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A HIGH-THROUGHPUT SOLID-PHASE EXTRACTION MICROCHIP COMBINED WITH INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY FOR RAPID DETERMINATION OF TRACE HEAVY METALS IN NATURAL WATER

Annotation

Herein, a hyphenated system combining a high-throughput solid-phase extraction (htSPE) microchip with inductively coupled plasma-mass spectrometry (ICP-MS) for rapid determination of trace heavy metals was developed. Was improved the processing speed for individual samples by increasing the operation flow rate during SPE procedures. To this end, an innovative device combining a micro mixer and a multi-channeled extraction unit was designed. Furthermore, a programmable valve manifold was used to interface the developed microchip and ICP-MS instrumentation in order to fully automate the system, leading to a dramatic reduction in operation time and human error.

Validation procedures demonstrated that the developed method could be satisfactorily applied to the determination of trace heavy metals in natural water. Each analysis could be readily accomplished within just 186 s using the established system. This represents, to the best of our knowledge, an unprecedented speed for the analysis of trace heavy metal ions.

Key words: heavy metals, monitoring, multi-isotopic analysis, analytical reagent, matrix-mimic solution, microchip, multi-channeled extraction, concomitant ions, turbulence mechanisms, microscopic scale.

Introduction

Trace heavy metal elements occur naturally in the earth's crust [1] and are fatally discharged into the environment by either natural processes or human activities [2]. High levels of these metals in aquatic environments are well - known to cause serious pollution problems and are toxic to a variety of ecosystems [3]. Conversely, some metal species are essential for certain physiological reactions, and enter the human body through the food chain [4]. However, excessive intake of these metals is harmful to human health [5]. Thus, from the viewpoint of environmental management and contamination prevention, the fast and accurate monitoring of the levels of trace heavy metals in natural water is of great importance.

Inductively coupled plasma-mass spectrometry (ICP-MS) is widely recognized as one of the most powerful methods for trace metal analysis by virtue of its superior analytical features, such as its wide linear dynamic range, its suitability for simultaneous multi-elemental and multi-isotopic analysis, and its ability to facilitate the ultra sensitive analysis of numerous samples [6]. However, the effects of salt build-up and polyatomic interference remain problematic for accurate determination of trace metals in complicated matrices using ICP-MS instrumentation [7]. Thus, while raw samples are unsuitable for direct analysis, applying appropriate pretreatment procedures to the collected sample prior to ICP-MS measurement is indispensable [10].

Up to date, numerous sample pretreatment techniques, such as liquid extraction [11], solid-phase extraction (SPE) [14] and microwave digestion [19, 20], have been successfully applied to the determination of trace heavy metals with ICP-MS instrumentation. Of these techniques, SPE is especially popular because both sample clean up and analyse concentration can be accomplished simultaneously [21]. Recently, the development of small SPE-capable devices has surged due to their unique advantages, such as very low reagent/sample requirement, potential for multi-functionality, and so on, which benefit from downsizing the dimensions of analytical systems [22].

Materials and methods

Chemicals and materials. All chemicals were of analytical reagent grade and used as received unless otherwise stated. Were used deionized water, sodium dodecyl sulfate (SDS), sodium hydroxide (NaOH), nitric acid (HNO_3, 70.0%, ultra pure reagent grade), and ammonium hydroxide (NH_4OH, 20%, ultra pure reagent grade) and Maleic acid disodium salt hydrate and rhodamine B. Stock solutions of analytes (Mn, Co, Ni, Cu, and Pb) and elements (Na, Mg, Al, K, Ca, Sr, and Ba) were purchased from Merck (Darmstadt, Germany).

Fresh working standards for calibration were prepared daily by stepwise dilution of stock solutions using DI H_2O. The matrix-mimic solution used to evaluate interference effects was prepared by diluting stock solutions of possible interferon's using DI H_2O. Detailed information on the components of the matrix-mimic solution is provided in Table 1. All processes involving reagent preparation were carried out in a class 100 laminar flow hoods.

All containers and pipet tips used in this study were cleaned by overnight immersion in concentrated HNO_3, followed by rinsing with DI H_2O. The tubes used to connect the components of the apparatus were flushed with DI H_2O until all contaminants were eliminated. To avoid additional contamination, fully plastic Norm - Ject syringes (Tufflingen, Germany) were used throughout this study.

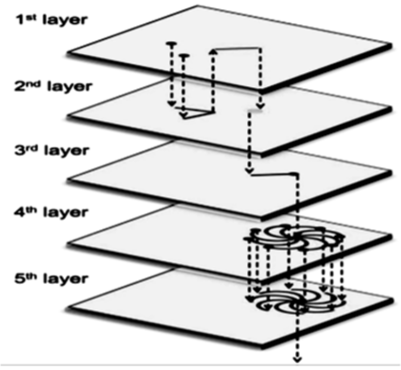
Table 1 - Composition of the matrix-mimic solution.

Composition of the matrix-mimic solution	
Element	Concentration
Na	20
Mg	8000
Al	100
K	2000
Ca	30 000
Sr	300
Ba	500

Fabrication of the htSPE microchip. Was used the microchip with carbon dioxide laser micro machining system (LES-10, Laser Life Co. Ltd., Taipei, Taiwan). Consists five-layered htSPE microchip divided into mixing unit (the first, second, and the third layer) and extraction unit (the fourth and fifth layer). And mixing unit consisted of a chamber containing a small bar. The extraction unit consisted of eight circle-shaped extraction channels and each channel had two semicircular channels connected via an access hole. [28].

The channel features were inspected using a high-resolution optical microscope. The length of each effective extraction channel, defined as the distance around the circumference of a circle-shaped channel, was approximately 25 mm. All the channels and ports used for sample/reagent introduction and confluent outlet were machined meticulously. The microchip was completed after subjecting the two units to cleaning, bonding, and conduit installation. The fluid flow in the htSPE microchip is illustrated in Fig. 1.

The developed micro mixer was assessed by visually estimating the degree of mixing of two different liquids. DI H_2O and 0.5% rhodamine B were selected as indicators [36] and loaded into the microstructure via the reagent introduction ports. Photographic images of the detection points positioned at 2 mm upstream and downstream from the micro mixer in the micro channel were acquired using an optical microscope with a charge-coupled device camera (0.41 million pixels, 40 ms exposure time). Photographs captured at the two positions were used to compare the liquid streams in the initial state and after the two liquid streams had passed through the micro mixer. The color photographs were converted into grayscale images and then analyzed using the freely available image processing program [37, 38].



Figur 1 - The fluid flow in the htSPE microchip.

Construction of the hyphenated htSPE micro chipe ICP-MS system. A diagram of the hyphenated htSPE micro chipe ICP-MS system can be divided into two main parts; the sample pretreatment system and the detection system. The sample pretreatment system consisted of a six-port electric actuator valve, two ten-port electric actuator valves, and the in-house-fabricated htSPE microchip. All of the valves were programmed and controlled by a personal computer via a serial valve interface. An ICP-MS instrument was used as the detection system. A Micromist nebulizer was fitted to a Scott-type quartz double-pass spray chamber. Polytetrafluoroethylene (PTFE) tubes were used to connect all components of the system. A peristaltic pump with peristaltic tubing was employed to deliver sample solutions and reagents. The outlet of the peristaltic tubing was modified for attachment to the PTFE tube via a conical adapter.

Analytical procedure. Initially, a conditioning solution (0.2% NH_4OH) was passed through the microchip to transform the channel surface into a chemical form suitable for the retention of metal ions. The sample solution and buffer solution were then simultaneously introduced to the microchip and mixed via the micro mixer to attain the desired pH for on-chip extraction, and the desired analytes were retained in the channel interior. Finally, the retained analytes were eluted from the channel interior by a 0.5% HNO_3 solution, and directly delivered into the ICP-MS detection system.

Sample preparation. The water sample was collected from the river using polyethylene (PE) screw-cap bottles containing an aliquot of concentrated HNO_3. The bottles were cleaned with 40% HNO3 and then flushed with DI H_2O. The bottles were rinsed thoroughly with the designated water prior to sample collection. The collected samples were stored at 4 ^{0} C in the dark and filtered through a PTFE membrane prior to use.

Results and discussion

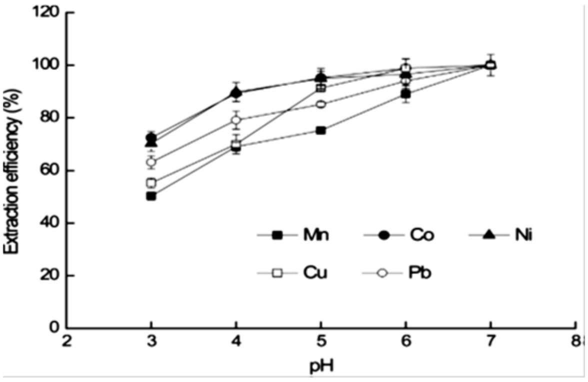
Design of the htSPE microchip. Efficient on-chip SPE techniques that can rapidly complete analytical work are needed for trace metal analysis. Thus, an alternative strategy based on increasing the operation flow rate during SPE procedures and shortening the processing period for individual samples was adopted in this study. However, adverse effects on the extraction efficiency of this strategy might be possible. For example, insufficient mixing between sample and buffer solutions caused by increasing the operation flow rate during SPE procedures might lead to undesired extraction conditions for analytes. Also, lower residence time for analytes caused by increasing the operation flow rate during SPE procedures could lead to insufficient contact with the extraction medium. To overcome these problems, both a micro mixer and a multi-channeled extraction unit were integrated into the developed device.

Efficient mixing in order to assist effective reactions is essential in various analytical applications. However, mixing two or more fluids at the microscopic scale within a short period is extremely difficult because mixing in micro-space relies mainly on diffusion rather than traditional convection or turbulence mechanisms [40]. A diverse range of micro mixers have been designed for enhancement of fluid mixing in micro fluidic systems [43, 44]. However, most of these micro mixer designs are complicated in terms of fabrication. Therefore, a simple way to prepare a mixing unit was proposed in this study.

To address problems associated with limited contact time between analytes and extraction media at increased flow rates during SPE procedures, a two-layer crossing extraction unit in which eight circle-shaped extraction channels were connected in parallel was employed. The extraction unit distributes the stream into eight channels from the center and then recombines the separated streams at the confluent outlet (see Fig. 1A). The logic behind this design is that the linear flow rate of the stream is reduced by a factor of eight as the stream transfers from a single channel to the eight circle-shaped channels [44]. Consequently, the residence time for analytes is 8-fold longer than in a single channel design at the same flow rate. This separated two-layer arrangement was used to efficiently exploit the substrate space so that the size of the htSPE microchip could be reduced.

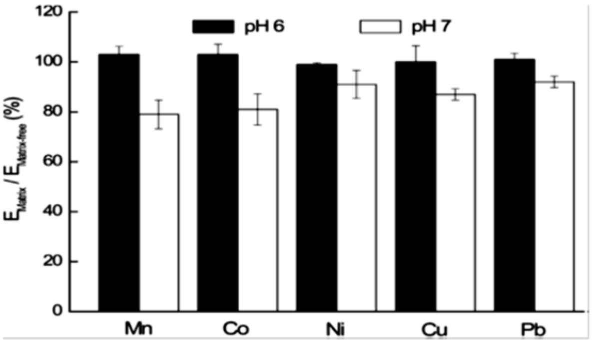
Optimization of htSPE in the microchip. To optimize the analytical performance of the htSPE microchip for collecting analyte ions from sample matrices, operating conditions, such as the pH of the extraction environment and the extraction flow rate, were investigated.

Influence of pH on extraction efficiency. Complexation between metal ions and the carboxylate moieties in the functionalized PMMA microchip is the main mechanism facilitating the efficient retention of analytes in the channel interior [28]. In other words, such retention behavior is critically dependent on ionic interactions. Considering the extraction mechanism of metal ions on the channel surface used in this study was essentially the same as that used in our previous work, the acidity of the extraction environment that determines the charge status of the analytes was examined. As shown in Fig. 3, an increasing trend in the extraction efficiency for all metal ions was observed in the pH range from 3.0 to 6.0, and the extraction efficiency for most ions reached a plateau as the pH exceeded 6.0.



Figur 2 - Variations in the extraction efficiency of analytes with respect to pH.

Nevertheless, some ions present in natural water might hamper efficient extraction of trace metals. Thus, the competition between analyte ions and concomitant ions for the carboxylate moieties on the interior of the extraction channels needed to be carefully evaluated. Fig. 4 shows the difference in the extraction of analyte ions from a matrix-mimic solution and matrix-free DI H2O (EMatrix/ EMatrix-free) at pH 6.0 and 7.0. The ratio at pH 6.0 was quite apparently superior to that obtained at pH 7.0, suggesting that the adverse effects caused by the concomitants could be lessened by using an extraction environment of pH 6.0. Therefore, a pH value of 6.0, which provided desired signal intensity for each analyte in the presence of concomitant ions, was selected as the working pH for the subsequent experiments.



Figur 3. Change in the extraction efficiency of analytes prepared in the matrix-mimic solution to those prepared in matrix-free DI H2O (EMatrix/EMatrix-free) at pH 6 and 7. Information on the components of the matrix-mimic solution is provided in Table 1.

Conclusions

In this work, high-throughput sample processing for trace heavy metals was accomplished by an htSPE microchip which consisted of a micro mixer and a multi-channeled extraction unit. Successful combination of the developed htSPE microchip, a programmable valve manifold, and ICP-MS instrumentation expedited fully automated analysis. Satisfactory analytical results indicated that the proposed system exhibited superior features of rapidly determining metal ions of interest in natural water. Remarkably, the hyphenated htSPE microchip system developed in this study can complete an individual analysis for trace heavy metal ions in an unprecedented time scale.

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ТАБИҒИ СУЛАРДА АУЫР МЕТАЛДАРДЫҢ МИКРОЭЛЕМЕНТТЕРІН ТЕЗ АНЫҚТАУ ҮШІН ЖОҒАРЫ - БІРГЕ СЫЙЫМДЫЛЫҚТЫ ИНДУКТИВТІ ПЛАЗМАЛЫҚ МАСС-СПЕКТРОМЕТРИЯ ҚАТТЫ ФАЗАЛЫ MICROCHIP ҚОЛДАНУ

Түйін

Ауыр металл іздерін тез арада анықтау үшін қатты фазалы өндіру біріктіретін жүйесін индуктивті-байланысқан плазмалық масс-спектрометрия (ICP-MS) жоғары сыйымдылығы микрочип дайындалды. Аналитикалық қабілетін арттыру үшін, SPE рәсімдер өңдеу жылдамдығы кезінде ағын жылдамдығын арттыру арқылы жеке үлгілер жақсартылды. Осы мақсатта біз микро-миксер және көп арналы өндіруші блок үйлестіре инновациялық құрылғы дайындалды. Сонымен қатар, жүйені толық автоматтандыру үшін, бағдарламаландырған клапан қолданды, микрочип және ICP-MS құралдарын қосу үшін арналған, жұмыс уақытын және адам қателіктер айтарлықтай төмендетуге арналған аспап қолданды. Сынақ талдау әдісі, табиғи суларда металдардың іздерін анықтау үшін пайдалануға мүмкін екенін көрсетті. Орнатылған жүйе бойынша әрбір талдау оңай, 186 с ішінде жүзеге асырылуы мүмкін.

Кілт сөздер: ауыр металдар, мониторинг, мультиизотоптық бақылау, аналитикалық реагент, матрицалық мимикалық ерітінді, микрочип, көпарналы экстракция, байланысты иондар, турбуленттік механизм, микроскопиялық масштаб.

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МИКРОЧИП С ВЫСОКО - ПРОПУСКНОЙ ТВЕРДОФАЗНОЙ ЭКСТРАКЦИЕЙ В СОЧЕТАНИИ С ИНДУКТИВНО СВЯЗАННОЙ ПЛАЗМОЙ МАСС-СПЕКТРОМЕТРИИ ДЛЯ БЫСТРОГО ОПРЕДЕЛЕНИЯ МИКРОЭЛЕМЕНТОВ ТЯЖЕЛЫХ МЕТАЛЛОВ В ПРИРОДНЫХ ВОДАХ

Аннотация

Для быстрого определения микроэлементов тяжелых металлов было разработано системное комбинирование экстракции твердой фазы с высокой пропускной способностью микрочипа с индуктивно связанной плазмой масс-спектрометрии (ICP-MS). Для повышения аналитической пропускной способности, путем увеличения скорости потока во время процедур SPE была улучшена скорость обработки данных для отдельных образцов. С этой целью, было разработано инновационное устройство комбинирования микро-смесителя и многоканального блока экстракции. Кроме того, чтобы полностью автоматизировать систему был использован программируемый клапанный коллектор для соединения разработанного микрочипа и ICP-MS измерительных приборов, что также приводит к предотвращению ошибок и траты лишнего времени. Анализ проверки показал, что разработанный метод может быть удовлетворительно использован для определения микропримесей тяжелых металлов в природных водах. Каждый анализ с помощью установленной системы может быть легко осуществлен в течение 186 с.

Ключевые слова: тяжелые металлы, мониторинг, мультиизотопный анализ, аналитический реагент, матричный мимический раствор, микрочип, многоканальная экстракция, сопутствующие ионы, механизмы турбулентности, микроскопический масштаб.

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ПОЛОЖИТЕЛЬНАЯ РОЛЬ РАСТЕНИЙ В ТЕХНОГЕННОЙ СРЕДЕ

Аннотация

В статье приведены данные положительной роли растений в техногенной среде. Показатели газообмена в течение вегетационного периода у разных деревьев неодинаковы. Известно более 500 видов растений, обладающих в разной степени фитонцидными свойствами.

Ключевые слова: растения, газообмен, влажность воздуха, пылезащитные свойства, техногенная среда, снижение шума.

Введение

Растения обогащают воздух кислородом, увлажняют и очищают его, способствуют снижению шума, влияют на микроклимат территории. Загрязнение воздуха, воды, почвы оказывает влияние на физиологические функции растений, их внешний облик, состояние, продолжительность жизни, генеративную серу. Вещества – токсиканты адсорбируются на клеточных оболочках растений, проникают внутрь клеток, нарушают обмен веществ; в результате резко снижается фотосинтез, усиливается дыхание. Обычно признаки поражения растений токсикантами выражаются в некрозе края листа, побурении листьев и хвои, появлении уродств, отмирании. Пыль, оседающая на листья, действует как экран, снижающий доступ света и усиливающий поглощение тепловой радиации. Кроме того, возможна закупорка листьев пылевыми частицами.

Материалы и методы исследований

В ходе исследований были использованы биологические методы исследований для изучения состояния лесного фонда в наиболее типичных участках [1,2].

Результаты и обсуждение

Насаждения, как известно, поглощают из воздуха углекислоту, выделяемую человеком, и обогащают воздух кислородом. Это свойство насаждений используется для улучшения состава воздуха, его оздоровления. Некоторыми учеными даже выдвигалась теория нормирования количества насаждений в городах применительно к этому свойству растений. Взрослый здоровый лес на площади 1 га поглощает 220-280 кг углекислого газа,