Monolithic stationary phases in analysis of neurotransmitters

Martina Komendová, Jiří Urban

Department of Chemistry, Masaryk University, Kamenice 5, 625 00 Brno, Czech Republic

Dopamine belongs to a group of compounds known as neurotransmitters. Disorders of dopamine metabolism are associated with many neurological illnesses, such as schizophrenia, depression, Parkinson's disease, and tumors. The aim of the presented dissertation thesis is a development of monolithic stationary phases applicable in chromatographic screening of diseases related to a dopamine metabolism. The main focus of the thesis is the preparation and characterization of monolithic capillary columns and especially their surface modification providing fine control over the separation selectivity and efficiency.

At first, UV-initiated two-step photografting has been utilized to prepare monolithic capillary columns providing two zwitterion functionalities. Segments of five sulfobetaine polymer monoliths have been modified by phoshorylcholine and columns with 0, 33, 50, 66, and 100% of modified length. Effect of the length of the modified segment and mobile phase composition have been tested to improve selectivity of prepared columns.

Next, we have used grafting technique to hypercrosslink the surface of monolithic columns with poly(ethylene glycol) dimethacrylate. Length of crosslinking monomer, its concentration in the modification mixture, and a time of the modification reaction have been selected to control the extent of hypercrosslinking modification by a design of experiments protocol. Hypercrosslinked monolithic stationary phases provided lower effect of mass transfer resistance and allowed isocratic separation of dopamine-related compounds at higher mobile phase linear velocity.

An important part of the thesis is a development of a miniaturized separation device with electrochemical detection that might be applicable in integrated and portable analytical systems. Separation selectivity of monolithic stationary phases was controlled by different crosslinking monomers. We have developed a four-channel 3D printed separation device with integrated electrochemical detection allowing parallel analysis of a single sample.