



In-situ differential pulse anodic stripping voltammetry combined with hollow fiber-based liquid-three phase micro extraction for determination of mercury using Au-nanoparticles sol-gel modified Pt-wire

Ali A. Ensafi *, Ali R. Allafchian, M. Saraji, B. Farajmand

Department of Chemistry, Isfahan University of Technology, Isfahan 84156-83111, Iran

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ABSTRACT

A new method has been proposed based on hollow fiber-based liquid three-phase micro extraction and in-situ differential pulse anodic stripping voltammetry (DPASV) for the micro extraction and quantification of mercury(II) ions. Different factors affecting the liquid-three phases micro extraction, including organic solvent, pH of the donor and acceptor phases, concentration of the complexing agent, extraction time, and stirring rate were investigated and the optimal extraction conditions were established. Three microelectrodes designed and constructed for this study were inserted into the two ends of a hollow fiber inside the acceptor solution, and then voltammetric analysis was performed in-situ during the extraction time. After 1600 s, final stable signal was used for the analytical applications. Under the optimized conditions, an enrichment factor of 277 was achieved and the relative standard deviation (R.S.D.) of the method was 6.2% ($n=5$). The calibration curve was obtained in the range of $0.2\text{--}30.0 \text{ nmol L}^{-1}$ Hg(II) with a reasonable linearity ($R^2 > 0.9880$) and a limit of detection of 0.06 nmol L^{-1} . Finally, the applicability of the proposed method was evaluated by extraction and determination of mercury in real samples such as fish and rice.

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1. Introduction

Heavy metals are persistent environmental contaminants. They cannot be metabolized by the body and are not stable and bioaccumulative. These toxic metals are sometimes passed up the food chain to humans. They have toxic effects on the environment and life in aquatic system at trace level too. Among the toxic trace metals, mercury ion is one of the most hazardous environmental pollutants that can affect the nervous system. It is found as an industrial waste because of its growing area in production of some batteries, thermometers, cameras, mercury vapor lamps, calculators, and has been used as a catalyst in the production of urethane polymers for plastics. Thus, it is very important to determine mercury at trace levels in different samples.

Various common methods are used to determine mercury ions such as potentiometry [1,2], spectrophotometry [3], atomic absorption spectrometry [4–6], inductively coupled plasma [7,8], atomic fluorescence spectrometry [9], X-ray fluorescence [10], voltammetry [11–14], liquid-liquid extraction [15], and complexometry [16]. Among these techniques, electrochemical

methods are interesting because of simplicity, environmental friendly and sometimes good sensitivity.

Anodic stripping voltammetry (ASV) and cathodic stripping voltammetry (CSV) have received very much attention for metal ions determination owing to their intrinsic sensitivity [13,17–19]. It occurs because in stripping analysis, a preconcentration step is combined with a stripping step, thereby enhancing the sensitivity [20,21]. Leakage in selectivity is an essential problem of ASV, especially in analyzing complex real samples. In fact, it often happens that different species undergo redox reactions at potential values that are very close to each other. Prevalent experimental manipulations, such as changing the supporting electrolyte pH or using modified electrodes and chemometrics methods, offer efficient options to overcome the problem of overlapping signals [22]. Moreover, the application of sample preparation techniques could be an effective alternative for elimination of interferences encountered in electrochemical analysis of complex matrices such as food, blood and wastewater.

Hollow fiber-based liquid three-phase micro extraction (HF-LPME) method has been a powerful preparation method in recent years [23–27]. This technique can provide preconcentration and clean up of analytes simultaneously. In this method, analytes of interest are extracted from aqueous samples (donor solution) through the thin layer of an organic solvent, which is immobilized within the pores of a porous hollow fiber and then back-extracted

* Corresponding author. Tel. +98 311 3912351; fax: +98 311 3912350.

E-mail addresses: ensafi@cc.iut.ac.ir, ensaf2009@gmail.com (A.A. Ensafi).