

Towards PFAS-free high temperature proton exchange membrane fuel cell cathodes: Evaluating PEEK as an alternative to PTFE

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ABSTRACT

Polytetrafluoroethylene (PTFE) is used in various applications throughout the renewable energy sector: in wind turbine bearings, as back sheets for solar panels or as separators in batteries. In particular, in the catalyst layer (CL) of a high temperature proton exchange membrane (HT-PEM) fuel cell PTFE is dominant due to its hydrophobic properties while being chemically and thermally resistant to the working environment of the FC. In search for a suitable replacement for PTFE with respect to a possible per- and polyfluoroalkyl substances (PFAS) ban, polyether ether ketone (PEEK) based CLs are intensively studied in this work as a possible substitute for PTFE. PEEK is commercially available and a production would be scalable. It exhibits a high thermal and chemical resistance suitable for the HT-PEM environment. Loadings of 20, 30 and 40 wt. % PEEK were tested against PTFE loadings of 0, 10, 20 and 30 wt. %. The following measurements were used: Micro X-ray fluorescence analysis (μ -XRF), scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX), contact angle analysis, inductively coupled plasma mass spectrometry (ICP-MS), nano computed tomography (n-CT) and electrochemical measurements. Even though the hydrophobicity of the PEEK based CL is not comparable to PTFE based electrodes, the electrochemical half-cell investigation of the oxygen reduction reaction (ORR) showed promising results. PEEK based electrodes in general lead to a better performance compared to a CL without polymer or a non-optimal PTFE loading such as 10 wt. % PTFE. The electrode with 30 wt. % PEEK delivered a potential E at $-0.5 \text{ A mg}_{\text{Pt}}^{-1}$ only 8 % lower than the commercial PTFE based reference. The incorporation of PEEK into HT-PEM cathodes achieves this by compared to PTFE increased open porosity and decreased agglomeration the catalyst material.

1. Introduction

With the climate crisis at hand, the transition to energy supply without emission of greenhouse gases like CO_2 becomes urgent. But even though the energy sector reduces CO_2 emissions it is connected to environmental harm with pollutants such as per and polyfluorinated alkyl substances (PFAS). They are used for their exceptional properties like chemical inertness, surface lubrication, hydrophobicity and thermal stability [1]. Because of that, they are intensively used in applications like electronics, batteries and fuel cells [2]. But PFAS and the surfactants used for their production are also known to accumulate in plants and therefore in animals and the human body which causes multiple health issues like decreased liver function and cancer [3–5]. Due to the environmental concerns of PFAS, the European Union (EU) is stepping up efforts to restrict its use [6]. Even though a temporary exception might

be possible for sectors like renewable energy, the reduction or substitution of PFAS is already an intensively discussed topic [2].

The specific technology this work focusses on, is the high temperature proton exchange membrane fuel cell (HT-PEM-FC). As part of the fuel cell technology in general, it can contribute to a possible renewable energy infrastructure. The HT-PEM-FC operates at temperatures of 120 – 200 °C which leads to a higher CO tolerance and simpler heat management than the well-known low temperature PEM-FC [7]. Instead of the often PFAS containing ionomers in LT-PEM-FC in HT-PEM-FC phosphoric acid is used as proton conductor. The proton conducting and already PFAS free polybenzimidazole (PBI) based membrane is doped with it before assembly [8]. Concerning the PFAS topic, this is a major benefit for HT-PEM-FC because the membrane does not have to be replaced.

However, the electrode of a HT-PEM-FC typically consists of carbon

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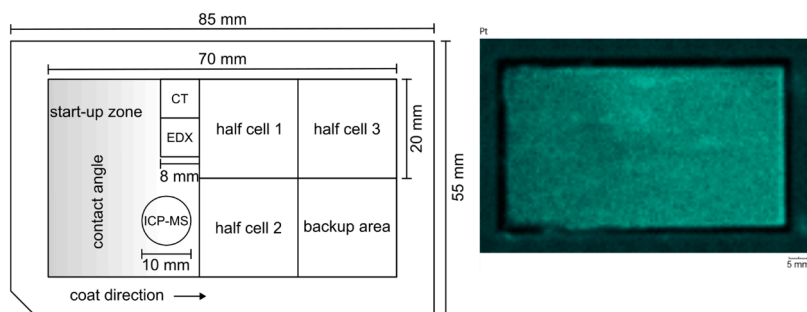


Fig. 1. Sampling plan after GDE preparation. The outer perimeter marks the size of the MPL/GDL and the inner perimeter shows the coated area. The right image is a mapping of platinum with μ -XRF.

supported platinum catalyst and the PFAS polytetrafluoroethylene (PTFE) [9]. The polymer's chemical inertness delivers great resistance against the harsh HT-PEM-FC environment, high hydrophobicity and acid retention due to its network structure in the pores of the CL [8].

Electrodes with alternatives to PTFE such as poly(2,3,5,6-tetrafluorostyrene-4-phosphonic acid) (PWN) were already tested and showed increased peak power density of about $11 \pm 12\%$ after conditioning when compared to PTFE based electrodes. The measurements were carried out in a single cell at 180°C [10]. PWN does not fit the exact definition of PFAS because the fluorine bound to an aromatic six membered ring instead of directly to the alkyl chain as demanded by the PFAS definition. But it still has a highly fluorinated backbone. For comparison, 76 wt. % of PTFE is fluorine and 41 wt. % of the best performing PWN consists of fluorine as well. The lack of the label PFAS does not determine a substance less harmful [11]. Fluoropolymers such as PWN exhibit similar properties to PTFE like the strong C-F-bond and the high temperature resistance [12]. The basic building blocks (pentafluorostyrene/pentafluorobenzene) are ecotoxicologically relevant and exhibit aquatic toxicity (cf. ECHA data for CAS 363–72–4).

This example shows, why the research into fluorine free substitutes is necessary. Polybenzimidazole (PBI) and polyurethane-polyethylene-co (acrylic acid) were tested and resulted in a 3 % lower Pt utilisation compared to PTFE based electrodes (5 and 20 wt. %) and when PTFE was added to the PBI containing electrodes the cell voltage at 0.3 A cm^{-2} increased by 20 mV [13]. This leaves the question whether a fluorine-free and commercially available polymer can replace PTFE in the HT-PEM electrode without sacrificing electrochemical performance.

In this work, polyether ether ketone (PEEK), which is a commercially available polymer, is tested as a potential substitute for PTFE in the CL of HT-PEM electrodes. PEEK provides a high thermal stability up to 575°C , a melting point of up to 340°C [14] and a resistance to phosphoric acid as well as most other acids [15] besides concentrated sulfuric acid, which would sulfonate the polymer quickly [16,17]. The viability of PEEK in HT-PEM cathodes is evaluated with electrochemical measurements in a gas diffusion electrode (GDE) half-cell in order to directly identify the effect of PEEK in the cathode and to directly compare PTFE-versus PEEK-based cathodes without any further impacts during a fuel cell operation. The following additional characterisations were performed: Micro X-ray fluorescence analysis (μ -XRF), scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX), contact angle analysis, inductively coupled plasma mass spectrometry (ICP-MS) and nano computed tomography (n-CT).

2. Experimental

2.1. Electrode preparation

GDEs were prepared by doctor blading the catalyst suspension at 30 mm s^{-1} with a Proceq ZUA 2000 universal applicator onto the microporous layer of the GDL (Freudenberg H23C2). The exact dimensions of the GDE and the very homogeneous coating are shown in

Table 1

Recipe for catalyst paste. The resulting quantities were multiplied by 3. The blade height was calculated based on the densities of the materials and the area to be coated.

Material	Quantity
PtNi (3:1) 40 wt. % on Vulcan XC-72	Target: 2.64 mg cm^{-2} ($0.85\text{ mg}_{\text{Pt}}\text{ cm}^{-2}$)
PTFE; PEEK	0, 10, 20, 30 wt. % PTFE; 20, 30, 40 wt. % PEEK
Ultra-pure water ($>18.2\text{ M}\Omega$)	60 wt. % of catalyst material
Isopropyl alcohol	adjusted to result in $\sim 13\text{ wt. \%}$ solids total

Fig. 1. As described in Table 1 the catalyst paste was prepared with a PtNi catalyst (Pt:Ni in 3:1 ratio, 40 wt. % metal on Vulcan XC-72 from FuelCellStore), PTFE (60 wt. % dispersion in water from Sigma Aldrich, $0.2\text{ }\mu\text{m}$ particle size) or PEEK (VESTAKEEP® 2000 UFP20 powder from Evonik, $20\text{ }\mu\text{m}$ particle size) at the desired loading (PTFE loading: 0, 10, 20 and 30 wt. %; PEEK loading: 20, 30 and 40 wt. %), pure water (Millipore, at least $18\text{ M}\Omega\text{cm}$) to prevent sparking and isopropyl alcohol ($\geq 99.5\%$ for synthesis, Carl Roth, Germany) in a 5 ml polypropylene tube. The suspension was mixed in a ball mill (Retsch MM400) at 30 Hz for 10 min to homogenise. The coating was done immediately after mixing with a blade height between 220 and $360\text{ }\mu\text{m}$ depending on the polymer loading and an area of $40\times 70\text{ mm}$. To evaporate isopropyl alcohol and water safely, the sample was put in a nitrogen stream for three hours. All samples were sintered at 350°C for 10 min in nitrogen. For the electrochemical measurements PBI based Celtec-P® membranes from BASF were hot-pressed onto the CL for 30 s at 140°C at a pressure of 1 kN. Metal shims with a thickness of about 80 % of the total height of the GDE and membrane were used to prevent the membrane from getting pressed too thin. Before use, the membrane was stored in 50 vol% H_3PO_4 (85 %, Merck suprapur, Germany) for at least 25 h to ensure a consistent acid doping level and reach swelling equilibrium.

2.2. X-ray fluorescence (μ -XRF) analysis

The Horiba XGT-9000 device was used for Pt mapping of the CL with the 1.2 mm capillary focusing the x-rays in helium environment. This was done to discern anomalies in the CL like spots with significantly higher amounts of platinum than the rest. Spots like this can occur for reasons like droplets of catalyst paste falling on the CL after coating or problems during the coating. Larger bright spots or dark spots on the mapping were avoided for further characterisation to ensure better reproducibility. If the μ -XRF mapping showed major inhomogeneities of platinum distribution across the coating, such electrodes were discarded.

After validating the coated GDE by μ -XRF, the samples for the different characterisation methods were cut out of the GDE like schematically shown in Fig. 1. The backup area was mostly used for electrochemical purposes because they were the most likely to result in failed measurements and they use up the most space. That means if one

Table 2

Experimental parameters of the n-CT measurements.

Parameter /unit	Value
Source Voltage /kV	70
Source Current / μ A	120
Random movement	40
Resolution / μ m px ⁻¹	1.75
Exposure time /ms	980 – 1300 to ensure similar contrast

of the areas dedicated to half-cell measurements is determined to be inhomogeneously coated via μ -XRF, the backup area can be used.

2.3. Scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDS)

The surface morphology and the PTFE loading of the CL were analysed with SEM and EDS. A GDE sample of 8 mm squared was attached to the aluminium sample holder with carbon tape. A Jeol JSM-IT800 SEM was operated with an acceleration voltage of 10 kV. For EDS three to four of $300 \times 400 \mu\text{m}$ areas were scanned for fluorine, carbon, oxygen, platinum and nickel detection. The fluorine amounts were used to confirm the expected PTFE loading in the CL. It is assumed that ~ 76 wt. % of PTFE consist of fluorine based on the monomer C_2F_4 .

2.4. Nano computed tomography

GDE samples were placed vertically in the Bruker SkyScan 2214 CMOS Edition n-CT. In Table 2 the experimental parameters of the CT-measurements are listed.

For determining the CL thickness, the CL was reconstructed with NRecon (Bruker). In DataViewer (Bruker) two lines were placed visually on the bottom and the top of the CL and the distance between them was measured. This was done three times with different cross-sections and the average was calculated. The two lines also served as limits for the open porosity analysis in CTAn (Bruker). Open porosity is the percentage of volume open to the environment without solids in a given volume of interest (VOI). The VOI varied between 0.1 and 0.8 mm^3 . The particle size distribution was measured with the same constraints. The images were made with CTVox (Bruker).

2.5. Contact angle analysis

Contact angles were measured with the OCA25 from dataphysics. GDEs were heated to 160°C to align with the HT-PEM conditions in the half-cell, and concentrated H_3PO_4 was used (Merck Emsure 85 vol.% H_3PO_4) drops of $9 \mu\text{l}$ were deposited onto the CL surface. A cannula with

0.6 mm inner diameter was used. The analysis was done using Young-Laplace fitting within the Dataphysics dpiMAX software.

2.6. Inductively coupled plasma mass spectrometry (ICP-MS)

To quantify the catalyst loading, a 12 mm circle was cut out of the GDE and put into a small beaker. 1.2 ml HNO_3 (Rotipuran® Supra 69 wt. %, Carl Roth, Germany) and 1.6 ml HCl (Rotipuran® Supra 30 %, Carl Roth, Germany) were added and sonicated for 5 min. The sample was kept at room temperature overnight to dissolve platinum. Afterwards, the remaining solid was filtered and the filtrate diluted with 2 wt. % HNO_3 until a volume of 250 ml is reached. Before measuring ICP-MS, the sample was acidified with HNO_3 and then diluted 1:20 with ultra-pure water, until 3 vol.% HNO_3 is reached. A $1000 \text{ mg Pt l}^{-1}$ ICP-MS standard solution from Carl Roth was used for calibration with the following concentrations: 1; 5; 10; 50; 100; 150; 200 $\mu\text{g l}^{-1}$. The platinum isotopes ^{194}Pt , ^{195}Pt and ^{196}Pt were detected. ^{175}Lu was used as internal standard (1 mg l^{-1}). A correlation coefficient had to be at least 0.999 for the calibration to be used.

2.7. GDE half-cell measurements

Electrochemical measurements were performed with a Gaskatel Flexcell®. A PTFE shield reduced the active geometric area to 0.785 cm^2 allowing higher current densities with the same current. A copper plate with the active area cut out backed the GDE and made it possible to electrically connect to it. The assembled half-cell can be seen in the supporting information in Figure S 1. A platinum coil served as counter electrode and a Hydroflex® from Gaskatel reversible hydrogen electrode (RHE) as reference electrode. The used electrolyte was concentrated phosphoric acid (Merck Emsure 85 vol.% H_3PO_4) at 160°C . Nickel dissolution was not tested because the influence on the comparison was deemed inconsequential.

The Ametek Modulab2100A potentiostat equipped with an external 12 V/20 A Ametek Booster was used for all measurements. The electrochemical protocol is based on the protocol reported by Ehelebe et al. [18] Table S 1 shows the first part of the electrochemical protocol performed under nitrogen flow, while Table S 2 shows the galvanostatic steps of the polarization curve done with pure oxygen flow. For the accelerated stress test (AST) 1000 cyclovoltammograms with 500 mV/s were performed in a potential range of 0.6 to 1.5 V. All electrochemical measurements were done three times with a different sample and averaged. The standard deviation was calculated with the following method:

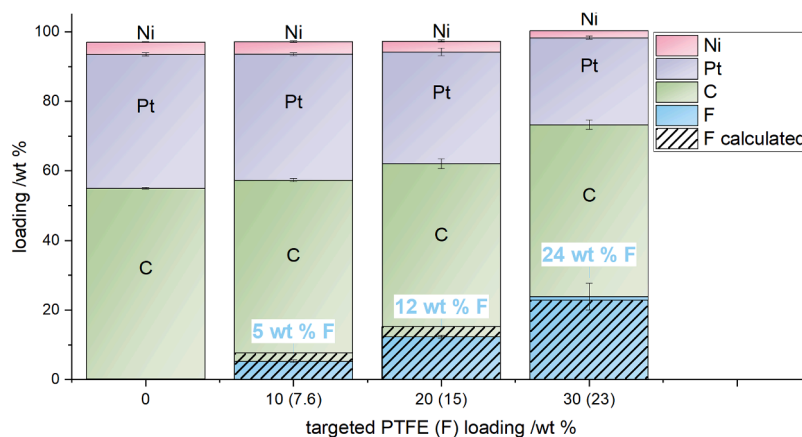


Fig. 2. EDS measurements of different PTFE loadings. The targeted PTFE loading is shown on the x-axis and the calculated F loading is in brackets for each loading. The measured loadings of F are shown for each column. The share of C, Pt and Ni are also shown.

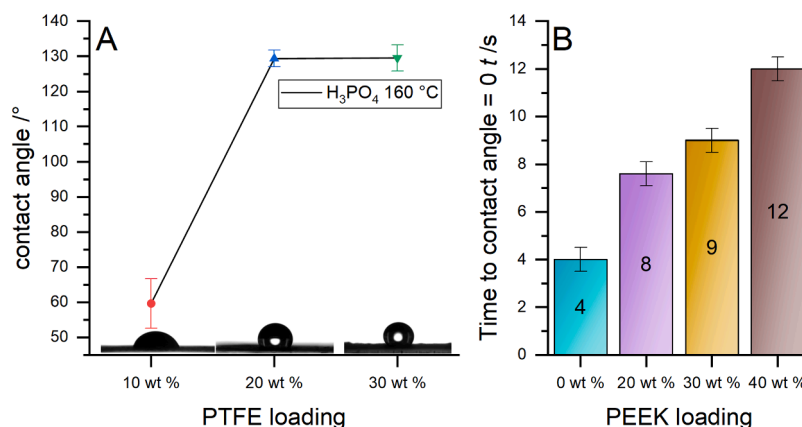


Fig. 3. Results of contact angle measurements with pictures of the H_3PO_4 drops on the electrode surface at different PTFE loadings in the left graph. Time until the H_3PO_4 drops on the electrode surface flatten completely at different PEEK loadings in the right graph.

$$\text{standard deviation} = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2}$$

3. Results

3.1. Catalyst layer composition

While the PTFE based CLs are tested in PTFE contents of 10, 20 and 30 wt. %, the PEEK based ones are tested in contents of 20, 30 and 40 wt. %. The necessity of a higher PEEK loading was assumed before the measurements to compensate for the lower hydrophobicity of the PEEK particles discovered in preliminary tests. After doing the electrochemical tests and the lowest PEEK loading of 20 wt. % was determined to be the worst, a 10 wt. % PEEK sample was deemed unnecessary.

The EDS measurements of the CLs of the PTFE based electrodes verify the targeted amounts of PTFE in the CL and the homogeneity in the CL. In Fig. 2 the detected fluorine contents are displayed at the bottom of each column and compared against the calculated fluorine amounts displayed as the overlaid slanted line columns. The differences between the detected and the calculated values are only small and can be attributed to the unstable nature of the PTFE dispersion used in the recipe. C and Pt are displayed as well to demonstrate that even though the amount of the catalyst material made of Pt, C and Ni decreases, the total C content increased because of the C in PTFE. The amount of PEEK in the CL cannot be reasonably measured, because the majority of PEEK is C and there is no other easily detectable element like F in PEEK. But there is no uncertainty about the amount of material transferred into the catalyst paste and the CL because powder was used instead of a dispersion.

3.2. Wetting

The contact angle analysis in Fig. 3 shows the wettability with concentrated H_3PO_4 recorded at 160 °C to reflect the conditions found in the HT-PEM environment. In Fig. 3A the data from the PTFE containing electrodes can be seen and in Fig. 3B the data from the PEEK containing electrodes are displayed. At the lowest PTFE concentration of 10 wt. % H_3PO_4 forms a contact angle of $60 \pm 7^\circ$. At 20 and 30 wt. % PTFE the contact angle then increases to $129 \pm 2^\circ$ and $130 \pm 4^\circ$, respectively. In contrast, the PEEK based electrodes are not hydrophobic enough to prevent the H_3PO_4 from draining into the CL. However, to still make the difference between the PEEK loadings visible the time for the drop to seep in completely is shown in Fig. 3B. Without any polymer present in the CL the drop takes four seconds to disappear completely, and with increasing PEEK content the drop takes longer to seep in. With 40 wt. % PEEK it takes 12 s for the drop to fully flatten. Overall, this clearly shows that the hydrophobicity of PEEK based electrodes is not comparable to the hydrophobicity of PTFE based electrodes. But still, the hydrophobicity increases with increasing PEEK amounts in the catalyst layer. This is expected because of the non-polar nature of the PEEK molecule providing increased hydrophobicity. Untreated PEEK by itself exhibits a water contact angle of 77.6 ± 0.3 [19].

3.3. Morphology of the GDE

To be able to compare the morphological effects of the polymers in the CL, computed tomography (CT) and scanning electron microscopy (SEM) were used to image the CLs in 2D and in 3D and on different scales. The CT imaging was furthermore used to determine open porosity and particle size distribution of the CL. For each PTFE and PEEK loading Fig. 4 shows the SEM image on the left and the CT image on the

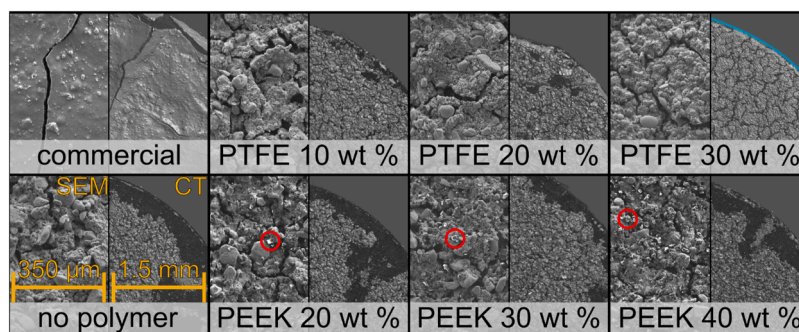


Fig. 4. SEM and CT images of the CLs at different loadings of PTFE or PEEK. SEM measured with Jeol JSM-IT800, $V_{\text{acc}} = 10$ kV. CT-images measured with Bruker SkyScan 2214 CMOS Edition. Red circles mark single PEEK particles and the blue line shows the cutting edge of the electrode.

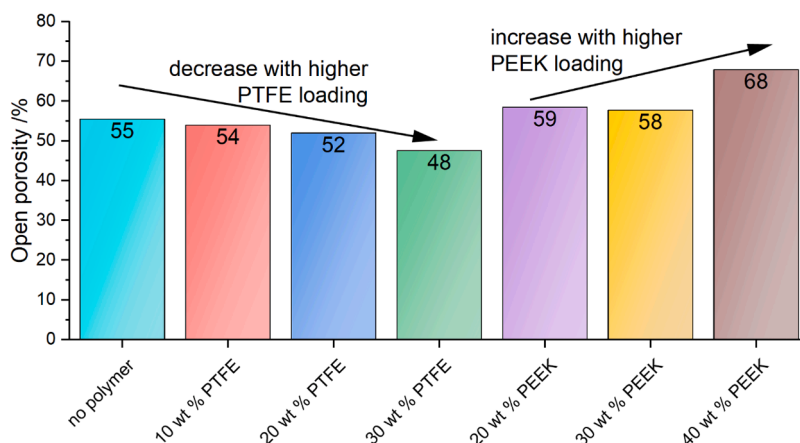


Fig. 5. Open porosity is the percentage of volume without solids in a given volume of interest (VOI) that is open to the environment. The VOI varied between 0.1 and 0.8 mm³.

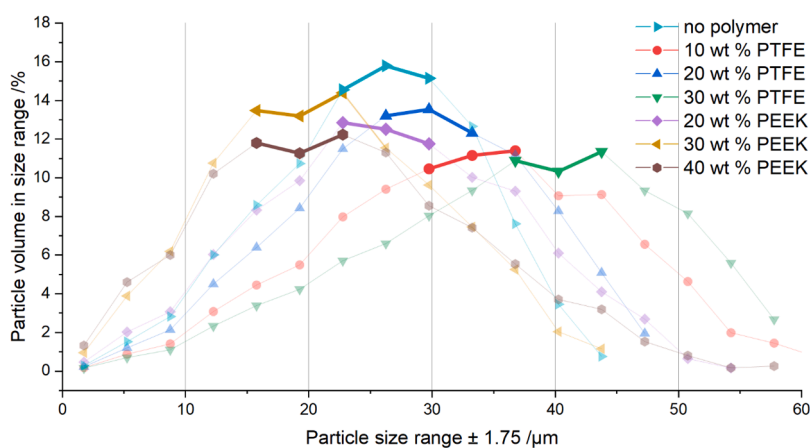


Fig. 6. In this particle size distribution the three particle ranges with the highest number of particles for each CL were used. The particle size distribution was calculated with the same data set as the open porosity.

right. The surface of the commercial electrode looks smoother compared to the other electrodes because of proprietary additives in the CL. CT imaging indicates that PTFE based CLs establish more distinct cracks the higher the PTFE content rises. The CLs with PEEK differ by the increasing amount of PEEK particles visible in the image. In each SEM image one PEEK particle is marked with a red circle, it was identified by shape, size and its tendency to show up brighter on the SEM image because of it being a nonconductive material. While the CL adheres better to the underlying MPL in the case of the PTFE based CLs when compared to the PEEK based ones, it also adheres better the higher the PTFE loading is. This is visible at the cut edges in the CT images shown in the CT image of the 30 wt. % sample by the blue line as an example. While the PTFE based CLs cover the MPL to the edge, the PEEK based CLs flake off easier. This indicated a worse mechanical stability of the CL. Cutting samples without polymer out of the initial large GDE results in the CL flaking off. Even the electrostatic force from the polymer petri dish lid was able to lift catalyst material up from the GDL to the lid. With PEEK this was not much of a problem. This indicates a stabilizing effect of the PEEK in the CL compared to using no polymer. The mechanical stability is important during the production of the GDE and the assembly of the membrane electrode assembly (MEA) [20].

To further characterise the morphology, the open porosity was compared between the different CLs which is displayed in Fig. 5. In general, the open porosity of the CL is lower with PTFE in it and higher with PEEK in it when compared to using no polymer resulting in an open porosity of 55 %. With an increasing PTFE loading, the open porosity

decreased from 54 % at 10 wt. % to 48 % at 30 wt. % PTFE. In contrast; PEEK increases the open porosity from 59 % at 20 wt. % up to 68 % with a loading of 40 wt. % PEEK. In this context the two polymers behave in opposite ways. While PEEK increases the open porosity of the CL, PTFE decreases it with higher loading. This is also supported by literature in which an increase of the PTFE loading from 0 to 40 wt. % leads to a decreased total pore volume by about 30 % [8]. The higher open porosity of the PEEK based CLs likely stems from the 20 μm large PEEK particles not fitting in the pores of the catalyst material. This is because even though its melting temperature of 340 °C is exceeded during sintering the PEEK particles hold their shape and do not seal the pores. Because of the voxel size of the CT measurement (1.75 μm), it is important to note, that this open porosity refers specifically to larger macropores. An optimised macro pore volume can reduce the molecular diffusion resistance and therefore benefit mass transport [21] and increase current densities [22] in studies of PEMFC CLs. The higher open porosity of the PEEK based electrodes might therefore be responsible for the measured electrochemical performance.

A look into the particle size distribution (PSD) of all particles present in the CL (Fig. 6) supports the findings of the open porosity. In the graph the PSDs of the different GDEs are displayed in whole, while the lines of the three highest particle size ranges are shown thicker. The distribution can be split in two overlapping ranges when it comes to the particle sizes most prevalent in each electrode: First, the PEEK based electrodes have their most abundant particle sizes in the range of 17 – 30 μm, and second, the PTFE based electrodes in the range of 26 – 44 μm. The electrode

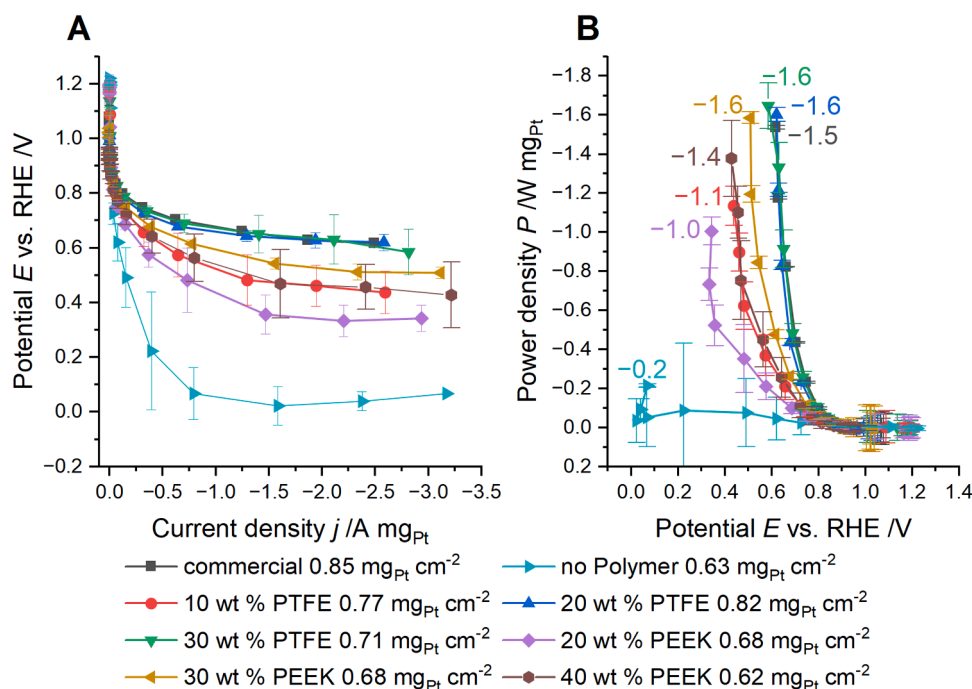


Fig. 7. A) Polarisation curves of PtNi based GDEs with varied amounts of PTFE or PEEK. B) Power density curves with the measured power density at 2 A cm⁻² written next to each curve. The measurements were conducted in a Gaskatel Flexcell GDE half-cell setup (160 °C, 1.5 L min⁻¹ oxygen, conc. H₃PO₄).

without polymer fits in the middle with a range of 23 – 30 μm for its most common particle sizes.

The shift to smaller particle sizes for the PEEK based electrodes can partially be explained by the 20 μm PEEK particles which make up a significant part of the CL. The initial PTFE particles have a much smaller average diameter of 220 nm but during sintering at 350 °C the evenly distributed PTFE nanoparticles melt into a much larger network structure linking the PTFE particles in the CL together [8]. As Fig. 6 shows, this leads to larger agglomerates, consisting of PTFE and the catalyst compared to the agglomeration of the catalyst particles among each other. This agglomeration could be the reason for the lower open porosity of the PTFE based electrodes compared to the PEEK based electrodes. And it explains the more mechanically stable CL the PTFE containing electrodes exhibit as previously shown in Section 3.3 and in literature [20]. The CL without polymer is in the overlapping area of PEEK and PTFE based CLs. This matches the trend observed in the open porosity analysis. Between the PTFE samples there is no clear trend visible. For PEEK a higher loading generally shifts the peak of the particle size range to lower particle sizes even though the 40 wt. % sample was expected to have a higher share of particles around the 20 μm particle size range compared to the 30 wt. % sample.

3.4. Electrochemical characterisation

The following electrochemical results show the ORR performance for all loadings of PTFE, PEEK, and no polymer in the CL, as determined by galvanostatic polarisation curves and concurrent EIS measurements. For these measurements, a phosphoric acid doped polybenzimidazole membrane was hot-pressed onto the CL and mounted in a commercial GDE half-cell like shown in the supporting information in Figure S 1. Concentrated H₃PO₄ was used as electrolyte and heated to 160 °C. The polarisation curves are displayed in Fig. 7A with the correlating power density curves in Fig. 7B. The graph of the polarisation curves shows the potential E plotted against the current density j and it compares the seven different polymer loadings against each other and a commercial HT-PEM cathode. Furthermore, due to slightly different Pt contents in the electrodes, the current is normalized to the Pt mass. In Figure S 3 the

Table 3

Electrochemical results of the ORR polarisation curves compared between the different polymer loadings and the commercial electrode.

Sample	Potential E @ - 0.5 A mg _{Pt} ⁻¹	Loss compared to commercial reference	Power density @ 2 A cm ⁻²
commercial	0.72 ± 0.004 V	0 ± 01 %	-1.54 ± 0.009 W mg _{Pt} ⁻¹
no polymer	0.18 ± 0.216 V	-75 ± 30 %	-0.22 ± 0.008 W mg _{Pt} ⁻¹
10 wt. % PTFE	0.61 ± 0.052 V	-15 ± 07 %	-1.14 ± 0.101 W mg _{Pt} ⁻¹
20 wt. % PTFE	0.70 ± 0.014 V	-3 ± 02 %	-1.60 ± 0.036 W mg _{Pt} ⁻¹
30 wt. % PTFE	0.71 ± 0.034 V	-1 ± 05 %	-1.65 ± 0.118 W mg _{Pt} ⁻¹
20 wt. % PEEK	0.54 ± 0.118 V	-25 ± 16 %	-1.01 ± 0.070 W mg _{Pt} ⁻¹
30 wt. % PEEK	0.66 ± 0.014 V	-8 ± 02 %	-1.59 ± 0.031 W mg _{Pt} ⁻¹
40 wt. % PEEK	0.62 ± 0.063 V	-14 ± 09 %	-1.38 ± 0.195 W mg _{Pt} ⁻¹

same graphs are plotted normalised to the area instead of the Pt mass. In Table 3 the potential E @ - 0.5 A mg_{Pt}⁻¹ is listed for all polarisation curves with the percentage loss compared to the commercial electrode. A current density of 0.5 A mg_{Pt}⁻¹ was chosen, because this is a reference point used in literature, in which the measurements were acquired in the same environment resulting in comparable data [23].

The commercial electrode shows the best performance with a potential of 0.72 ± 0.004 V at - 0.5 A mg_{Pt}⁻¹ and across the measured current density range, as well as the most reproducible results. Although the 30 wt. % PTFE sample can perform better according to the error bars, the 20 wt. % PTFE sample led to more reproducible results and will be used for comparison to PEEK based electrodes. Comparing the PEEK based electrodes, 30 wt. % is the best performing electrode. At - 0.5 A mg_{Pt}⁻¹ it delivers a potential of 0.66 ± 0.014 V, which is only 8 ± 2 % lower than the commercial electrode and 6 ± 2 % lower than 20 wt. % PTFE.

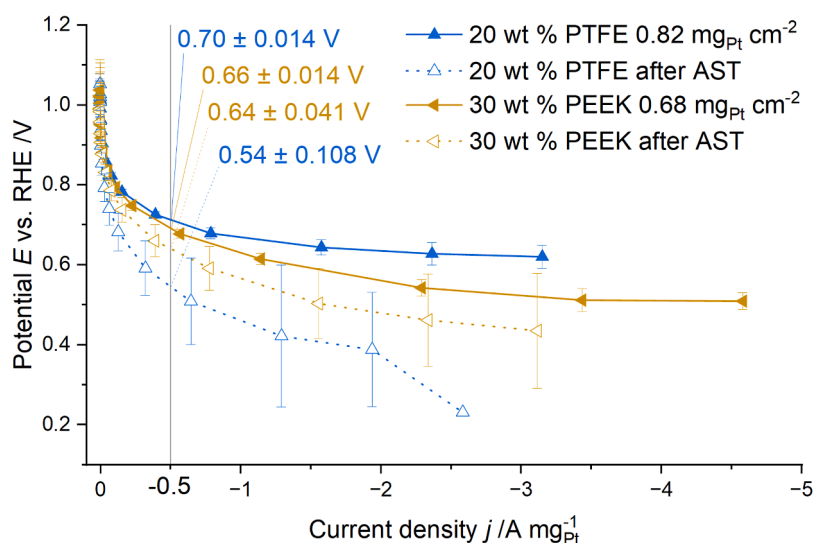


Fig. 8. Polarisation curves of PtNi based GDEs of the 20 wt. % PTFE and the 30 wt. % PEEK sample before and after the accelerated stress test. The potential at $-0.5 \text{ A mg}_{\text{Pt}}^{-1}$ for each of the curves is displayed. The measurements were conducted in a Gaskatel Flexcell GDE half-cell setup (160°C , 1.5 L min^{-1} oxygen, conc. H_3PO_4).

Considering the power density at 2 A cm^{-2} , 30 wt. % PEEK outperforms the commercial electrode with $-1.59 \pm 0.031 \text{ W mg}_{\text{Pt}}^{-1}$ compared to $-1.54 \pm 0.009 \text{ W mg}_{\text{Pt}}^{-1}$. While the suitable performance of PTFE based GDEs is based on their hydrophobicity leading to appropriate water removal, the good performance of PEEK based GDEs is attributed to the larger open porosity. This results into less agglomeration of the catalyst particles (Fig. 6), which likely improves the mass transport [24].

The electrode without polymer has a significant lower electrode potential and therefore a lower performance across the whole current density range. At $-0.5 \text{ A mg}_{\text{Pt}}^{-1}$ the loss of potential compared to the commercial electrode is $75 \pm 30 \%$. The measured power density at 2 A cm^{-2} is at $-0.22 \pm 0.008 \text{ W mg}_{\text{Pt}}^{-1}$ compared to $-1.54 \pm 0.009 \text{ W mg}_{\text{Pt}}^{-1}$ of the commercial electrode. The power density at -2 A cm^{-2} was used for comparison instead of the peak power density because the measurements were carried out to -2 A cm^{-2} and no peak was reached at that point. 10 wt. % PTFE leads to a lower potential across the current density range compared to 20 and 30 wt. % PTFE and the commercial sample. The potential at $-0.5 \text{ A mg}_{\text{Pt}}^{-1}$ is $15 \pm 7 \%$ lower than the potential of the commercial electrode. The polarisation curves of the different PEEK based electrodes exhibit a significantly performance across the current density range compared to not using any polymer.

In Fig. 8 the polarisation curves of the 20 wt.% PTFE and the 30 wt. % PEEK sample are shown before and after the AST. The AST does not accurately reflect real world durability but it can provide a first insight. The potential range of 0.6 to $\sim 1.0 \text{ V}$ degrades the platinum catalyst [25]. The potential range between ~ 1.1 and 1.5 V degrades the carbon support [26]. While the PEEK sample delivers less performance initially, after the AST the performance is better. The Potential of the 20 wt.% PTFE at $-0.5 \text{ A mg}_{\text{Pt}}^{-1}$ decreases from $0.70 \pm 0.014 \text{ V}$ to $0.54 \pm 0.108 \text{ V}$ by 23 % and the 30 wt. % PEEK decreases from $0.66 \pm 0.014 \text{ V}$ to $0.64 \pm 0.014 \text{ V}$ by 4 %. This indicates a possible better long-term performance for the PEEK based electrodes which is likely the result of a better thermal stability of the PEEK particles.

4. Conclusion

PEEK was tested as a replacement polymer for PTFE in the HT-PEM catalyst layer to find a suitable substitute in the light of possible PFAS bans from the EU. Although PEEK in the catalyst layer offers lower mechanical stability and hydrophobicity than PTFE, its electrochemical performance is highly comparable.

It is only $8 \pm 2 \%$ worse than the commercial electrode regarding the

potential at $-0.5 \text{ A mg}_{\text{Pt}}^{-1}$ and even exceeds it in the power density at -2 A cm^{-2} . This is attributed to the increased open porosity and less agglomeration of the catalyst particles because unlike PTFE, PEEK does not melt into a network during sintering due to its high thermal stability. This might both benefit the mass transport to the active sites according to literature mentioned in Section 3.3 thus increasing performance. After an AST the 30 wt. % PEEK electrode performs better than the 20 wt. % PTFE electrode. This indicates an improved real world durability of PEEK based electrodes.

These results strongly indicate that morphology and porosity of the HT-PEM electrode are important parameters for the electrochemical performance comparable in importance to a high hydrophobicity of the CL.

This work widens the spectrum of viable polymers for HT-PEM fuel cells significantly. Thus, the impact of a PFAS ban would be less problematic. While the results are positive, further research needs to be done to test smaller PEEK particle for a possible performance improvement and other polymers need to be tested as well. For future work, the PEEK based electrodes should be scaled to single cell and stack development.

CRediT authorship contribution statement

Arne Schechterle: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Software, Visualization, Writing – original draft, Writing – review & editing. **Dana Schonvogel:** Conceptualization, Supervision, Writing – review & editing. **Henrike Niehoff:** Writing – review & editing. **Steffen Rehse:** Formal analysis, Investigation, Writing – review & editing. **Michael Wark:** Conceptualization, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.electacta.2026.150042](https://doi.org/10.1016/j.electacta.2026.150042).

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