Contribution ID: **521** Type: **Oral presentation**

Quantitative EPR study of electron activated poly(vinylidene fluoride) and poly(ethylene tetrafluoroethylene) foils

The use of renewable energy sources and efficient energy storage systems is one option to reduce the future CO2 emission. Presently, redox-flow batteries are considered as potential alternative energy storage systems. In vanadium redox-flow batteries, a low cost and widely useable polymer electrolyte membrane (PEM) with high proton conductivity, low electron conductivity and low gas permeability as well as good mechanical and thermal stability is required. Previously, low-temperature polymer electrolyte membranes were prepared by electron induced activation of poly(vinylidene fluoride) (PVDF) and poly(ethylene tetrafluoroethylene) backbone material (ETFE). After electron activation in air, the activated PVDF and ETFE foils was stored at -30 ℃ prior to grafting in order to limit the loss of trapped radicals before subsequent graft radical copolymerization in the temperature range from 60 ℃ to 80 ℃.

The radical copolymerization depends on the thickness and swelling of backbone material, diffusion of monomers, density and types of trapped radicals, and microstructure of the backbone material.

With respect to a tailored design of the electron-induced activation of backbone material, detailed information on the influence of type and number of trapped radicals on the graft radical copolymerization is required. The electron paramagnetic resonance (EPR) spectra of PVDF and ETFE films were studied in order to identify type and concentration of trapped radicals after an electron treatment with 125 kGy in air at room temperature. The observed EPR spectra of PVDF and ETFE consist of superimposed signals of eight different radicals and six different radicals, respectively. The individual spectra were simulated by first derivatives of normalized Gaussian functions including intensity, linewidth, g value, and hyperfine splitting constants of the unpaired electron with hydrogen or fluorine atoms in α or β positions. In addition, the number of trapped radicals was estimated using a standard sample of known spin concentrations. The predominant radical in PVDF and ETFE is the CH-based mid-chain radical (-CF2-C*H-CF2-) and the peroxy radical, respectively.

Detailed EPR measurements in the temperature range from 40 ℃ to 80 ℃ without grafting agent will be compared with those EPR measurements at defined time of graft radical copolymerization. This information will be used to evaluate the effect of different types of trapped radicals on the subsequent graft radical copolymerization.

Acknowledgements

The authors acknowledge the German Science Foundation DFG for funding this work within the project “Polymer electrolyte membranes (PEM) for vanadium redox flow batteries” (project number: 411688235).

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Track Classification: Track 1: Advanced radiation chemistry and trends in radiation science and technology