



Determination of trace cadmium in rice by liquid spray dielectric barrier discharge induced plasma – chemical vapor generation coupled with atomic fluorescence spectrometry

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ARTICLE INFO

Article history:

Received 16 October 2017

Received in revised form 10 December 2017

Accepted 28 December 2017

Available online 02 January 2018

Keywords:

Plasma chemistry vapor generation
Liquid spray dielectric barrier discharge
Atomic fluorescence spectrometry
Rice
Cadmium

ABSTRACT

Cadmium contamination in rice has become an increasing concern in many countries including China. A simple, cost-effective, and highly sensitive method was developed for the determination of trace cadmium in rice samples based on a new high-efficient liquid spray dielectric barrier discharge induced plasma (LSDBD) vapor generation coupled with atomic fluorescence spectrometry (AFS). The analytical procedure involves the efficient formation of Cd volatile species by LSDBD plasma induced chemical processes without the use of any reducing reagents (Na/KBH₄ in conventional hydride generation). The effects of the addition of organic substances, different discharge parameters such as discharge voltage and discharge gap, as well as the foreign ion interferences were investigated. Under optimized conditions, a detection limit of 0.01 μg L⁻¹ and a precision of 0.8% (RSD, *n* = 5, 1 μg L⁻¹ Cd) was readily achieved. The calibration curve was linear in the range between 0.1 and 10 μg L⁻¹, with a correlation coefficient of R² = 0.9995. Compared with the conventional acid-BH₄⁻ vapor generation, the proposed method not only eliminates the use of unstable and expensive reagents, but also offers high tolerance for coexisting ions, which is well suited to the direct analysis of environmental samples. The validation of the proposed method was demonstrated by the analysis of Cd in reference material of rice (GBW080684). It was also successfully applied to the determination of trace cadmium in locally collected 11 rice samples, and the obtained Cd concentrations are ranged from 7.2 to 517.7 μg kg⁻¹.

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1. Introduction

Cadmium (Cd) is one of the most toxic metal elements for humans, and its widespread industrial uses result in pollution of natural and agricultural environments [1]. In China, soil has been proved seriously polluted by Cd, and with the following trend: northwest > south-west > south central > east > northeast > north [2]. Cd in soil disperses into agricultural crops through a variety of biological paths and then enters the food chain with the potential to cause serious health problems such as kidney disease, skeletal damage, and even cancers [3,4]. There are at least 500 million people eating rice as staple food for daily energy in China [5]. Considering that the dietary status of rice and the dangers of Cd, accurate determination of trace Cd in rice is particularly important [6].

Elemental determination in rice samples is not an easy task, especially when analytes are present at low concentrations. It is generally believed that efficient sample introduction techniques are a guarantee of atomic spectrometry techniques for trace or ultra-trace element

analysis, so various effective sample introduction techniques have been developed. As an excellent sample introduction technique, chemical vapor generation (CVG) method has been increasingly used in the determination of trace Cd by coupling with spectrometric techniques (atomic absorption spectrometry (AAS) [7–10], atomic fluorescence spectrometry (AFS) [11–13], and even inductively coupled plasma mass spectrometry (ICP-MS) [14], due to its high sensitivity and selectivity. And several enhancement reagents including iodate [15], thio-urea together with cobalt [16], and hexacyanochromate [14] have been reported to improve the Cd CVG efficiency and analytical performance. However, the conventional CVG is achieved by use of unstable, and expensive reagents. Moreover, it suffers from the significant interference from some transition and noble metals [17,18], which greatly limits its usability. Therefore, great efforts have been paid to develop alternative green vapor generation techniques for Cd.

As an emerging novel vapor generation method, several elements have been reported successfully by photochemical vapor generation (PCVG), including Se, Hg, As, Sb, Cu, Au, Ni, Co, and Fe etc. [19–21] Unfortunately, since its introduction, progress in research and real application to trace Cd analysis remains limited. Until 2011, an efficient PCVG method for Cd was proposed by the Sturgeon group [22]. But the

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relative standard deviation (RSD) was near 30%, and it is difficult to be applied to the measuring of real samples. Electrochemical hydride generation (EcHG) is another emerging vapor generation method. The EcHG of Cd was first reported by Arbab-Zavar et al., who used a lead–tin alloy as the cathode [23]. However, this method can only be applied for the determination of Cd in tap water by standard addition method, because of significant interferences from some coexisting ions. In 2012, Zhang et al. developed a modified version of EcHG reactor for the determination of Cd in water and rice samples, in which Ti foil was used as cathode [5]. Although EcHG has been considered a green vapor generation method, some problems urgently need to be solved to make it a more acceptable sample introduction technique, for example, the poor stability of the cathode and interferences from coexisting ions [24–26].

Plasma induced CVG, which requires only a simple plasma reactor, is another emerging green and cost-effective vapor generation technique [18]. In 2013, a thin-film DBD (TFDBD) plasma-assisted chemical process for Cd vapor generation was first developed by our group [24]. In later developments, we demonstrated that the addition of non-ionic surfactants could enhance Cd vapor generation efficiency and thus improve the detection limits of the TFDBD plasma-CVG [27]. In 2014, Zheng et al. reported a single drop solution electrode glow discharge (SD-SEGD) plasma vapor generator and achieved the determination of ultratrace Cd in human hair samples and paramecium cells [28]. In 2016, our group developed a solution anode glow discharge (SAGD) atomic emission source, and demonstrated that the volatile species of Cd are readily generated in SAGD [29]. Subsequently, similar results were also described by Greda et al. [30] and Jamroz et al. [31]. Recently, our research group further proposed a new strategy for the high-efficient generation of volatile species of Cd based on our previous research [26]. These plasma-induced vapor generation methods offer several advantages, including low cost, high vapor generation efficiency and being environmentally friendly. However, despite these progress, the application of plasma-CVG for analysis of practical sample is rather limited. For DBD-CVG, the significant interferences from coexisting ions may account for its limited application [18,24,27]. For solution electrode induced vapor generation methods, it has higher tolerable levels for coexisting ions in the sample compared to DBD-CVG, but this device is less portable and requires strict assurance of the stability of the liquid electrode [26,28].

Our lately work successfully established a new method for trace Pb determination in geological samples by liquid spray dielectric barrier discharge plasma induced — chemical vapor generation (LSDBD-CVG) technique [32]. The most distinguish characteristics of this LSDBD — CVG method is its high tolerance for coexisting ions in the sample compared to other plasma induced vapor generation techniques [24,27]. Its excellent anti-interference properties will likely to be applied to other elements because of its unique advantages. In this work, the purpose is to develop a simple, sensitive and green LSDBD-CVG method to monitor the Cd pollution of the rice by AFS. In the present study, the vapor generation of Cd by LSDBD plasma were evaluated, and the effects of operating parameters were investigated, analytical figures of merit for the apparatus were determined. Several real rice samples were determined by the proposed method and compared with ICP-MS. It has been demonstrated that LSDBD-CVG-AFS provides a sensitive, ease of use, green and robust method for Cd determination in rice.

2. Materials and methods

2.1. Instrumentation

The configuration of the liquid spray DBD plasma reactor is similar to that reported in our previous work [32]. It contains two major parts: the nozzle of a concentric micronebulizer (Beijing Zhanxing Technology Service Center, Beijing, China) wrapped with copper foil as one electrode and a piece of copper foil attached onto the outer surface of a

glass plate acting as the counter electrode. The discharge gap is set at a distance of 3 mm, and the volume of the reactor is about 10 mL. The aqueous sample solution is nebulized into fine droplets and simultaneously reacted with the DBD microplasma sustained between the nozzle tip and the glass plate base, which results in the generation of volatile species of Cd by plasma induced chemical process.

The determination of the volatile cadmium species was performed with a model AFS-9130 (Beijing Titan Instruments Co. Ltd., Beijing, China) double-channel non-dispersive atomic fluorescence spectrometer fitted with an electrically heated quartz tube atomizer (QTA) in this study. The QTA used in our study is a flame-in-gas-shield atomizer, which consist of a concentric inner tube and outer shielding tube. The dimensions of the QTA were as follows: concentric inner tube—5.6 mm i.d. and 7.9 mm o.d.; outer shielding tube—10.0 mm i.d., 12.0 mm o.d. The detailed operating conditions are listed in Table 1. A commercial neon lamp power supply (25 W, Guangzhou Jinshi electronic equipment Co., Ltd., Guangzhou, China) is employed to sustain the DBD plasma. The sample solution is delivered into the micronebulizer by a peristaltic pump (BT 101 L, Leadfluid Co., Ltd., China). In addition, a condensed gas-liquid separator (GLS) was added between the reactor and QTA to remove the large droplets before introduced into the QTA. An Agilent 7700× ICPMS was applied to measure the rice samples and estimate the vapor generation efficiency of the proposed LSDBD-CVG method. The following operating conditions are employed: RF power: 1550 W, plasma gas flow: 15.0 L min⁻¹, auxiliary gas flow: 1.0 L min⁻¹, carrier gas: 1.1 L min⁻¹, sampling depth: 7 mm, isotope used: ¹¹²Cd, internal standard used: ¹⁰³Rh.

2.2. Analytical procedures for LSDBD-CVG-AFS

A schematic diagram of the experiment setup of the LSDBD-CVG-AFS is shown in Fig. 1. During the analysis, the sample solution was continuously introduced into the reactor by a peristaltic pump at a flow rate of 2.0 mL min⁻¹. Argon was streamed at a flow rate of 600 mL min⁻¹ into the micronebulizer for solution nebulization. In the next step, the DBD plasma was ignited by turning on the neon lamp power supply, and the volatile species of Cd were immediately generated. Then, the generated volatile species produced from the plasma chemical process were swept by an argon stream through the outlet of the GLS and then to a T-tube and mixed with auxiliary H₂. Last, the gas containing volatile species were transported to the QTA for atomization and detected by AFS.

2.3. Reagents

All chemicals used were at least of analytical grade and all solutions were prepared using high-purity water with a resistivity of 18.2 MΩ cm, obtained from a water purification system (Labconco, Kansas, USA). Stock standard solutions of 1000 mg L⁻¹ of Cd were obtained from National Analysis Center for Iron and Steel (Beijing, China). Methanol was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai).

Table 1
LSDBD-CVG-AFS operating parameters.

LSDBD-CVG-AFS parameter	Description
Lamp	Double cathode hollow cathode lamp
Atomizer	Quartz furnace atomizer (shielded flame)
Lamp current	80 mA (auxiliary cathode current: 40 mA)
Wavelength	228.8 nm
Photomultiplier tube voltage	−270 V
Carrier gas (discharge gas)	600 mL min ⁻¹
Hydrogen	40 mL min ⁻¹
Sample flow rate	2.0 mL min ⁻¹
Discharge gap	3 mm
Discharge power (input voltage)	25 W (220 V)
Methanol concentration	2%
Potassium chloride concentration	0.01 mol L ⁻¹

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