SUMMARY

Drinking and natural environmental waters contain trace amount of metal ions. On the one hand, metal ions are necessary in order to regulate biological system of living organisms. On the other hand, some metal ions have harmful, toxic and carcinogenic effect. Scientific literature have proposed to use solid sorbents in order to preconcentration/separation of trace amount of metal ions from environmental waters. Most of them show adsorption properties towards cationic species of elements, and only few towards anionic species.

The aim of the dissertation is synthesis of new carbon nanocomposites based on graphene (G) and graphene oxide (GO) decorated with cerium (IV) oxide nanoparticles $(G/CeO_2 \text{ and } GO/CeO_2)$ and aluminium oxide nanoparticles $(Al_2O_3/nano-G \text{ and } Al_2O_3/GO)$. The synthesized carbon nanocomposites are characterized by good adsorption properties towards cationic as well as anionic species of selected elements. Obtained nanocomposites were investigated by spectroscopic and microscopic techniques, which confirmed that the structure of nanocomposites and the surfaces of G and GO were covered by CeO_2 and Al_2O_3 nanoparticles. The pH conditions of adsorption of selected metal ions on synthesized nanocomposites were investigated.

The maximum adsorption capacities of carbon nanocomposites were calculated using Langmuir isotherms and they were in the range $8.4 - 119.4 \text{ mg g}^{-1}$ for G/CeO₂, $5.8 - 30.0 \text{ mg g}^{-1}$ for GO/CeO₂, 32.8 mg g^{-1} for Al₂O₃/nano-G and $43.9 - 53.9 \text{ mg g}^{-1}$ for Al₂O₃/GO. Adsorption of selected metal ions on nanocomposites were studied for simultaneously influence contact times and sample volumes, influence of coexisting ions and humic acid (HA) the analyte recoveries. Each of obtained carbon nanocomposites was used to develop a new analytical procedures.

Dispersive micro solid phase extraction (DMSPE) with nanocomposites as solid sorbents in preconcentration and determination of metal ions in water samples was developed. Preconcentrated analytes were directly determination by energy dispersive X-ray fluorescence spectrometry (EDXRF) without elution step. DMSPE/EDXRF procedure eliminates sources of errors related to possible analytes losses and sample contamination, as well as the principles of Green Analytical Chemistry are regulated. In order to obtained a good results for determination of chromium ions, analyte preconcentrated onto G/CeO₂ and GO/CeO₂ was eluted and determined by inductively coupled plasma - optical emission spectrometry (ICP-OES). In this case EDXRF analysis was impossible due to the spectral coincidences between Cr K_a (5.41 keV) and Ce L_a (4.84 keV), Ce L_β (5.26 keV) observed in EDXRF spectra.

Developed analytical procedures were validated. The following parameters were determined: linearity, limit of detection (LOD), limit of quantification (LOQ), precision and accuracy. Selective adsorption of nanocomposite towards Se(IV) in presence of Se(VI), As(V) in presence of As(III) and Cr(III) in presence of Cr(VI) allowed to carry on speciation analysis as well as determination of selected metal species.