



# Bifunctional magnesium oxide crystal successively as adsorbent and matrix modifier for preconcentration and determination of arsenic by graphite furnace atomic absorption spectrometry



Wang Qiang<sup>a</sup>, Yu Zhuanni<sup>a</sup>, Lan Jing<sup>a,\*</sup>, Liu Aifeng<sup>b</sup>, Tian Yong<sup>b,\*</sup>

<sup>a</sup> College of Chemistry and Pharmaceutical, Qingdao Agriculture University, Qingdao 266109, PR China

<sup>b</sup> CAS Key Laboratory of Bio-based Materials, Qingdao Institute of Biomass Energy and Bioprocess Technology, Chinese Academy of Sciences, Qingdao 266101, PR China

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## ABSTRACT

A novel method for determination of total inorganic arsenic in water samples, based on magnesium oxide crystals quantitatively preconcentration and graphite furnace atomic absorption spectrometry (GFAAS) detection has been developed. As the MgO crystals could successively act as the adsorbent and matrix modifier, the method presents great potential in practical routine analysis of inorganic arsenic. With optimization of the experimental conditions, 1.5 mg of MgO was added into 4.0 mL water sample and ultrasonic dispersing 15 min for adsorbing the arsenic at first. Then, the mixture was centrifuged 5 min for separating the adsorbents and the water solution; the deposits were collected and dissolved with HNO<sub>3</sub> for arsenic determination. The enrichment factor of 13, limit of detection (LOD) of 0.087 μg L<sup>-1</sup>, relative standard deviation (RSD) of 4.5% ( $n = 7, c = 2.0 \mu\text{g L}^{-1}$ ) were achieved under the optimized conditions. The procedure was validated by analysis of certified water sample (GBW08605), which results in good agreement between the certified and the found values. The method was further demonstrated for analysis of lake water, snow water and tap water, and satisfactory spiked recoveries of 98.5% to 103.0% were achieved for these samples. These preliminary results indicated the present method was practical and accurate for analysis of trace-arsenic in natural water samples.

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## 1. Introduction

Arsenic in drinking water has caused severe health problems in many areas of the world, including South Asia, Middle of China, West of the USA, Mexico and Argentina, and even worse in developing countries and areas with low level of medical treatment and cognitive ability [1–3]. Millions of people are suffering the chronic poisoning and carcinogenic effects of arsenic. Melanosis, keratosis peripheral neuropathy, diabetes, ischemic heart disease, and impairment of liver function are commonly occurred with long-term exposing to the polluted drinking water [4–7]. Although the arsenic in drinking water has aroused the world wide attentions, and WHO (World Health Organization) has pointed out the maximum arsenic concentration of 10 μg L<sup>-1</sup> as the provisional guideline value in the guidelines for drinking-water quality [8], the drink water arsenic problems especially in the developing countries/areas are still unsolved because of insufficient understanding of the arsenic pollution levels [3,4,9]. At present, the detection and notification of the arsenic levels in these polluted areas are even more urgent than other remedial measurements. Consequently, the economic and practical arsenic detection methods are highly necessitated [10–13].

In natural waters, the arsenic is mostly inorganic, i.e., arsenate and arsenite ions, denoted as As(V) and As(III) [1,14]. However, the As(III) is unstable and rapidly converted to As(V) under ambient conditions [15]. The common methods for As(V) detection are atomic spectrometry methods, such as graphite furnace/electrothermal atomic absorption spectrometry (GFAAS/ETAAS) [16–18], hydride generation atomic fluorescence spectrometry (HGAFS) [19–23] and inductively coupled plasma mass spectrometry (ICPMS) [24–26]. The ICPMS provides the best sensitivity and accuracy among all the commercial detectors, but its high price and maintenance cost is still unaffordable for routine analysis in the developing countries and areas [10,11,27,28]. The GFAAS and HGAFS are more economic and would also provide accurate results while detecting clean samples with no/low level of interference ions [10,29]. The GFAAS and HGAFS methods require adding matrix modifier and hydride generation reagents for each sample detection, respectively. Because the limits of quantitation of arsenic by GFAAS and HGAFS are close to the suggested values of WHO (10 μg L<sup>-1</sup>) for the real water samples, the AAS and AFS cannot be directly used for arsenic determination in real drink water. The preconcentration and separation of arsenic in the water samples are necessary for the GFAAS and HGAFS methods [16,19,23,30].

To date, solid-phase extraction (SPE) [23,31,32], liquid-liquid extraction [33–35], cloud point extraction [30,36,37], co-precipitation [38] and atom/hydride trapping [39–41] have been established for arsenic

\* Corresponding authors.

E-mail addresses: [m13295322818@163.com](mailto:m13295322818@163.com) (J. Lan), [tianyong@qibebt.ac.cn](mailto:tianyong@qibebt.ac.cn) (Y. Tian).

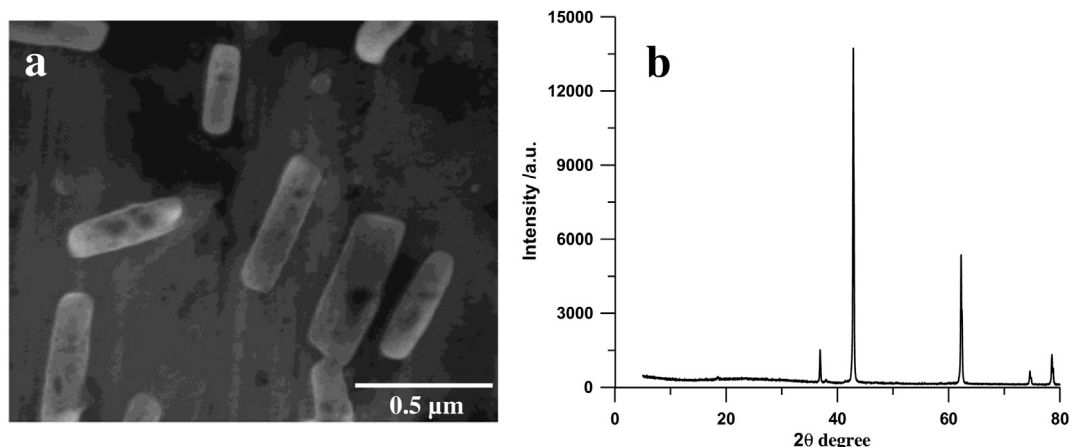


Fig. 1. SEM image (a) and the XRD pattern (b) of the MgO crystal particles.

preconcentration and determination. Recently, Chen et al. has invented a series of SPE methods for arsenic enriching and analytical performance improving of GFAAS and HGAAS, the adsorbents including akaganeite decorated graphene oxide composite [22], branch-polyethyleneimine modified carbon nanotubes [42] and esterified egg-shell membrane [43] were investigated for arsenic preconcentration, respectively. As a result, the limits of detection of HGAAS and GFAAS were significantly improved to tens nanogram per liter. Arsenic in snow, rain, pond water, spring water and certified reference materials could be accurately analyzed by these methods. Zhou et al. also reported a magnetic SPE method by using Fe@polyethyleneimine in combination with hydride generation atomic fluorescence spectrometry (HGAFS) for arsenic adsorption and detection, which enabled HGAFS to accurately analyze the arsenic in river water, reservoir water and cosmetic samples [23].

According to our knowledge, bifunctional materials as adsorbents and also as GFAAS matrix modifiers for preconcentration and determination

of arsenic have not been reported. Herein, MgO crystals were synthesized and used for arsenic adsorption, the MgO adsorbed with arsenic were then eluted and dissolved by nitric acid, and consequently the dissolved  $Mg^{2+}$  was functioned as matrix modifier for As detection by GFAAS. The primary results showed MgO crystals were practical for the arsenic preconcentration in real waters, and the analytical procedure were also simplified by  $Mg^{2+}$  performing as matrix modifier.

## 2. Experimental

### 2.1. Reagents and instrumentations

All chemicals used were at least analytical grade. De-ionized water ( $18.2 M\Omega\text{ cm}$ , Milli-Q water purification system) was used for standard reagents and sample dilutions. The standard stock solutions ( $1000\text{ mg L}^{-1}$ ) of As(V) were prepared by  $Na_3AsO_4 \cdot 12H_2O$  (Sigma). The As(V) standard

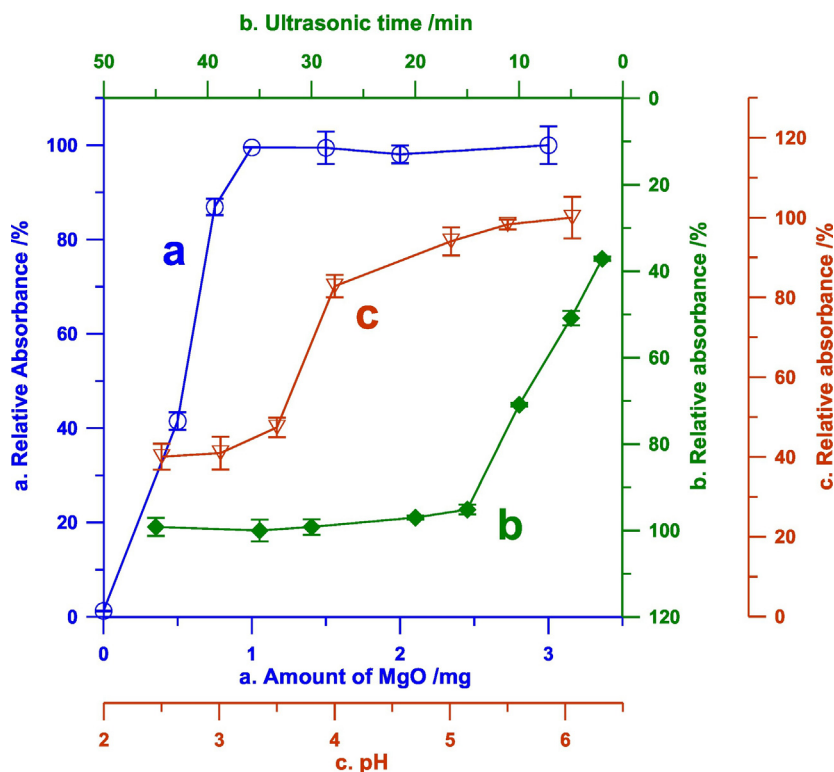


Fig. 2. The influences of the amount of MgO (a), ultrasonic time (b) and sample pH (c) on arsenic preconcentration and determination by GFAAS. Other experimental conditions are given in Table 1.

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