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# Magnetic solid-phase extraction to preconcentrate ultra trace amounts of lead(II) using modified-carbon nanotubes decorated with $NiFe_2O_4$ magnetic nanoparticles

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Here, multiwalled carbon nanotubes decorated with $NiFe_2O_4$ nanoparticles were synthesized by a simple reaction. Then, the nanoparticles were modified with 3-(trimethoxysilyl)-1-propanthiol. The modified-multiwalled carbon nanotubes decorated with $NiFe_2O_4$ nanoparticles were applied as a solid-phase extraction (SPE) sorbent for the extraction of ultra trace amounts of Pb(II) from water samples. Detection in this technique was performed by flame atomic absorption spectrometry. The influences of effective parameters on the extraction efficiency were studied. The best results were obtained at pH 6.0 (acetate–borate buffer) with 2.0 mL eluent solution of 1.5 mol $L^{-1}$ nitric acid and an extraction time of 30 min. The detection limit was 0.5 ng $mL^{-1}$ Pb(II) with a precision of 2.3–4.1%. The effects of matrix ions present in natural waters and some transition metals on the recoveries of the analyte ions were also examined in model solutions. Finally, the proposed method was used for the measurement of Pb(II) in water from the Shiraz Khoshk river (Shiraz, Iran), food, and industrial waste water from Isfahan's Mobarakeh Steel Company.



## 1 Introduction

Some heavy metals like copper and cobalt are beneficial for human health, however some other heavy metals like lead, mercury and cadmium are harmful to humans. The main sources of heavy metals in environmental samples are industrial facilities and traffic.[1,2] With increasing industrial activities, waste water from many industries such as chemical manufacturing, mining, battery manufacturing industries, *etc.*, contains toxic heavy metals. These are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders.[3]

Various analytical techniques are commonly used in the determination of trace metal ions, including spectrophotometry,[4] atomic absorption spectrometry,[5,6] inductively coupled plasma atomic emission spectrometry,[7,8] polarographic analysis, inductively coupled plasma mass spectrometry[9,10] and potentiometric stripping analysis.[11] These methods are usually inappropriate alone because of a variety of factors, particularly the low concentrations of metal ions and matrix effects of real samples. Under these situations, in order to achieve accurate and reliable results, an efficient separation and preconcentration procedure prior to the determination of trace metal ions could be a good choice, such as ion exchange, chelating matrices, liquid–liquid extraction,[12,13] solid-phase extraction (SPE)[14–16] and cloud point extraction.[17]

Analytical methods based on sorbent extraction have proven to be the most attractive ones due to their specificity and high preconcentration efficiency. The wide range of choice of sorbent materials along with various chelating reagents and eluents make this technique very attractive for sample pretreatment.[18–22] Separation technology using magnetic materials has received considerable attention in recent years.[23] It provides great potential for applications in cell separation.[24] The sorbent need not be packed into the cartridge, as in traditional SPE, and the phase separation can be realized easily by applying an external magnetic field. Various magnetic nanoparticles have been used with different surface functionalization that allow immobilized affinity ligands to capture target biomaterials. Surface modifications are usually achieved through the attachment of inorganic shells or/and organic molecules that stabilize the nanoparticles and protect them from being oxidized. Recently, a lot of investigations have been devoted to designing nanoparticles with surface coating layers sensitive to variations of temperature,[25,26] pH value[27,28] and specific analytes.[29]

By using an external magnetic field, magnetic materials can be readily isolated through sample solutions. This property makes them particularly suitable for sample preparation because no centrifugation or filtration of sample is needed after

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