Measuring Intrinsic Membrane Permeability Using FTIR

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Abstract

Polymer electrolyte membranes (PEM) are an important component of artificial photosynthesis devices. Membrane permeability determines the transport of solutes between electrodes, which affects device efficiency. In order to measure membrane permeability, an *in situ* FTIR probe was used to measure the transport of methanol through a Nafion 1100 PEM. In order to collect accurate data, a baseline was defined. A 1-point baseline was subject to significant instrument drift, but a 2-point baseline improved the stability of absorbance measurements. Frequent collection of background spectra further suppressed residual instrument drift.

Introduction

In solar fuels devices, polymer electrolyte membranes promote crossover of electrolyte ions while limiting the crossover of CO_2 reduction products. Limiting the crossover of CO_2 reduction products is essential since it will improve device efficiency. To measure methanol transport across the membrane, an *in situ* FTIR probe was employed in a standard diffusion cell.

Team

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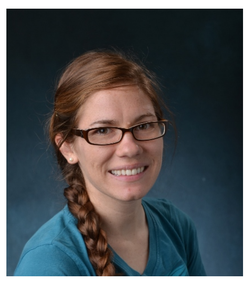


Sarah Dischinger Daniel Miller

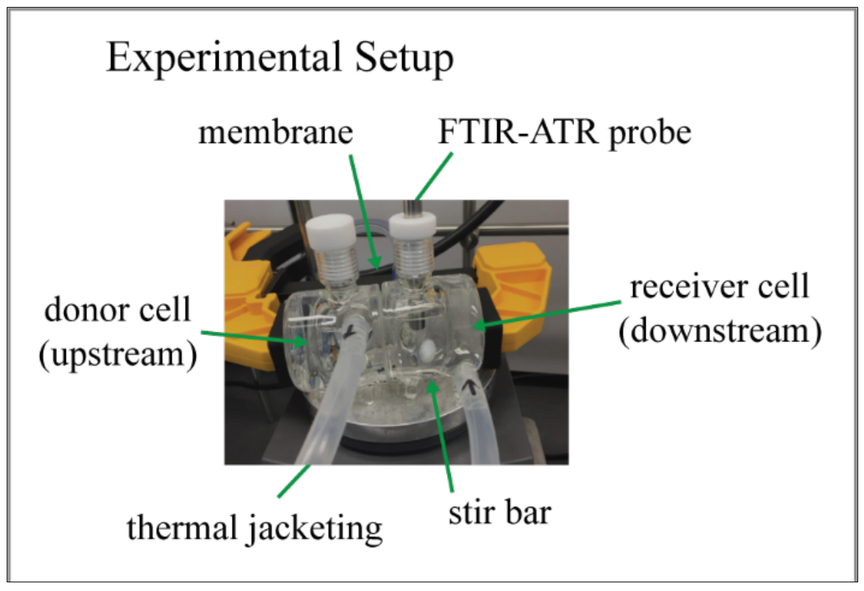
Conclusions

A 2-point baseline correction and frequent background collection yielded improved accuracy of methanol concentration measurements by negating instrument drift during long methanol permeation experiments with Nafion 1100 membranes. Highly accurate measurements of solute transport in PEM will enable determination of intrinsic membrane properties, facilitating fundamental study of membrane structure-property

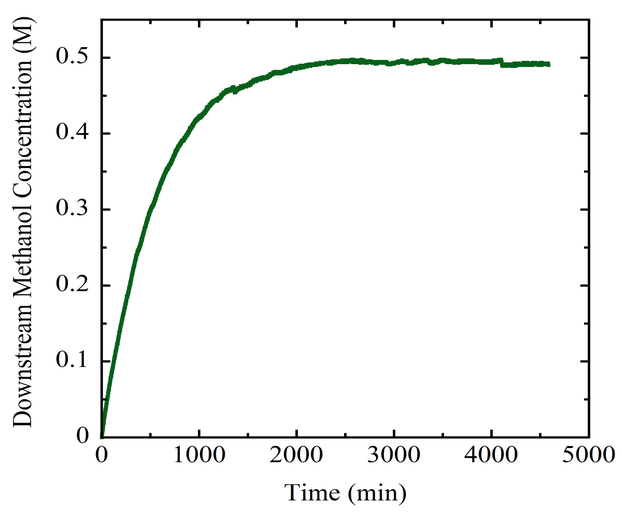
Acknowledgments

This material is based upon work performed by the Joint Center for Artificial Photosynthesis, a DOE Energy Innovation Hub, supported through the Office of Science of the U.S. Department of Energy under Award Number DE-SC0004993

Results

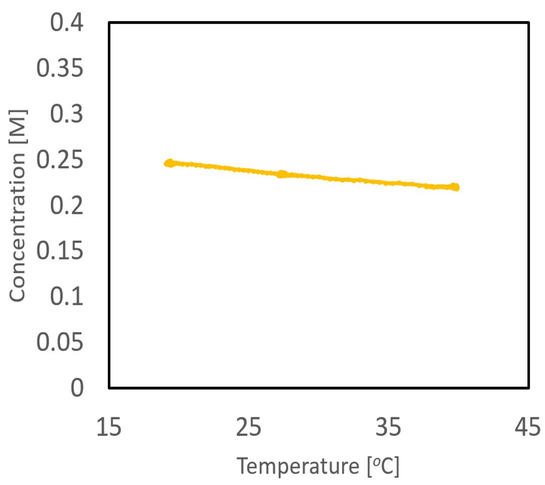


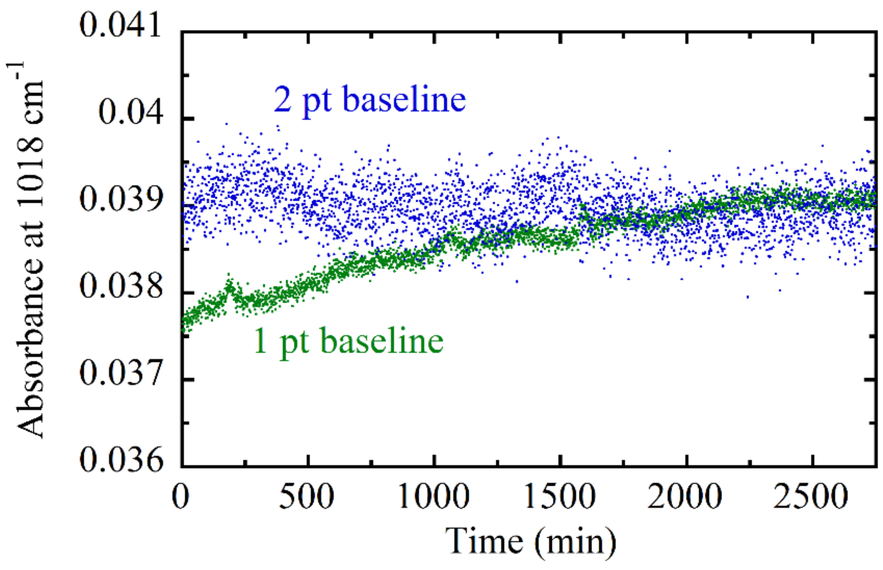
An *in situ* ATR-FTIR probe placed in the receiver cell was used to monitor the transport of methanol through PEM. The donor cell was initially charged with a 1 M methanol solution and the receiver cell was initially charged with ultrapure water. Methanol diffused through the membrane due to the concentration difference between the donor cell and the receiver cell. The concentration of methanol in the receiver cell was calculated from the solution absorbance and the calibration curve.



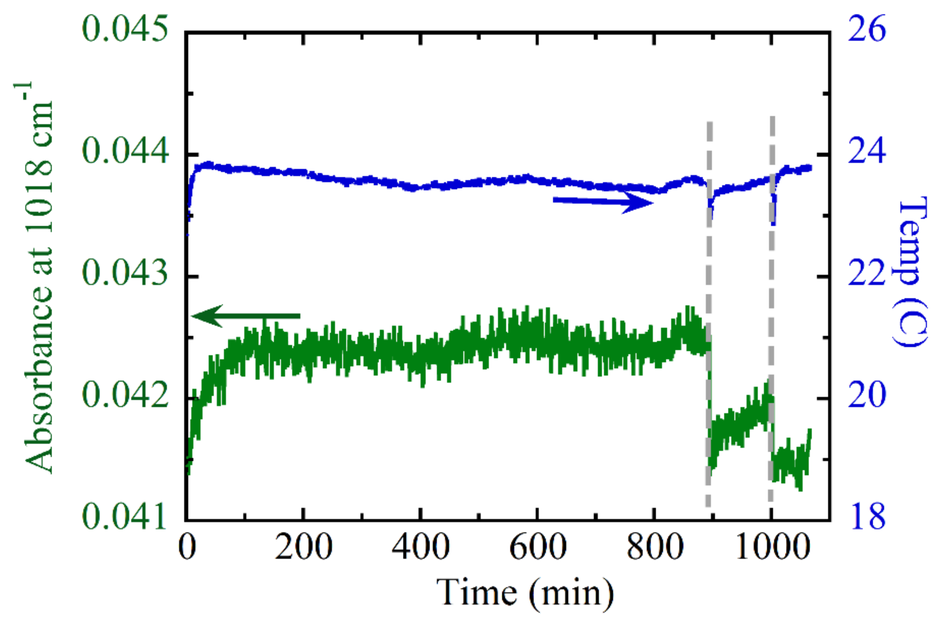
An example concentration profile collected from the receiver cell (above) is used to calculate the intrinsic membrane permeability. However, it is important to evaluate the accuracy of this raw data before using it for further calculations.

One potential source of inaccuracy is the variation of sample temperature during the experiment. By converting the observed signal height to an effective methanol concentration for a single solution held at various temperatures, it was observed that a 20°C variation in sample temp. corresponded to a 0.5M difference in concentration. Thus, variations in temp. on the order of 1°C, as is observed during experiment, were determined to be negligible.





To understand the variability inherent to the ATR-FTIR instrument, signal drift was measured as a function of time (above). The ATR-FTIR probe tip was submerged in a sealed container of DI water, and the peak height at 1018 cm^{-1} was recorded over 48 h was recorded. Instrument drift was compared using a 1-point baseline (absorbance fixed to zero at 1200 cm^{-1}) and a 2-point baseline (absorbance fixed to zero along the line defined by 1243 and 1187 cm^{-1}). The increase in absorbance with time for the green trace is indicative of instrument drift that is not properly accounted for using a 1-point baseline. When absorbance was normalized relative to a 2-point baseline (blue trace), the signal noise increased but instrument drift was minimal.



While the use of a 2-point baseline substantially improved absorbance measurements, a fresh background was collected often to further suppress instrumental drift. The figure above shows the impact of collecting fresh background spectra (at times indicated by the gray dashed lines) when the probe is submerged in sealed container of DI water. While the peak height at 1018 cm^{-1} generally increased due to instrument drift, collection of a fresh background returned the peak height to its initial value. Therefore, collecting a fresh background often is important to achieve accurate methanol concentration measurements.