



Colorimetric sensor for thiocyanate based on anti-aggregation of citrate-capped gold nanoparticles

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ARTICLE INFO

Article history:

Received 16 April 2013

Received in revised form 2 October 2013

Accepted 12 October 2013

Available online xxx

Keywords:

Colorimetric sensor

Thiocyanate

Gold nanoparticles

Surface plasmon resonance

Anti-aggregation

ABSTRACT

A convenient colorimetric sensor based on anti-aggregation of citrate-capped gold nanoparticles (AuNPs) is demonstrated for thiocyanate (SCN^-) sensing. The citrate-capped AuNPs aggregated readily in the presence of sulfuric acid, leading to a color change from red to blue. This particle aggregation could be suppressed by SCN^- , which had strong protective effect on the gold colloid. The thiocyanate recognition suffers insignificant interference from most of common ions. With this strategy, $1 \mu\text{M}$ of SCN^- can be recognized within 5 min by naked-eye observation. This simple, selective, and sensitive approach can be potentially applied for SCN^- assays in environmental aqueous samples.

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1. Introduction

Thiocyanate is widely employed in medicine, dyeing, photography, prevention of erosion and other applications. It is very detrimental to the environment due to the generation of highly toxic chemical species, such as CN^- , CNCl by irradiation and chlorination. Therefore, the determination of SCN^- is of important practical significance. Many strategies, such as colorimetry [1], atomic absorption spectrometry [2], electrochemistry [3–6], gas chromatography/mass spectrometry [7,8], ion mobility spectrometry [9], electrophoresis [10,11], fluorimetry [12], and double-valve sequential injection analysis [13], have been documented for the quantification of thiocyanate in the literature. However, many of these methods are complicated, time-consuming, costly, and tedious to perform and involve unpleasant or harmful reagents. It is essential to establish a simple, rapid, and sensitive method for the determination of SCN^- .

Metal nanoparticles have received considerable attention in many fields because of their unique chemical, optical, electronic, magnetic, and catalytic properties, which make them ideal candi-

dates for signal generation and transduction in the determination of various analytes. Among the nanomaterials used as component in sensing, gold nanoparticles (AuNPs) have received greatest interests because of their strong surface plasmon resonance (SPR) absorptions in the visible region, simple preparation, high stability, excellent biocompatibility, and versatile surface chemistry [14]. A great many analytical strategies based on AuNPs have been developed, including fluorescence, electrochemistry, colorimetry, surface enhanced Raman scattering, quartz crystal microbalance, bio-barcode assays and so forth [15]. Among these approaches, colorimetry, mostly based on the surface plasmon resonant properties of AuNPs, has attracted fast-growing interest because the analyte recognition events can be directly monitored by the naked eye with high sensitivity, simplicity, and low cost without the requirement of the sophisticated instruments. In such assays, color change of AuNPs aggregation (or redispersion of an aggregate) is triggered in the presence of the analyte of interest. Many sensing approaches based on the aggregation of AuNPs have been established in a variety of detection formats for DNA [16], proteins [17], cells [18], small molecules [19–21], and ions [22–24]. However, it should be noted that AuNPs aggregation processes can be induced by many external factors, leading to false positive signals and incorrect results. In order to obtain higher selectivity, colorimetric sensor based on anti-aggregation or re-dispersion of AuNPs becomes a good alternative. Recently, some colorimetric methods based on anti-aggregation of AuNPs have been proposed [25–29]. In these assays, the AuNPs

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surface is usually modified with specific binding ligand (such as DNA), which increases the tedious labor and the corresponding cost. Therefore, the challenging question whether AuNPs without complicated modification can recognize targets specifically arises.

In this study, we demonstrated a convenient colorimetric method for the determination of SCN^- by employing citrate-capped AuNPs as probes without further modification. We found that the citrate-capped AuNPs aggregated readily in the presence of sulfuric acid (8 mM), leading to a color change of the colloidal solution from red to blue. This particle aggregation was, however, selectively suppressed by SCN^- , which had strong protective effect on the gold colloid. We applied this method to determine SCN^- in real water samples, and satisfactory results were achieved.

2. Materials and methods

2.1. Chemical and apparatus

Potassium thiocyanate (KSCN) and trisodium citrate were purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). $\text{HAuCl}_4 \cdot 4\text{H}_2\text{O}$ and ethylenediaminetetraacetate (EDTA) were purchased from Aladdin Reagent Company (Shanghai, China). All other reagents were at least analytical reagent grade. All solutions were prepared with Milli-Q water from Millipore system.

The UV–vis spectra of AuNPs were recorded by a Shimadzu UV-2450 spectrophotometer (Shimadzu, Japan).

2.2. Synthesis of the citrate-capped AuNPs

All glassware used in the following procedures was cleaned in a bath of freshly prepared solution of HNO_3 – HCl (1:3, v/v), rinsed thoroughly in water and dried in air prior to use. AuNPs colloids with an average diameter of 13 nm were prepared according to previously published protocols [30]. Briefly, 1 mL of 1% HAuCl_4 solution was dissolved in 100 mL of water and boiled. 3 mL of 1% trisodium citrate solution was quickly added to the refluxed HAuCl_4 solution, resulting in a color change from pale yellow to wine-red, indicating the formation of gold nanoparticles. After a continuous reflux for an additional 15 min, the solution was slowly cooled down to room temperature. The wine-red solution of AuNPs was stored at 4 °C in refrigerator. The particle concentration of AuNPs (ca. 3.2 nM) was

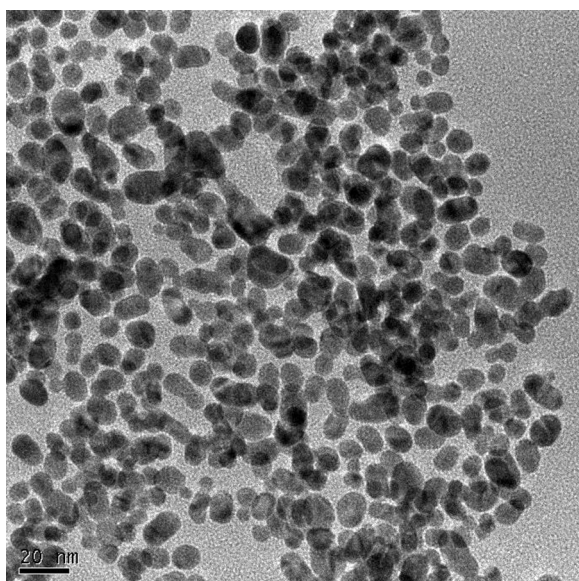


Fig. 1. TEM image of the citrate-capped gold nanoparticles.

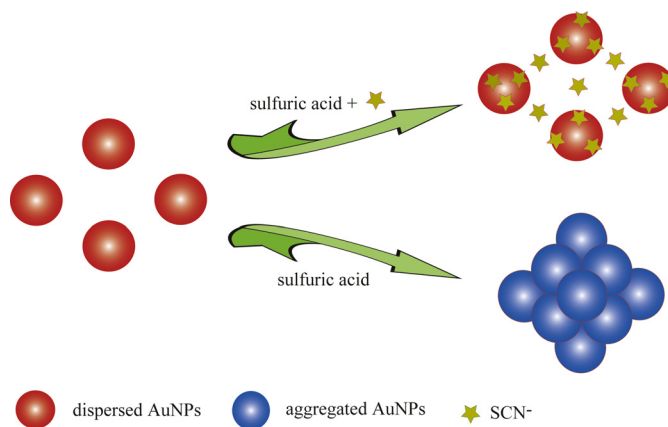


Fig. 2. Schematic illustration of colorimetric detection of SCN^- based on the anti-aggregation of citrate-capped AuNPs.

determined according to Beer's law using an extinction coefficient of ca. $2.7 \times 10^8 \text{ M}^{-1} \text{ cm}^{-1}$ at 520 nm for 13 nm AuNPs [31].

2.3. Sample pretreatment

For the water samples analysis, 10 mL of the tap water was collected and EDTA (final concentration was 1 mM) was added to eliminate the influence of some metal ions. After filtered through a 0.22 μm membrane, the water samples were spiked with different amounts of SCN^- and used for colorimetric assay.

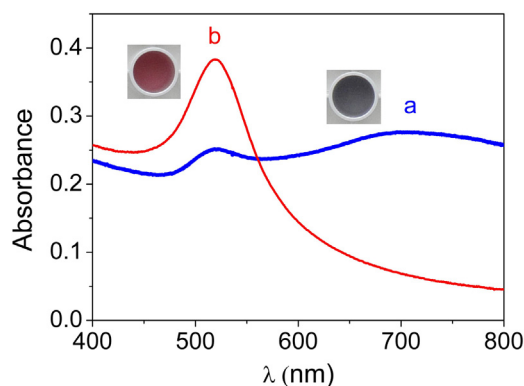


Fig. 3. The absorption spectra of (a) citrate-capped AuNPs + 8 mM H_2SO_4 and (b) citrate-capped AuNPs + 2.5 μM SCN^- + 8 mM H_2SO_4 . Inset: the corresponding photographs.

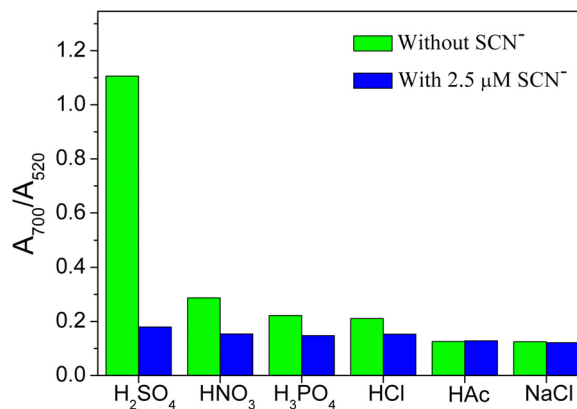


Fig. 4. Effect of the type of aggregation inducer (8 mM) on the assay.

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