

Electrophoretic NMR (eNMR) – methods and applications

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1. Introduction

Methodological improvements to disentangle signal attenuation from electrophoretic displacement and convective bulk flow induced by Joule heating during electrophoretic pulses are presented, together with applications to fuel cell-related transport phenomena.

2. Experiments

Measurement procedures were tested both in a standard U-tube arrangement and in a new cylindrical cell geometry within a standard 5 mm diameter NMR tube. In the latter setup hydrogen gas bubble absorbing palladium electrodes were successfully tried. CPMG-like pulse sequence blocks designed to suppress convective bulk flow [1] were used and we demonstrated that the efficiency of those was improved by increasing the number and shortening the duration of repeating units. Improvements were particularly substantial for the cylindrical eNMR cell which has the advantage of a much larger filling factor and correspondingly higher signal-to-noise ratio over the U-tube cell. No bubble formation at the electrodes was detected for currents below 8 mA in the cylindrical eNMR cell used. The test experiments were made on 10-50 mM tetramethyl ammonium bromide (TMAB) solutions.

One of the applications of eNMR has been to probe electroosmotic drag in polymer electrolyte membranes [2]. Here we applied an electrophoretic stimulated echo pulse sequence on Nafion[®] membranes pretreated to contain water solutions of 0, 10, 20 and 30 mole % methanol. A 10 mm diameter cylindrical sample cell that contained a layered membrane stack and where one side of the cell was in contact with a solution reservoir was constructed for the study. Separation of the water and the methanol signals was achieved by slice selection techniques. The electroosmotic drag coefficient was obtained for each molecular component in the samples. Corresponding diffusion experiments were also performed. The thickness and resistance of the cells were also monitored and a higher degree of swelling and a higher resistance was noted as the methanol concentration increased. NMR imaging was applied to map the concentration profile of the electrolyte in the cell and the effects of current load on the liquid content and distribution within the membrane. Effects of Joule heating during the eNMR experiments were minimized by long repetition times and suitable cycling of the current over the consecutive NMR scans

3. Conclusions

The concept of eNMR has existed for some decades, but the technique(s) are still non-standard, and open for considerable development. eNMR is capable of providing unique multi-component information even on complex sample types. The field is still open for many new types of applications, as well as to advances in measurement procedures.

References

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