The water balance of polymer membrane fuel cells is of critical significance for the proper functioning of cells. ZSW has comprehensive expertise and extensive experimental and simulation methodology at its disposal for research and further development in this area. The experimental methodologies are presented here. The corresponding simulation methods are discussed in a separate document (see download box).

// INVESTIGATIONS INVOLVING X-RAYS
(SYNCHROTRON OR µ-CT)

The water balance of gas diffusion layers (GDL) can be investigated during operation (in situ) using X-rays. Depending on the specific issue being studied, such investigations are carried out either using a microcomputer tomograph (µ-CT) at ZSW or a synchrotron system in cooperation with partners at Helmholtz-Zentrum Berlin (HZB). Both radiographic and tomographic methods may be used here, again depending on the issue being investigated.

Radiographic examinations

Using specific measurement cells, integral water distributions – including their time dependency – can be determined using synchrotron radiography. In this way, the behaviour of a membrane-electrode-GDL unit can be characterized as a function of component selection and chosen operating conditions.
The capabilities of these investigation techniques can be summarised as follows:

- In-situ investigation possible for specific measurement cells
- Differentiate i.e. between microporous layer (MPL) and GDL (substrate)
- Smallest water clusters detectable in the nanolitre range
- Very high spatial and time resolution (0.9 µm per pixel at 3 seconds per image)

Tomographic investigations

Using specific measurement cells, spatially resolved water distributions can be determined for e.g. a GDL unit based on synchrotron or µ-CT tomography and, in this way, the local and integral water saturation can be determined as a function of component selection and chosen operating conditions. In addition, the individual pressure dependence of the water saturation of a GDL can be determined (see fig. below).
**Ex-situ investigations of the water balance (Leverett function)**

Using specially developed apparatus, the permeation properties of water into a GDL can be measured as a function of pressure and thus the capillary pressure - saturation relation (Leverett function) to the specific GDL can thus be identified. This function can be used e. g. to facilitate a representation of GDL properties that is as accurate as possible in CFD cell modelling.

![Schematic experimental set-up for measuring the functional dependency of water saturation on water pressure in comparison to a standard approach](image)

**// CONTACT ANGLE MEASUREMENTS**

A distinction should be made between the outer or surface contact angle and the inner contact angle in the context of the wetting characteristics of a porous material. The latter determines the properties of water inside the material and is dependent on the chemical make-up of the surface. In contrast, the surface contact angle is also determined by the structural properties of the surface. Surface structuring on the µm scale often results in increased hydrophobic properties (lotus effect), meaning that the contact angles enclosed based on the shape of a drop on the surface for GDL materials are often considerably larger than would be expected based on the chemical properties of the material surface (inner contact angle).

**Drop contour**

A drop contour measurement can be used to determine the outer contact angle of a fuel cell component. Furthermore, an inclination function of the associated apparatus (goniometer) allows the roll-off angle of the material to be determined too. This information is used both directly for assessing the suitability of fuel cell components in specific areas of application and as input parameters for modelling of fuel cells additionally. Such measurements can also be performed within the channels of bipolar plates.
ZSW characterisation of cell components: Gas diffusion layers and microporous structures

Liquid sorption measurements

Liquid sorption measurements can be used to determine the inner contact angle of porous materials. This involves bringing the material into contact with a wetting fluid and determining the contact angle over time from the weight increase resulting from sorption of the fluid. For non-wetting fluids – e.g. water on most GDL materials – this property can be determined using measurements with several wetting fluids and extrapolation to the fluid of interest. A corresponding device (tensiometer) and suitable fluids are available at ZSW.

Inverse gas chromatography (iGC)

For a liquid sorption measurement, the uptake of the liquid depends both, on the chemical properties of the surface and the structure of the material. More specifically, the capillary pressure that determines fluid absorption is dependent both on the contact angle of the sample material and the diameter of the cavity (structural dependency).

As a purely gas-phase method, "inverse" gas chromatography (iGC) eliminates this structural dependency. This method determines the interaction of specific chemicals with the surface by measuring the time these remain on the surface in a carrier gas stream directed over the sample. This allows the components of surface energy and, as a result, the contact angle to water to be calculated. The term "inverse gas chromatography", however, originates from the fact that in this case the sample constitutes the solid phase, in contrast with analytical gas chromatography. Such a device is routinely used at ZSW to determine inner wetting properties.
Hydro head

The hydrostatic pressure at which water starts to penetrate through the material is called “hydro head.” It can be determined as a potential comparative criterion for various gas diffusion layers. As in the cell, the microporous layer (MPL) constitutes the greatest transfer resistance to liquid water in this case too, meaning that this method in particular allows differences in relation to the largest pores in the MPL to be characterised in particular.

Bubble point

While the method used to determine the HydroHead considers the contact angle with the measurement fluid of water in addition to the material structure, the result of the related method of determining the so-called "bubble point" depends only on the material structure. This method attempts to force a fully wetted fluid out of the saturated sample under increasing gas pressure. The bubble point corresponds to the pressure at which gas seepage first occurs, which can be detected as bubbles forming at the surface. This is the point at which fluid is released from the largest passage pore in the material and the result is characteristic of its diameter.

Vapour sorption

The method of dynamic vapour sorption (DVS) can be used to determine the volume of water absorbed by a sample from the gas phase at a given temperature and relative humidity, and to measure how quickly water held in the sample is released. This measurement provides information on the drying characteristics of gas diffusion layers and on the properties of fuel cell membranes, for example.

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