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Hydride generation coupled with thioglycolic acid coated gold nanoparticles as simple and sensitive headspace colorimetric assay for visual detection of Sb(III)

Tesfaye Tolessa ^{a, b}, Zhi-Qiang Tan ^a, Jing-Fu Liu ^{a, b, *}

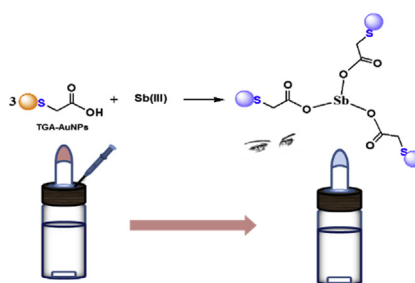
^a State Key Laboratory of Environmental Chemistry and Ecotoxicology, Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, P. O. Box 2871, Beijing 100085, China

^b University of Chinese Academy of Sciences, Beijing 100049, China

HIGHLIGHTS

- Thioglycolic acid coated gold nanoparticles were evaluated as headspace nanosensors for colorimetric detection of Sb(III).
- Chromogenic and hydride generation conditions were optimized to increase the method sensitivity and tolerance.
- The method exhibited quite high sensitivity, and acceptable tolerance with the aid of masking agent.
- The method showed high performance in detection of Sb(III) from environmental waters.
- This study is the first report on Sb(III) colorimetric detection on the basis of using nanosensors as chromophores.

GRAPHICAL ABSTRACT



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ABSTRACT

Antimony (Sb) is a toxic element which causes different health problems including cardiac problems and lung cancer in humans, and its levels in surface water can be noticeably increased to 100 µg/L typically in the proximity of anthropogenic sources. Thus, besides instrumental techniques, it is of great significance to develop a simple, sensitive and selective analytical method for direct analysis of Sb(III) at trace level without the need of any expensive and/or complicated instrumentations and sample preparation processes. Herein, a simple and sensitive headspace colorimetric assay was developed for the detection of Sb(III) by hydride generation coupled with thioglycolic acid functionalized gold nanoparticles (TGA-AuNPs). Sb(III) in the 30 mL sample solution was converted into its volatile form (SbH₃) through hydride generation reaction and headspace extracted into 100 µL chromogenic reagent, which contains methanol as extractant and TGA-AuNPs as nanosensors, leading to aggregation of TGA-AuNPs and therefore a red-to-blue color change. Parameters influencing the chromogenic and hydride generation reactions were optimized. Addition of 300 µM ethylenediamine tetraacetic acid (EDTA) as masking agent largely suppressed the interferences from mercury and arsenic. The proposed method can tolerate at least 10-fold As(III) and 100-fold other metal ions including Hg(II). The detection limits were 6.0 and 1.2 µg/L Sb(III) by naked-eye and UV-Vis spectrometer, respectively, which meet the maximum admissible level in

* Corresponding author. State Key Laboratory of Environmental Chemistry and Ecotoxicology, Research Center for Eco-Environmental Sciences, Chinese Academy of Sciences, P. O. Box 2871, Beijing 100085, China.

E-mail address: jfliu@rcees.ac.cn (J.-F. Liu).

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drinking water (6 µg/L) set by the United States Environmental Protection Agency. The feasibility of the proposed method was demonstrated by rapid detection of Sb(III) in river water, lake water, ground water and sea water samples by naked-eye at a spiking level of 6 µg/L Sb(III).

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1. Introduction

Antimony (Sb) is a toxic and hazard element which enters the aquatic environments through rock weathering, soil run off and anthropologic activities [1,2]. Prolonged exposure to Sb can lead to serious health problems including keratitis, dermatitis, gastritis and conjunctivitis. Long term exposure is also associated with the occurrence of cardiac problems and lung cancer in humans [3,4]. Thus, Sb and its compounds have been designated as priority pollutant by the United States Environmental Protection Agency (USEPA) and World Health Organization (WHO), which have set the maximum admissible level in drinking waters as 6 and 20 µg/L, respectively [5,6]. The physiochemical as well as toxic properties of Sb strongly depend on its oxidation state rather than total concentration. From the two main oxidation states in the environment, Sb(III) is about ten times more toxic than Sb(V), which is the dominant species in environmental waters [7,8]. Dissolved Sb is usually present in surface water at very low concentrations, typically less than 1 µg/L, in unpolluted water samples [9]. However, the concentrations can be noticeably increased to 100 µg/L in the proximity of anthropogenic sources [10,11]. For this reason, detection and monitoring of Sb have been compulsory.

Various instrumental techniques such as inductively coupled plasma mass spectrometry [12,13], inductively coupled plasma atomic emission spectrometry [12,14], hydride generation atomic absorption spectroscopy [15], hydride generation atomic fluorescence spectrometry [16] and electrothermal atomic absorption spectrometry [17,18] have been employed for the determination of Sb species after their separation and preconcentration. Although hydride generation (HG) coupled with atomic spectroscopy and mass spectrometry are the most widely used methods for antimony detection because of their high sensitivity and high selectivity [15,16], these methods are often negatively affected by the presence of some transition and noble metals that may produce severe interferences and thus involve complicated sample preparation processes to achieve better selectivity [19]. In addition, these methods often require quite expensive, sophisticated and bulky instrumentations with various long-drawn-out and complicated operational procedures. Therefore, these techniques are hardly suitable for routine and in-field analyses. Thus, it is of great importance to develop a simple, rapid, sensitive and inexpensive method for *in-situ* Sb(III) detection.

Recently, colorimetric methods based on nanosensors are regarded as promising approaches and have already obtained increasing attention for detection purpose because of their simplicity, high sensitivity, low cost and applicability for naked-eye test [20]. Gold nanoparticles (AuNPs) have been widely used as nanosensors in colorimetric detection recently because of their high extinction coefficient and therefore higher sensitivity than organic probes [21]. When AuNPs aggregate, the colour of AuNPs solution changes from red to blue and the surface Plasmon band broadens and shifts to longer wavelength [22]. Based on this mechanism, colorimetric detection techniques based on analyte-induced aggregation of AuNPs have been designed and employed to detect certain analytes by naked eyes at fairly low concentrations. However, most AuNPs-based colorimetric detection techniques suffer

from low tolerance to interferences, limiting their application in samples with complex matrices like environmental waters. To solve this problem, introducing an appropriate separation and preconcentration procedure prior to visual detection is a very efficient alternative [20,23].

Herein, for the first time, a simple, rapid and sensitive HG technique coupled with the AuNPs-based colorimetric detection technique for analytical purpose was developed for detection of trace Sb(III). Specifically, a novel approach termed HG coupled with headspace colorimetric detection (HG-HS-CD) was developed for the determination of Sb(III) at low ppb level by using analyte-induced aggregation of thioglycolic acid coated AuNPs with the assistance of ethylene diamine tetracetic acid (EDTA) to enhance method tolerance towards potential interferences. Sb(III) in a sample solution can be derivatized into its volatile form (SbH₃) through HG reaction, which can be rapidly transferred to the headspace above the solution. Then, SbH₃ can be headspace extracted or solubilized into the AuNPs solution containing methanol as extractant, and further reacted with TGA-AuNPs, leading to the aggregation of TGA-AuNPs that is induced by the binding between Sb(III) and TGA on the surface of AuNPs, resulting in a color change from red-to-blue. Based on the interaction between Sb(III) and TGA-AuNPs, we intended to design a headspace detection system by our naked eye. To the best of our knowledge, this is the first demonstration for simple and direct analysis of Sb(III) on the basis of colorimetric assay using nanoparticles as chromophores.

2. Experimental section

2.1. Instruments

The transmission electron microscopy (TEM) images were recorded by a Hitachi H-7500 (Tokyo, Japan) at an accelerating voltage of 80 kV. A double beam UV-Vis spectrometer (a Shimadzu UV-3600; Kyoto, Japan) was used to measure the absorption spectra of AuNPs. A multimode microplate spectrometer (Varioscan Flash, Thermo) with a 384 well-plate was employed for recording the absorbance at 520 and 650 nm. Since the maximum absorbance wavelength (λ_{max}) of the TGA-AuNPs solution shifted from 520 to 650 nm in the presence of Sb(III) and the ratio of absorbance at 650 to that at 520 nm ($A_{650/520}$) increases with Sb(III) concentration, $A_{650/520}$ was selected as instrumental response. ICP-MS instrument (Agilent 7700cs, USA) was used to quantify Sb(III) contents in the real water samples using Sb(III) standard solutions prepared by diluting a reference material (1000 mg/L) with 5% nitric acid.

2.2. Reagents and solutions

Chloroauric acid tetrahydrate (HAuCl₄·4H₂O) and trisodium citrate were purchased from Sigma-Aldrich (St. Louis, MO). Thioglycolic acid was obtained from Tokyo Chemical Industry (Tokyo, Japan). Antimony trichloride (SbCl₃) and potassium antimony tartrate (C₄H₄KO₇Sb·0.5H₂O) were obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Potassium hexahydroxyantimonate (KSb(OH)₆) was obtained from Sigma-Aldrich (St. Louis, MO). EDTA and KBH₄ were obtained from Sinopharm

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