

Capillary zone electrophoresis (CZE) is not only a widely used analytical separation method, but it is also an important technique for determining physico-chemical parameters of materials. Significant use is also in the characterization of potentially interesting macromolecular substances. In this master's thesis, CZE was applied to the study of polyelectrolyte–oppositely charged ionic surfactant complexes. For the first studied complex formed by poly(ethylene oxide)-*block*-poly(sodium methacrylate) and surfactant N-dodecylpyridinium chloride, the electrophoretic mobility of surfactant depending on the ratio of surfactant concentration in the sample to the concentration of polyelectrolyte present in the background electrolyte was measured. However, we did not get the significant decrease in mobility as was predicted in the literature based on measuring the  $\zeta$ -potential. This can be explained by the low stability of the complex in an electric field. The second system, the polyelectrolyte–surfactant complex of cationic–neutral hydrophilic block copolymer poly[3,5-bis(trimethylammoniummethyl)-4-hydroxystyrene iodide]-*block*-poly(ethylene oxide) and surfactant sodium dodecyl sulfate, could not be characterized using conventional CZE experiments described. The reason was probably due to a strong interaction of newly formed complex with the wall of the capillary, which could not be mitigated by the use of coating nor by the use of commercially coated capillaries.

In the second part, the electrophoretic behavior of block copolymer vesicles as a function of pH and ionic strength of the background electrolyte was investigated using capillary zone electrophoresis. The vesicles were comprised of polystyrene-*block*-polyacrylic acid. Electromigration behavior was interpreted in relation to the hydrodynamic radius of these particles measured by dynamic light scattering and compared with the acrylic acid behavior, which is a fundamental building block of vesicle polyelectrolyte brush. It was shown that vesicles compared to acrylic acid act as stronger acids and in a basic solution significantly respond to changes of bulk electrolyte concentration as a result of ion atmosphere formation, and localization of counterions in the interior volume of the polyelectrolyte brush. Electromigration behavior of vesicles depending on the time elapsed from their preparation was also observed as a part of this work. It was shown that even in solutions of low pH the mobility of the vesicles is not dependent on time.

In the last step, the cryogenic transmission electron microscopy was used to image the vesicles in the bulk.