ABSTRACT

Charles University in Prague, Faculty of Pharmacy in Hradec KrálovéDepartment of Pharmaceutical Chemistry and Drug ControlCandidateMgr. Jana KresováConsultantDoc. PharmDr. Petra Kovaříková, Ph.D.Title of ThesisStudy on possibility of utilization of alternative sorbents for sample preparation

Sample preparation belongs to basic operations during biological sample analysis. Overall success of analytical determination depends on its accomplishment. Nowadays, Solid Phase Extraction (SPE) is positively a dominant technique in the field of biological materials sample preparation.

Graphene is one of the new forms of carbonaceous materials. Due to its extraordinary properties (large surface area, structure rich in π -electrons, good thermal and chemical stability) it became a suitable adsorbent in analytic sample preparation.

The aim of this work was to evaluate extraction recoveries of several sorbents: ZirChrom[®]-SHAX (SHAX, polyethyleneimine-coated zirconia), ZirChrom[®]-SAX (SAX, polyethyleneimine-coated zirconia – less cross-linked than SHAX), ZirChrom[®]-PEZ (PEZ, ethylendiamine-N,N,N',N'-tetramethylenphosphoric-acid-coated zirconia), aminosilica (AS) both unmodified and graphene-oxide modified followed by thermal reduction. The extraction efficiency was tested using the model mixture containing a hydrophilic, a lipophilic, an alkaline, an acidic and a neutral analyte.

The recovery was evaluated with respect of the effect of pH, a volume of both sample and washing solution, including organic solvent addition and a degree of graphene reduction.

A higher recovery (over 70%) for all analytes was achieved using the sorbents modified with graphene when compared to those without modification. In comparison to neutral environment, a drop in recovery was observed for lidocaine on SHAX sorbent in acidic conditions, whereas ibuprofen was poorly extracted in alkaline environment. Neither

the amount of washing solvent, nor the volume of sample significantly influenced the recovery. The presence or organic modifier in washing solvent or sample caused the decrease in analyte retention. However, the extraction recovery was sufficient for most of the analytes. Regarding the preparation of the sorbent, the best results were achieved on SHAX sorbent modified with graphene, which was thermally reduced over 30 minutes.

A substantial drop in recovery was observed during the extraction of analytes from pure rabbit plasma. Various methods of plasma sample preparation prior to SPE were tested and the best results were achieved using precipitation with acetonitrile

In case of rabbit urine only dilution of the sample with water was tested.