Contribution ID: **68** Type: **Oral contribution**

In situ study of ion-exchange membranes for electrochemical devices by ambient pressure hard X-ray photoelectron spectroscopy

Thursday, 8 December 2022 11:10 (20 minutes)

Ion-exchange membranes (IEMs) are a critical component of electrochemical conversion and storage devices, ranging from electrolyzers to batteries, where the IEM not only separates different media, but also ensures the selective transfer of specific ions from one medium to another in order to sustain the electrochemical processes of interest. The ion selectivity and conductivity, and the mechanical/chemical stability are key properties of the IEM, directly influencing the device performance and lifetime. These properties are often interdependent, and their optimization for the targeted reaction environment is a crucial challenge in the development of IEMs, and hence of efficient devices. A rational optimization requires a molecular level understanding of the processes occurring in the membrane, particularly during device operation. In this talk, we will describe an in situ ambient pressure hard X-ray photoelectron spectroscopy (AP-HAXPES) investigation performed at the SpAnTeX end-station (BESSY II),[1] of selected commercial CEM and AEM to characterize their chemical properties close to the vapor pressure of water at room temperature and under operating conditions. First, we characterized the Nafion® 115 (N115) and the Fumasep® PEEK-reinforced FAA-3-PK-75 (FAA-3-75) membranes under hydrated conditions by means of AP-HAXPES. Second, we used a customized hybrid gas/liquid phase electrolyzer to investigate the aforementioned membranes under working conditions. In a typical experiment, a membrane was placed between a compartment containing a NaCl aqueous solution (1 M) and a Pt electrode on one side, and an Au electrode and the analysis chamber on the other side. This configuration enabled the polarization of the membranes by applying a potential difference between both electrodes. We observed the unambiguous sodium and chlorine fingerprints for N115, and FAA-3-75, respectively. Interestingly, these fingerprints were independent of the applied potential –whether negative or positive –and were observable already at equilibrium conditions, i.e. OCP. Therefore, the ion out-diffusion is likely predominantly mediated by water diffusion and not by the electric field. Furthermore, although a polarization is still effective upon potential switch as indicated by the core level binding energy shift, the respective amount of out-diffused species does not change significantly, thereby pointing to the irreversible accumulation of ions at the membrane surface. Our work illustrates the potential of AP-HAXPES as a tool to investigate the chemical properties of IEMs in electrochemical configurations and under operating conditions. Our findings suggest that commercial membranes do not exhibit optimal ionic selectivity, which in turns plays a crucial role in the electrochemical performance and lifetime of the applied device.
[1] M. Favaro et al., Surf. Sci. 2021, 713, 121903

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Track Classification: Materials science