Abstract

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Title of the diploma thesis: HPLC method development and testing of a new nanofiber format for the extraction of polycyclic aromatic hydrocarbons (PAHs)

For the development of a new method for the determination of important polycyclic aromatic hydrocarbons, namely: Anthracene (ANT), acenaphthene (ACP), pyrene (PYR), chrysene (CHR), benzo(k)fluranthene (BkF), benzo(a)pyrene (BaP), dibenzo(a,h)anthracene (DaHa), indeno(1,2, 3-cd)pyrene (IcdP) and perylene (BghiP), high-performance liquid chromatography (HPLC) coupled with off-line solid-phase extraction (SPE) using nanofibres placed in spin-filters was used. The HPLC method was optimized in terms of choice of the mobile and stationary phases, flow rate, temperature, gradient elution, and wavelengths of fluorescence detection.

After deposition and extraction of a working solution of a mixture of PAHs on a nanofibrous sorbent embedded in the spin filter, the sample was eluted using acetonitrile (ACN) and centrifuged at 4000 rpm. Nanofiber sorbents made of polyamide (PA), polyimide (PI), polycaprolactone (PCL) or polybutylene terephthalate (PBT) and their various modifications with metal oxides and reactive dyes were used to test the extraction efficiency. Subsequently, a volume of 10 µl extracted sample was loaded onto an Ascentis Express AQ C-18 (150 mm x 4.6 mm, 2.7 µm particle size) analytical column. The separation was carried out under gradient elution with mobile phase A: Acetonitrile (70%) , B: distilled water (30%) at a flow rate of 1.5 ml/min at a constant temperature of 30 °C. Detection was performed using a fluorescence detector at excitation and emission wavelengths of 225/315 for ACP, 260/400 for ANT; PYR; CHR, 294/300 for BkF; BaP; DahA, 250/490 for Icdp, and 294/430 for BghiP.

In the last part of the work, the test of extraction and purification efficiency was performed on a real sample from the Elbe River collected in Hradec Kralove on the selected most suitable discs made of PA 11, PA 6/10 and "TACR" PBT 8 mm materials which showed the highest value of PAHs extraction.

Keywords: Polycyclic aromatic hydrocarbons (PAHs), HPLC, off-line SPE, nanofibers, anthracene (ANT), acenaphthene (ACP), pyrene (PYR), chrysene (CHR), benzo(k)fluranthene (BkF), benzo(a)pyrene (BaP), dibenzo(a,h)anthracene (DahA), indeno(1,2,3-cd)pyrene (IcdP) and perylene (BghiP)