ABSTRACT

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Title of Doctoral Thesis Nanofibers as modern extraction sorbents for sample extraction in

chromatographic analysis

Extraction techniques represent one of the most common approaches to sample handling. The analytes of interest can be isolated easily even from complex matrices, especially by solid-phase extraction (SPE). Therefore, this technique became very popular and routinely used in laboratories. The variety of commercially available sorbents has made this method even more attractive. Nevertheless, the new sorbent materials are still looked for because of the broadening spectrum of analyzed substances and the increasing demands on selectivity and extraction efficiency. Nanomaterials have been increasingly associated with these properties over the last ten years. Therefore, they are receiving more and more attention related to extraction procedures.

The dissertation thesis is focused on the application of nanofibrous polymers as innovative sorbents for solid-phase extraction. First, the extraction efficiency of nanofibers was tested in an offline set-up. The samples were extracted using a vacuum SPE manifold, and after that, the isolated analytes were determined by high-performance liquid chromatography (HPLC). However, most of the experiments were performed in the online connection of an extraction cartridge with a separation column via six-port switching valve directly in the chromatographic instrument. This online coupling ensured the time- and cost-effective analysis while the extraction efficiency remained or was improved. Thus, the requirements for modern sample pretreatment methods were met.

Besides the standard analyte solutions, the extraction efficiency of nanofibrous sorbent was also tested for biological samples. The ability of nanofibers to remove interfering substances was tested with bovine milk and human serum matrices. Most of the nanofibers were able to retain the low-molecular-weight analyte in the presence of proteins. This fact makes the nanofibrous sorbents very promising also for bioanalytical applications. The online extraction method for non-steroidal anti-inflammatory drugs was developed, validated, and applied on real samples of human serum to verify this statement.

To broaden the spectrum of analytes that are retained on the nanofibrous surface, the nanofibers were modified with specific functional groups. Since the selected analytes were β -lactam antibiotics, the quaternary ammonium group and sulphonyl group were bonded on the surface. These moieties should have to facilitate ion exchange between the sorbent and antibiotics to reach higher extraction efficiencies. The comparison between nanofibers from different fabrication procedures is included in this thesis. The suitability of sorbents made by polymer meltblowing, needleless electrospinning, and alternating-current

electrospinning for online coupling with a high-performance liquid chromatography system is evaluated. Furthermore, the influence of physicochemical properties of analytes on their retention during the extraction is discussed.