image was then segmented using median filtering for denoising and thresholding steps combined with binary morphological operations, resulting in a binary mask for the water inside the cell (Fig. 2d). For the dry structure segmentation (Fig. 2c), a combination of median filtering for denoising, thresholding and binary morphological operations was used. This was necessary because the SGL GDL features a microporous binder material that exhibits strong grayscale variations in the tomographic image, resulting in unsatisfactory results when using a simple global thresholding. The porosity of the binder could not be resolved after breakthrough.

Visualization of the water and GDL structure segmentations obtained from the XTM scans taken during the continuous injection, arranged to show the droplet formation. (a) Scan 1 (0 s), (b) Scan 6 (12.75 s), (c) Scan 4 (7.65 s) and (d) Scan 7 (15.3 s).

Table II. Imaging parameters used for the fast XTM scans during water injection and the slow scan used to obtain a high quality reference image of the dry structure as well as radiographic imaging.

| Scan Mode       | Exposure time [ms] | Projections/Frames [-] | Beam energy [keV] | Scan time [s] | Voxel/Pixel edge length [μm] |
|-----------------|--------------------|------------------------|-------------------|--------------|-----------------------------|
| Fast (injection) | 3                  | 251                    | 13.5              | 0.75         | 3 (3D)                      |
| Slow (dry)      | 12                 | 2001                   | 13.5              | 24           | 3 (3D)                      |
| Radiography     | 10                 | 2000                   | 13.5              | 20           | 3 (2D)                      |

Figure 3. Droplet volume evolution over time (dashed line) relative to the start of growth of a selected droplet. (I) water free GDL surface, (II) droplet growth and (III) detachment. (blue) linear growth region used to fit the growth rate. The solid line exemplary indicates the water injection rate into the setup. Two frames show radiographic images of the droplet 1 s and 2.5 s after breakthrough.

The segmented data of the droplet in the channel was used in collaboration with Martin Andersson et al., comparing droplet growth on the GDL surface modeled with a volume of fluid simulation to the droplet growth obtained from XTM imaging, ignoring any processes in the GDL.

**Liquid pressure in porous media.**—The pressure difference $\Delta p$ across the gas-water interface of a water cluster in a porous media can be described using the Young–Laplace equation:

$$\Delta p = \gamma \left( \frac{1}{R_1} + \frac{1}{R_2} \right)$$

where $R_1$ and $R_2$ are the principal radii of curvature. The gas-water interfacial curvature of the percolation water cluster was analyzed to estimate the pressure drop across the interface. This was done using a method of 3D surface identification, smoothing and characterization previously applied to multiphase flow problems in rocks by Lin et al., which determines the principal radii ($R_1$ and $R_2$) describing the surface curvature. Thereby, the voxelated liquid-void interface area is represented by a surface mesh that interpolates between the voxels to achieve a volume preserving surface smoothing.

Under the simplifying assumption of cylindrical pores, an alternative expression of the Young–Laplace equation can be used, linking the diameter of an idealized pore and the surface wettability to the pressure drop across the gas-water interface.

$$\Delta p = \frac{2\gamma \cos \theta}{R_{pc}}$$

Here, $\theta$ denotes the observed water-solid contact angle measured through the liquid phase, and $R_{pc}$ is the radius of the cylindrical pore surrounding the meniscus. From this equation it can be seen that interfaces in larger pores result in a lower pressure drop while more hydrophobic surfaces cause a higher pressure drop across the interface.

**Results and Discussion**

The X-ray radiography observation of droplet formation and detachment revealed three repeating states: (I) no droplet visible on the GDL surface, (II) growth of the droplet, and (III) droplet detachment. On average, the time between two consecutive droplet detachments was determined to be 4.3 s for the chosen combination of liquid injection rate and channel gas flow speed (350 nl min$^{-1}$ and 15 m s$^{-1}$ respectively), with no droplet visible on average for 0.7 s and droplet growth for 3.6 s. For different gas velocities in the gas channels it can be expected, that the droplet detachment diameter would decrease with increasing gas velocity$^{32-34}$ and increasing...
Fig. 5. (a) The water volume, determined in 3 μm thick slices at different heights of the GDL and channel for three representative states of droplet formation. (b) Renderings of two representative states before and after droplet formation with a cutout revealing the internal water distribution. (c) Water volume distribution in the GDL domain averaged for the four cases before droplet formation and the four cases after droplet formation with a large droplet. (d) Saturation at different heights in the GDL for a cylindrical region of interest of diameter 500 μm centered above the injection port and for the full 750 by 750 μm² GDL area shown in (b) and Fig. 4.
The interaction between the water phase and the gas channel (h). These images provide additional insights into intervals starting near the injection location (d) and ending close to extracted at different heights from the injection location in 30 μm intervals with (d) located close to the injection point and (h) near the gas channel as indicated by the outlines in (a–c).

Figure 6. Visualizations of a superposition of the two states, before and after droplet formation (red and blue). When both states were occupying the same voxel, priority was given to the state after droplet formation (blue). (a) The full GDL structure in the region of interest. (b) Here, only fiber voxels near the water cluster are shown. (c) The superposition of the water clusters. (d)–(h) In-plane slices extracted at 30 μm intervals with (d) located close to the injection point and (h) near the gas channel.

Visualizations of a superposition of the two states, before and after droplet formation (red and blue). When both states were occupying the same voxel, priority was given to the state after droplet formation (blue). (a) The full GDL structure in the region of interest. (b) Here, only fiber voxels near the water cluster are shown. (c) The superposition of the water clusters. (d)–(h) In-plane slices extracted at 30 μm intervals with (d) located close to the injection point and (h) near the gas channel.

amounts to a total of 0.3 nl (11000 voxels) between the two states corresponding to a 2% change relative to the total volume of the percolation network in the GDL before droplet formation (15 nl). The water distribution is in terms of GDL saturation is shown in Fig. 5d for two different analyzed GDL volumes. A strong dependency of the saturation values on the analyzed GDL volume can be found, since the percolation clusters stems from a very localized liquid injection into the GDL. The saturation stays constant for the lower 40 μm for the cylindrical as well as the rectangular GDL domain, which is due to GDL porosity fluctuations throughout the thickness of the GDL.

For a qualitative visual comparison between the two states of the percolation network before and after droplet formation, the data sets closest to the respective average water distributions are shown superimposed in Fig. 6. As before, red indicates the water cluster before droplet formation and blue after droplet formation. If voxels were water filled in both states, priority was given to the state after droplet formation (blue). With this labeling method, red features indicate an inflation of the percolation network before breakthrough and droplet formation.

While Fig. 6a shows the full GDL domain in the rendered subvolume, in Fig. 6b only the fibers touching and thus shaping the percolation network are visible. This highlights the difference between the two states in the pore throats of the GDL structure and it can be seen that most menisci in these throats show an inflation before droplet formation. Apart from the large deformation in the feed path at the breakthrough location, it is visible that especially the large throats show a significant volumetric change between the two states. This is to be expected as from equation 6 it can be seen that the pressure drop across a meniscus in a throat is inversely proportional to the throat radius. This means that the air-water interfaces spanning large pore throats experience a higher degree of deformation compared to the ones in small throats when exposed to the same pressure fluctuation. For completion, Fig. 6c shows just the superposition of the two states without any fibers.

The image series Figs. 6d–6h shows single in-plane slices, extracted at different heights from the injection location in 30 μm intervals starting near the injection location (d) and ending close to the gas channel (h). These images provide additional insights into the interaction between the water phase and the fiber structure in the GDL and verify the dominant deformation of the water cluster in the pore connecting the main body of the percolating network with the breakthrough location (center of (e) and (f)).

The observed deformations of the water percolation cluster are in good accordance with the characteristic pressure fluctuations reported for repeated breakthrough events such as droplet formation and detachment.13,14 Right before the breakthrough event, the pressure in the water structure is reported to peak, this is equivalent to the state before droplet formation, at which point we see an inflated percolation network. While after the breakthrough event, the pressure was found to drop, in the present study this correlates to the state after droplet formation where the percolation network contracts.

To determine the extent of this pressure fluctuation in the present case, two segmented data sets, one before and one after droplet formation, were analyzed for their principal radii on a discretized meshing of the gas-water interface. The analysis was limited to the interfaces in the GDL domain, excluding the droplet in the channel domain. Figure 7 shows the extracted interface curvature before and after droplet formation. The peak positions of the almost overlapping curvature distributions have been determined using curve fitting and correspond to a pressure drop Δp (using Eq. 5) across the interface of 24 mbar (2400 Pa) before and 22 mbar (2200 Pa) after droplet formation. This is in agreement with the observed behavior of the percolation network described above and also fits well to the range of reported breakthrough pressures for SGL 24 BA (~22 mbar).18 The interfacial curvature distribution showed a wide spread, which may be associated to the presence of small interfacial sections of the single water cluster being analyzed and the voxel size of 3 μm limiting the accuracy to which the interface curvature and the precise shape of the gas-solid-liquid interface can be determined. Furthermore, the segmentation of the noisy XTM data may have resulted in assignment errors of the phases at the gas-solid-liquid interface. The influence of such assignment errors on the curvature distribution is evaluated by imposing a distance requirement D and only including data points above this distance from the three-phase contact line. This excludes regions near the three-phase contact where segmentation errors are largest17 and the interactions of the fibers and the menisci cannot be resolved with the given resolution.
On the right side of Fig. 7a, the effect of varying distance requirements on the peak pressure drop is shown up to a distance of 5 μm. By increasing the distance over which values are ignored near the contact lines, the curvature distributions separate more clearly. A visual representation of the curvature distribution and the consequence of the distance requirement is shown in Fig. 7b. The black regions show the gas-water interfaces below a distance of D = 3 μm. For this distance requirement, the estimated pressure drop before droplet formation increases to 28 mbar, whereas the pressure drop after droplet formation decreases to 17 mbar with decreasing trend. Note that the number of valid surfaces evaluated drops significantly, as we exclude points near the contacts, with only ~7% of the total interface area having a distance from the interface larger than 5 μm. Hence, the determination of precise pressure values remains difficult. On the other hand, the fact, that for all distances the obtained values for Δp are larger before than after droplet formation indicates the robustness and potential of curvature based pressure drop determination in XTM data.

The influence of different GDL materials on the experimental findings is not trivial to anticipate. For similar droplet formation and detachment cycles there will be a change in difference pressure Δp before and after droplet formation. For smaller pores, higher pressure fluctuations and correspondingly more negative curvature values before droplet formation can be expected, but the water volume fluctuations might be smaller due to smaller menisci cross-sections.

Conclusions

A combination of X-ray radiography and X-ray tomographic microscopy was used to investigate the behavior of a percolating
water cluster during consecutive droplet formation cycles on the GDL surface in the course of liquid water injection into the GDL.

X-ray radiography allowed precise insight in the timing of the droplet release formation and detachment cycle with a period of about 4.3 s from which droplet grow rates were estimated. The X-ray tomographic microscopy scans captured different stages of the droplet growth and the corresponding percolating water cluster states within the GDL with a scan time of 0.75 s and a repetition rate of 2.55 s.

Periodic drainage of the percolation cluster in the top third of the GDL thickness after droplet detachment was observed, while the break-through location remained stable for the observation time. In the bottom 2/3 of the GDL thickness the percolation cluster showed much lower fluctuations of only 0.3 nl (2% of the total water volume contained in the GDL). The minor water volume change in the bulk GDL was clearly identified for the first time as gas-liquid menisci movement in the pore throats as predicted by theory.

It was further demonstrated how the determination of the gas-water interfacial curvature can be used to estimate the pressure drop across the gas-liquid interface of the percolation network from XTM images capturing the trend of pressure fluctuation that matches well with previously published values. This method holds great potential for future 4D imaging studies, especially thanks to recent beamline instrumentation upgrades, as it allows for the determination of water pressures during operando PEFC XTM imaging.

We are confident that future 4D XTM studies with such advanced analysis approaches will improve the understanding of transient two-phase water transport processes in PEFCs, provide crucial validation data for 3D PEFC microstructure models and ultimately enable improved GDL material design for increased PEFC efficiency.

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