Abstract

Within the scope of this thesis, four polar-embedded stationary phases were synthesized, characterized and their adsorption properties were investigated as well as their retention mechanism was described.

The stationary phases containing a phosphodiester group as a polar group and four different organic ligands - a decyl chain, an octadecyl chain, a benzyl substituent and a cholesterol molecule - were synthesized. This yielded two stationary phases previously developed by our research team (Diol-P-C10, Diol-P-C18) and two new stationary phases (Diol-P-benzyl, Diol-P-chol). The characterization began by determining the surface coverage density of the stationary phases with the modified ligands and determining their hydrophobicity using the Galushko test. The optimization of the packing process consisted of the selection of the slurry solvent, which was chosen one the basis of zeta potential results, microscopic studies and solvent viscosity. The final result was columns with efficiency similar to commercially packaged chromatography columns. In the following part of the work, the emphasis was on describing the adsorption properties of the prepared materials. Inverse size exclusion chromatography, the minor disturbance method and void volume marker analyses were used, of which the former proved to be the most suitable. The determination of the excess adsorption isotherms of acetonitrile and water confirmed the surface heterogeneity of each of the studied stationary phases. Chromatographic tests were performed to confirm the operation of the tested materials under pure water conditions with the principles of "green chromatography". The ability to separate polar and non-polar small-molecule compounds in both HILIC and RP LC systems was also confirmed. A mixture of purine alkaloids and nucleobases, as well as polycyclic aromatic hydrocarbons, were successfully separated. Due to the presence of polar and non-polar groups on the surface of the stationary phases, the retention mechanism was investigated by determining and modeling adsorption isotherms. For this purpose, the frontal analysis method and the inverse method were used. The applicability of the obtained materials was confirmed by analyzing a group of beta-blocker drugs, where computerized peak separation by intelligent peak deconvolution analysis was also used.

The results of the present study authorized a full characterization of stationary phases with embedded phosphodiester groups, which expands the range of materials used in high-performance liquid chromatography and develops the "green chromatography" trend.