New Sorbent For Solid Phase Extraction And ICP-AES or ICP-MS Determination Of Trace Elements In Aquatic Systems

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Determination of trace elements in aquatic environment is actual analytical task. Modern analytical methods could be applied for direct determination however this usually requires sample transport to the laboratory and eventual contamination or losses of trace elements and matrix interferences (sea water) during the analysis. Column solid phase extraction procedures could be performed immediately after sampling thus avoiding any transformations, losses and contamination of trace elements and combining advantages of high preconcentration factors and interference free instrumental determination. Extraction efficiency directly related to reliable and correct finals results depends mostly on the properties of the sorbent used. Several very important features of the sorbent defined its applicability in field solid phase extraction procedures – high preconcentration factors, fast complex formation and stability constants of the complexes formed, low backpressure of the column, stability during storage. In the present study new sorbent based on modified silicagel is proposed for successful determination of trace elements from varies (sea, lake, river, mineral) water samples. The silicagel is first activated with HCl and then modified with 3-aminopropyltrimethoxysilane - \((\text{CH}_3\text{O})_3\text{SiOCH}_2\text{CH}_2\text{CH}_2\text{NH}_2\) (APS). For model laboratory experiments the sorbent 150 mg was placed in a small plastic syringe (1 ml) and sample solution pumped through the column at a flow rate of 5 ml min\(^{-1}\). The sorbent Si-APS permitted quantitative sorption (recoveries higher than 95 %) of Cd (II), Co(II), Cr(II), Cu(II), Fe(III), Ni(II) and Pb(II) in the pH range 6-9. Several eluents (HCL, HNO\(_3\), EDTA) were tested and it was found that 2 mol L\(^{-1}\) HNO\(_3\) ensured quantitative elution for all studied elements at flow rate of 2 ml min\(^{-1}\). As a next step the maximum volume of sample solution was defined based on the sea water model solution – around 100 ml sea water could be passed without deterioration of the performance of the sorbent. Than fast elution of all sorbed elements in 2 ml eluent is achieved. The acidity of the eluent is enough low and direct connection with the nebulizer of the ICP AES spectrometer is possible. In this way fast simultaneous quantitative determination of all tested elements is achieved. For final determination with ICP-MS dilution of the eluate (2-3 times depending on the Cu and Fe content) is recommended. The most important interference except high salinity (sea water) is expected to come from competitive complex formation with humic substances (DOM) present in the sample. Therefore model experiments were performed with 10 to 30 mg L\(^{-1}\) humics (SRHF) in the sample solution and quantitative recoveries for elements like Cu, Ni and Pb obtained undoubtedly confirmed the applicability of the sorbent for reliable and accurate direct determination of total dissolved concentrations of trace elements even in the water samples with high DOM content. The sorbent is stable during storage – quantitative elution of sorbed elements is possible after 1 month if stored at 4°C, in plastic bag, avoiding drying. Finally experiments were performed with sea water samples for manual performance of the procedure – model sample solution was pumped manually by syringe through the sorbent and than trace element eluted – recoveries in the range 93-95 % were achieved.