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Talanta

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Short communication

A non-aggregation spectrometric determination for mercury ions based on gold nanoparticles and thiocyanuric acid



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ARTICLE INFO

Article history:

Received 21 October 2014

Received in revised form

27 November 2014

Accepted 29 November 2014

Available online 22 December 2014

Keywords:

Colorimetric

Gold nanoparticles

Thiocyanuric acid

Mercury ion

ABSTRACT

We provide a highly sensitive and selective colorimetric assay to detect mercury ions (Hg^{2+}) in aqueous environment using thiocyanuric acid (TCA) molecule-functionalized gold nanoparticles (AuNPs). This method is based on the thiophilicity of Hg^{2+} and AuNPs as well as the unique optical properties of TCA-functionalized AuNPs. In the presence of TCA, AuNPs aggregate due to the strong attraction between thiol groups of TCA and surface-bounded $\text{AuCl}_4^-/\text{AuCl}_2^-$ ions, which induces the visible color change from red to blue. With the addition of Hg^{2+} , Hg^{2+} is more apt to interact with thiols than AuNPs. Thus, Hg^{2+} can remove the AuNPs of the TCA-functionalized AuNPs and trigger AuNP aggregation redisperse again. This assay can selectively detect Hg^{2+} with the detection limits as low as 0.5 nM in aqueous solution.

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1. Introduction

Mercury is highly toxic element in the environment [1,2]. Mercury accumulation even in low concentration in the human body can cause diseases such as prenatal brain damage, serious cognitive and movement disorders, and Minamata disease [1,2]. Thus, it is highly desirable to develop facile, economical and rapid methodologies with favorable sensitivity and selectivity for real time Hg^{2+} detection in biological samples or in human bodies.

The detection of Hg^{2+} has become an increasing demand in recent years. Many efforts have been exerted to detect Hg^{2+} using various detection techniques, such as optical spectroscopy [3–6], electrochemical methods [7,8], high-performance liquid chromatography [9,10], and inductively coupled plasma mass spectrometry [10,11]. However, most of these methods require expensive instrumentation and complicated sample preparation, which make them inappropriate for in-situ applications. To overcome these drawbacks, a variety of colorimetric sensors based on gold nanoparticles (AuNPs) have been designed for the simple, rapid detection of Hg^{2+} [12–15].

Spectrometric determination based on AuNP aggregation have recently attracted considerable interests in diagnostic applications mainly due to their simplicity, versatility, and comparable sensitivity with the aforementioned methods. The aggregation of AuNPs can induce a rapid visible color change from wine-red to blue due to the coupling of interparticle surface Plasmon [16,17].

Thus, the color change of AuNPs induced by aggregation or redispersion of AuNPs provides a platform for the colorimetric detection of analyte. In most cases, AuNPs are modified with oligonucleotide- or thiol-containing organic molecules to indicate color change. For example, Jiang et al. provided a highly sensitive and selective method to detect Hg^{2+} through quaternary ammonium group-terminated thiols modified AuNPs [18]. Liu et al. demonstrated a novel and practical system for colorimetric detection of mercury based on DNA/nanoparticle conjugates [13]. However, these sensors are cost- or pollution-consuming. Therefore, it is vital to develop a facile, low cost, rapid, and eco-friendly method for Hg^{2+} detection.

Herein, by taking advantage of the formation of Au-S bonds between thiocyanuric acid (TCA) and AuNPs and higher affinity of TCA toward Hg^{2+} over AuNPs, a very simple colorimetric sensor was designed for Hg^{2+} detection based on the TCA and AuNPs. This reliable sensing platform allows direct analysis of the samples by the naked eye or simple instruments. Furthermore, the limit of detection (0.5 nM) of this method is also lower than that of the other colorimetric sensors. Therefore, the developed colorimetric sensor was reliable, inexpensive, and sensitive.

2. Experimental

2.1. Reagents and apparatus

Chloroauric acid trihydrate and sodium citrate were purchased from Sigma-Aldrich (USA). Thiocyanuric acid was obtained from the Alfa Aesar (Tianjing, China) company. Standard mercuric ion in

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2–5% nitric acid was purchased from AccuStandard (Beijing, China). All other reagents were of analytical grade. All solutions were prepared with Milli-Q water from a Millipore system.

Ultraviolet-visible (UV–vis) absorption spectra were recorded on an UV-2550 Spectrophotometer (Shimadzu Corporation). Transmission electron microscope (TEM) images were obtained on a Hitachi (H-7650, 80 kV) transmission electron microscope.

2.2. Preparation of AuNPs

The 15-nm diameter AuNPs were prepared according to the literature [19]. First, an aqueous solution (100 mL) containing HAuCl₄ (0.25 mM) under stirring and brought to a boil in a conical flask (250 mL). Upon boiling, 1% sodium citrate (3 mL) was added quickly and left to continue boiling for another 30 min under stirring, until the solution turned red. The final product was stored at 4 °C. The size of 15 nm and concentration (10 nM) of AuNPs were obtained from UV–vis data according to reference [20].

2.3. Colorimetric detection of Hg²⁺

In order to demonstrate the detection of Hg²⁺, first, 20 μM TCA solution in 500 μL Tris–HCl buffer solution (10 mM, pH=7.0) was added into a 1.5 mL plastic vial containing 500 μL AuNP solution

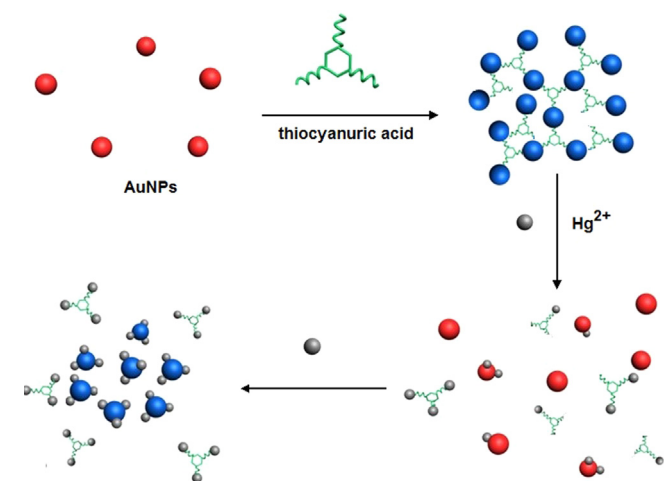


Fig. 1. Schematic of the colorimetric detection strategy for Hg²⁺ based on AuNPs and TCA.

(10 nM) under stirring. And then a series of Hg²⁺ with various concentrations was added into the mixture. The solution was mixed thoroughly, which ensured that Hg²⁺ completely bound onto the TCA-AuNPs. After that, the resulting solution was transferred to a 1 cm path length quartz cuvette for spectral recording. The UV–vis absorption spectra were measured over the wavelength range from 300 nm to 800 nm. All assays were performed at room temperature.

3. Results and discussion

3.1. Sensing strategy

Fig. 1 outlines the sensing mechanism employed in this work. The AuNPs in solution remain dispersed and the solution shows wine-red color because the AuNPs are capped by negatively charged citrate ions and the electrostatic repulsion between them keeps them separate from aggregation. The thiol group of TCA can be easily attached to the surface of the AuNPs by the formation of Au–S bonds. Accordingly, the AuNPs in the presence of TCA are crosslinked and the solution color changes to blue. While the addition of Hg²⁺ into AuNPs–TCA solution, a significant color change from blue to red is observed because Hg²⁺ is more apt to interact with thiols than AuNPs. Thus, it can remove the AuNPs and closely combine with thiols. When the content of Hg²⁺ combined with thiols of TCA reaches saturation, with the further addition of Hg²⁺, Hg²⁺ can absorb on the surface of AuNPs, decreases the negative charge density on the surface of AuNPs and diminishes the stability of AuNPs, resulting in the aggregation of AuNPs and a corresponding red-to-white color change.

3.2. Sensitivity of TCA-AuNPs to Hg²⁺

It is well known that Hg²⁺ is apt to interact with thiols and amino groups such as cysteine [21] and melamine [22]. Thus, a rational strategy to detect Hg²⁺ is to combine TCA-AuNPs and Hg²⁺. Inspired by this theory and under the optimized detection condition (C_{TCA}=20 μM, pH=7, real time detection (less than 30 s), Fig. S1), the sensitivity of the sensor to Hg²⁺ was investigated. As shown in Fig. 2A, with the increase of Hg²⁺ concentration, an increase in the intensity of the absorbance at 530 nm can be observed. It indicated that the TCA-AuNPs aggregation induced redispersion with the increase of Hg²⁺ concentration, and a progressive color change from blue to red was observed (inset of Fig. 2B). When Hg²⁺ interacted

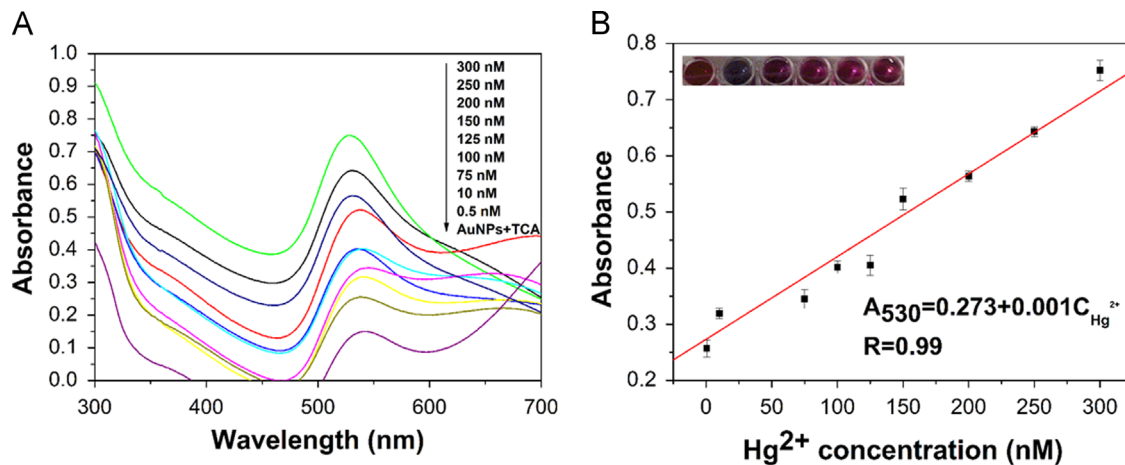


Fig. 2. (A) Absorbance response for different concentrations of Hg²⁺. (B) The peak absorbance value at 530 nm is linear with Hg²⁺ concentration over the range from 0.5 to 300 nM. Inset of Fig.2(B): The photographic images of TCA-AuNPs with various concentrations of Hg²⁺, from left to right: (a) AuNPs, (b) AuNPs-TCA, (c) 0.5 nM Hg²⁺, (d) 100 nM Hg²⁺, (e) 200 nM Hg²⁺, and (f) 300 nM Hg²⁺.

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